

**Measurement Technology Laboratories**

**PM2.5 Teflon Filters**

**Quality Assurance Project Plan**

**Category IV**

**Submitted to  
U.S. Environmental Protection Agency  
Research Triangle Park  
Durham, NC 27711**

**Project Officer:** Margaret A. Dougherty  
Phone: (919) 541-2344  
Email: [daugherty.margaret@epa.gov](mailto:daugherty.margaret@epa.gov)

**Administrative Contracting Officer:** Robert D. Flowers  
Phone: (919) 541-2182  
Fax: (919) 541-0611  
[flowers.rob@epa.gov](mailto:flowers.rob@epa.gov)

**Administrative Contracting Specialist:** Beverly A. Barbour  
Phone: (919) 541-4095  
Fax: (919) 541-0611  
Email: [barbour.beverly@epa.gov](mailto:barbour.beverly@epa.gov)

**Note: All Contacts Listed above have same address above.**

**Contract No.: EP-D-10-009**

**Requisition/Purchase Request Project No.: PR-NC-09-10333**

**Abstract:** This document details the Category IV Quality Assurance Project Plan to guide the successful implementation of supply of PM2.5 Teflon Filters used in the collection of PM2.5 by State and Local air pollution control agencies.

**A PROJECT MANAGEMENT****A1. Approval Sheet****US EPA Representatives**

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**Margaret A. Dougherty**  
U.S. Environmental Protection Agency  
Project Manager

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Date

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**Robert D. Flowers**  
U.S. Environmental Protection Agency  
Administrative Contracting Officer

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Date

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**Beverly A. Barbour**  
U.S. Environmental Protection Agency  
Administrative Contracting Specialist

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Date

**Measurement Technology Laboratories, LLC Representatives**

The key individuals involved in major aspects of the project (no contractors are engaged):

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**David A. Dikken**  
Measurement Technology Laboratories, LLC  
CEO

July 12<sup>th</sup>, 2010  
Date

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**Brian Dueber**  
Measurement Technology Laboratories, LLC  
COO (functioning Project Manager)

July 12<sup>th</sup>, 2010  
Date

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**Sonny Moore**  
Measurement Technology Laboratories, LLC  
Quality Manager

July 12<sup>th</sup>, 2010  
Date

- A2. Table of Contents (Not required for a Category IV QAPP)**
- A3. Distribution List (Not required for a Category IV QAPP)**
- A4. Project/Task Organization (Not required for a Category IV QAPP)**
- A5. Project Definition and Background (Not required for a Category IV QAPP)**
- A6. Project/Task Description**

### **Project overview**

The U.S. Environmental Protection Agency (EPA), Research Triangle Park, North Carolina is responsible for procuring, testing, and distributing filters of high purity to the State/local air pollution agencies. These filters are used in conjunction with air samplers to collect PM<sub>2.5</sub> sample (particulate matter with an aerodynamic diameter less than or equal to a nominal 2.5 micrometers). The quantification of the measurement is founded upon a gravimetric approach in which these filters are pre-weighed, then exposed via sampling to ambient air, and then post-weighed. The sampling and weighing processes require filters with sufficient conformance to specifications and practical application so as to provide successful completion of the PM<sub>2.5</sub> air monitoring program objectives.

### **Objectives of the project**

The objectives of this QAPP are to ensure submitted filters adhere to 40 CFR Part 50 Appendix L and all requirements within the Statement of Work. These parameters include:

1. Physical Size
2. Medium
3. Support Ring
4. Pore Size
5. Filter Thickness
6. Maximum Pressure Drop (clean filter)
7. Maximum moisture pickup
8. Collection efficiency
9. Alkalinity
10. Special Requirement (which includes the trace metals)

For both the supplier and the US Environmental Protection Agency, it is imperative that each batch shipment (1 yr supply) be in full conformance and as such a key element is to submit a qualification set of filters with reports for review prior to each batch run.

### **Project summary and work schedule**

This project's major tasks and timeline are outlined in the table below.

**Table: Schedule of Major Project Tasks**

| <b>Task Name</b>                    | <b>Task Description</b>   | <b>Start Date</b>          | <b>End Date</b>            |
|-------------------------------------|---|----------------------------|----------------------------|
| Production                          | Preparation of Specified PM2.5 Filters per Statement of Work  | Acceptance of QAPP         | 120 Days                   |
| Production Quality Assurance        | Perform Quality Assurance Sampling and Testing Protocol   | Production                 | End of Production Run      |
| Measures Identification             | Test & Qualification Report   | Completion Of Production   | With Submission of Filters |
| Submission of Qualification Filters | Submit 100 Tested / Printed / Packaged Filters for review by EPA. Step used to confirm all aspects before producing 200K filters. | At beginning of Production | 1 Week                     |
| Approval of Qualification Filters   | Confirmation of appropriateness of test results, packaging, and any other issues by US EPA  | TBD                        | TBD                        |
| Follow Up                           | After annual submission of filters, follow up with US EPA representatives to identify any quality improvement opportunities.      | Delivery Date +14 Days     | Upon Feedback              |

### Resource and time constraints

Key element is insuring approval of all technical aspects of Filter Production before lot run of approximately 240K filters is commenced. The production run will take 100+ days so care must be taken to ensure that the 120 day delivery date is maintained. To gain margin, the production and testing of the 100 qualification filters has commenced.

### Key Facility

The key facility in this project is Laboratory 201 the “filter production clean room and laboratory”. This facility is maintained according the requirements in the Quality Management Plan and this QAPP.

The clean room and laboratory performance details are compliant with ISO 14644 Parts 1 through 5 which are adopted into operations and included in appendix E 1.

Application specific requirements beyond those addressed in the ISO 14644 include:

- 1) Static Dissipative Methods.
  - a. Static dissipative methods are employed using Po210, an alpha particle emitter.
  - b. Corona approaches are not suitable for highly electronegative materials such as PTFE, PMP, and PFA.
  - c. Alpha emitter’s effective range is optimized at 16 mm ±4 mm.
  - d. Grounded surfaces, included filters storage, do not aid in discharge and provide a ready supply of electrons which will highly charge materials high on the triboelectric scale.
  - e. Static Dissipative actions should be taken aggressively during the weighing process. I.e. discharge occurring while the filter is on the weighing pan.
- 2) Static Nullification Methods.

- a. Excess charge on filters can be nullified by proper weighing set up.
  - b. The use of a Faraday Cage integral to the balance pan.
  - c. The grounded Faraday Cage will generate a mirror image to induce a force which is internal to the balance pan and does not affect weighing results.
- 3) Air Flow Rates
- a. Air Flow Rates identified within ISO 14644 are not appropriate for a microgram or submicrogram weighing environments.
  - b. Air Flow rates are controlled  $< 2\text{m}/\text{min}$ .
- 4) Filter production practices within the clean room include:
- a. All raw production material components are to remain sealed until in use in production.
  - b. All production events occur before examination for contamination and packaging.
  - c. All packaging is cleaned of particulates before entering clean room.
  - d. Clean room staff are trained in:
    - i. Clean room dress/clothing: including Head Cover, Foot Covers, and gloves.

## **A7. Quality Objectives and Criteria**

### **A7.1 Detailed Performance Measures**

This project is primarily interested in the following list of performance measures.

Physical Attribute and performance measures (required for any lots specifically identified with the PM2.5 Reference Method as well as submitted batches).

- 1) Size: Circular, 46.2 mm diameter  $\pm 0.25$  mm
- 2) Medium: Polytetrafluoroethylene (PTFE Teflon), with integral support ring.
- 3) Support Ring: Polymethylpentene (PMP) or equivalent inert material (PFA). 0.38 mm thick  $\pm 0.04$  mm. Outer diameter: 46.2 mm  $\pm 0.25$  mm, and width of 3.68 mm  $+0.00$  mm,  $-0.51$  mm.
- 4) Pore size: 2  $\mu\text{m}$  as measured by ASTM F316-94
- 5) Filter thickness: 30 to 50  $\mu\text{m}$ .
- 6) Maximum Pressure Drop (clean filter): 30 cm H<sub>2</sub>O column @ 16.67 L/min clean air flow.
- 7) Maximum moisture pick up: Less than 10  $\mu\text{g}$  weight increase after 24 hour exposure to air of 40% RH relative to weight after 24 hour exposure to air at 35% RH.
- 8) Collection Efficiency: Greater than 99.7% as measured by the DOP test (ASTM D 2986a-99) with 0.3  $\mu\text{m}$  particles at the sampler's operating face velocity.
- 9) Filter weight stability: The average filter weight loss shall be less than 20  $\mu\text{g}$ , as measured in each of the following two tests. Filter weight loss shall be the average difference between the initial and the final filter weights of a random sample of test filters selected from each lot prior to sale. The filters test shall be 250 filters for each of the tests. Conditioning and methods shall follow both 40 CFR, Part 50, Appendix L, and "Quality Assurance Guidance Document 2.12: Monitoring PM2.5 in Ambient Air using Designated Reference or Class I Equivalent Methods."

- a. Test for loose, surface particle contamination. After the initial weighing, each filter will be installed in a filter cassette, and dropped from a height of 25 cm onto a clean particle free surface. Repeat two more times for a total of 3 drops. Each filter is then removed and reweighed. The average change must be less than 20 µg.
- b. After 9(a) is completed each filter is then exposed to a drying oven set at 40°C ±2°C for not less than 48 hrs. Remove, condition, and reweigh each test filter. The average change must be less than 20 µg.
- 10) Alkalinity: Less than 25 micro-equivalents/gram of filter, as measured by the procedure given in the above reference.
- 11) Packaging: The filters of production lots shall be packed in two sets (of 25 filters each) for a total of 50 filters in a suitable clear, hard, dust-tight, non-contaminating plastic container. The top of each container shall be appropriately labeled, including serial numbers corresponding to the filters contained therein. The exterior of each box of filters shall be marked with the box number (i.e., Box 1 (T5000001 – T50000050)).
- 12) Compliance with Specifications: Table 1.

Table I. Physical Property Performance Measures &amp; Quality Control Quantity and Test Method

| Test No. | Filter Characteristic              | Quality Control Test Quantity (Per Lot) | Test Analysis               | Max                    | Min            | Method Description                        | Method Detail                                     |
|----------|------------------------------------|---|-----------------------------|------------------------|----------------|---|---|
| 1        | Reinforcing Ring, Outside Diameter | 50                                      | Caliper                     | 46.45 mm               | 45.95 mm       | Direct Outside Diameter Measurement       | Mitutoyo Metrology Handbook                       |
| 2        | Reinforcement Ring Width           | 50                                      | Caliper                     | 3.68 mm                | 3.17 mm        | Direct Ring Width Blade Point Measurement | Mitutoyo Metrology Handbook                       |
| 3        | Reinforcement Ring Thickness       | 50                                      | Micrometer                  | 0.42 mm                | 0.31 mm        | Direct Measurement With Set Pressure      | Mitutoyo Metrology Handbook                       |
| 4        | Pressure Drop at 16.7 L/min        | 50                                      | Monodisperse DOP Smoke Test | 30 cm H <sub>2</sub> O | Not Applicable | ASTM D2986a-99                            | Aerosol Generation w/ Light Scattering Photometer |
| 5        | Retention of Particles 0.3 µm dia. | 50                                      | Monodisperse DOP Smoke Test | na                     | 99.70 %        | ASTM D2986a-99                            | Aerosol Generation w/ Light Scattering Photometer |
| 6        | Number of Filter Container         | All Containers                          | Visual                      | No Defect Permitted    |                | Count                                     | Direct Count                                      |
| 7        | Calendar Year ID                   | All Containers                          | Visual                      | No Defect Permitted    |                | Observation                               | Direct Observation                                |
| 8        | Visual Test for Defective Filters  | 250                                     | Visual                      | 10% (10)               | none           | Visual Inspection per SOW Criteria        | See A7.1.13 For Details                           |
| 9        | Visual Test for Rejected Filters   | 250                                     | Visual                      | 5% (5)                 | none           | Visual Inspection per SOW Criteria        | See A7.1.13 For Details                           |

|    |                               |     |              |                    |        |                              |   |
|----|-------------------------------|-----|--------------|--------------------|--------|------------------------------|---|
| 10 | Filter Weight Stability       | 250 | Gravimetric  | +20 µg             | -20 µg | ABA Drift Corrected Weighing | NIST HB145<br>SOPs:2,4,29,30<br>GLP:1,GMP's:<br>10,13 |
| 11 | Filter Weight Moisture Pickup | 250 | Gravimetric  | 10 µg              | 0 µg   | ABA Drift Corrected Weighing | NIST HB145<br>SOPs:2,4,29,30<br>GLP:1,GMP's:<br>10,13 |
| 12 | Metals Analysis               | 50  | Spectroscopy | Varies per Element | 0      | EPA Compendium Method IO-3.3 | X-Ray Fluorescence (XRF)                              |

***Note on Test numbers 4 and 5: ASTM Method D2986-91 and subsequently ASTM Method D2986-94 titled "Standard Practice for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test" was withdrawn in 2004. However D ASTM D2986a-99 available even though according to ASTM the status is:***

**WITHDRAWN, NO REPLACEMENT**

**Developed by Subcommittee: D22.01**

**Withdrawn Rationale:**

The dioctyl phthalate (DOP) smoke test is a highly sensitive and reliable technique for measuring the fine particle arresting efficiency of an air or gas cleaning system or device. It is especially useful for evaluating the efficiency of depth filters, membrane filters, and other particle-collecting devices used in air assay work.

Formerly under the jurisdiction of Committee D22 on Sampling and Analysis of Atmospheres, this practice was withdrawn in December 2004. This practice is being withdrawn because the procedure is 34 years-old and the apparatus identified (the Optical Owl) is no longer available.

Table II. Physical Property Performance Measurement, Statistical Confidence Limits and Sampling Patterns

| Test No. | Filter Characteristic                       | Minimum Number of QC Measurements (Per Lot) | No. of Measurements Per Filter | Estimated Expanded Uncertainty $\pm (k=2)$ 95% Confidence | Sampling Pattern    | Sampling Timing & Schedule               |
|----------|---|---|--------------------------------|---|---------------------|--|
| 1        | Reinforcing Ring, Outside Diameter          | 50  | 1                              | $\pm 0.15$ mm   | Whole Unit          | 1 per 2000 filters                       |
| 2        | Reinforcement Ring Width                    | 50  | 1                              | $\pm 0.15$ mm   | Whole Unit          | 1 per 2000 filters                       |
| 3        | Reinforcement Ring Thickness                | 50  | 4                              | $\pm 0.04$ mm   | 0°, 90°, 180°, 270° | 1 per 2000 filters                       |
| 4        | Pressure Drop at 16.7 L/min                 | 50  | 1                              | $\pm 0.30$ cm H <sub>2</sub> O                            | Whole Unit          | 1 per 2000 filters                       |
| 5        | Retention of Particles 0.3 $\mu$ m dia.     | 50  | 1                              | $\pm 1.0\%$   | Whole Unit          | 1 per 2000 filters                       |
| 6        | Number of Filter Container                  | All Containers                              | 1                              | Exact Expected Whole Unit                                 |                     | Upon each completed package.             |
| 7        | Calendar Year ID                            | All Containers                              | 1                              | Exact Expected Whole Unit                                 |                     | Upon each completed package.             |
| 8        | Visual Test for Defective Filters           | 2% (approx. 4000)                           | 6                              | Qualitative Only  | Whole Unit          | 1 per 50 filters                         |
| 9        | Visual Test for Rejected Filters            | 2% (approx. 4000)                           | 6                              | Qualitative Only  | Whole Unit          | 1 per 50 filters                         |
| 10       | Filter Weight Stability and Moisture Pickup | 250   | 3 (triplicate)                 | 2 $\mu$ g   | Whole Unit          | 1 run of 250 Per 20,000                  |
| 11       | Metals Analysis                             | 50  | 1                              | <10%  | Center Of Media     | Beginning of Run (on Whole Media Supply) |
| 12       | Alkalinity                                  | 50  | 1                              | <10% of Result  | Whole Filter        | Beginning of Run (on Whole Media Supply) |

## 13) Visual Test for Filters used in Test 8 and Test 9 in Table I and Table II.

The following descriptions of visual defects are to be used in the acceptance inspection of the filters. Each filter is inspected, using a light screen or table or by viewing over a black surface, for the following defects:

- (a) Pinhole – a small hole appearing (1) as a distinct and obvious bright point of light when examined over a light table or screen, or (2) as a dark spot when viewed over a black surface. Such a filter is considered a reject or unusable filter.
- (b) Separation of ring – any separation or lack of seal between the filter and reinforcing ring. Such a filter is considered a reject filter.

- (c) Chaff or flashing – any extra attached residual material on the ring or heat seal area which would prevent obtaining an airtight seal when the ring is placed under compression. Such a filter is considered a reject filter.
- (d) Loose material – any extra loose material or dirt particles on the filter which would require removal by brushing prior to weighing. A filter with such a defect is considered a defective filter. Defective filters are considered usable.
- (e) Discoloration – any obvious visible discoloration which might be evidence of a contaminant. Such a filter is considered a reject filter.
- (f) Filter non-uniformity – any obvious visible non-uniformity in the appearance of the filter when viewed over a light table or black surface which might indicate graduation in porosity across the face of the filter. A filter with such a defect is considered defective.
- (g) Other – a filter with any imperfections not described above, such as irregular surfaces or other results of poor workmanship, may be considered defective.
- 14) Requirements for trace metal analyses on 46.2 mm teflon filters:
- a. 50 filters from each annual shipment will be analyzed by X-ray fluorescence (XRF) for trace metals. These filters will be selected from the lot of filters by a random selection process, prior to any identification marking. After sample selection, the filters will be processed for printing and packaging, before being submitted for analysis.
- b. The criteria for acceptance is that 95 percent (or 48 of the 50 filters) shall meet the acceptance criteria in the Statement of Work. That is, the average concentrations for the total of the 30 trace metals in 95 percent of the filters shall be less than the stated concentrations (which happens to be twice the minimum detectable concentrations). This will allow the acceptance of the filters with some margin of error. It is intended that this acceptance is identical to that of the EPA.
- c. The rejection of the filters and trigger of root cause analysis procedure will occur if the if any trace metal is ten times the minimum. It is intended that this acceptance is identical to that of the EPA.
- d. The full list of metals, as well as the concentrations, that will be tested are:

Maximum Concentration (ng/cm<sup>2</sup>) for  
Teflon Filters for XRF Analysis

| <u>Element</u> | <u>Concentration</u> |
|----------------|----------------------|
| Al             | 94.4                 |
| Si             | 32.8                 |
| P              | 22.6                 |
| S              | 13.4                 |
| Cl             | 9.4                  |
| K              | 5.6                  |
| Ca             | 8.2                  |
| Sc             | 7.2                  |
| Ti             | 13.8                 |
| V              | 4.8                  |
| Cr             | 2.2                  |

|    |      |
|----|------|
| Mn | 2.2  |
| Fe | 5.8  |
| Co | 4.0  |
| Ni | 3.0  |
| Cu | 2.8  |
| Zn | 2.2  |
| Ga | 1.8  |
| Ge | 3.0  |
| As | 2.8  |
| Se | 1.6  |
| Br | 2.0  |
| Rb | 2.0  |
| Sr | 2.2  |
| Y  | 14.6 |
| Zr | 13.2 |
| Mo | 11.6 |
| Rh | 9.4  |
| Pd | 9.6  |
| Ag | 9.6  |
| Cd | 10.8 |
| Sn | 15.2 |
| Sb | 14.4 |
| Te | 16.2 |
| I  | 18.6 |
| Cs | 25.0 |
| Ba | 32.2 |
| La | 87.6 |
| W  | 5.0  |
| Au | 4.4  |
| Hg | 4.4  |
| Pb | 4.8  |

- 15) Comparability and Sensitivity Requirements. In each case the measured parameter has been analyzed for required measurement performance and an appropriate instrument and process has been selected according to:
- An expanded uncertainty ( $k=2$ , 95% confidence) which is less than 25% of the specification requirement. (4:1, Tolerance: Uncertainty)
  - Equipment and standards which are Traceable to the SI units, and where applicable National Measurement Institutes (i.e. NIST).
  - If traceability is not readily available, (i.e. Filter Efficiency), methods and equipment used or stated in ASTM methods will be adopted.
  - With consideration of points a,b,and c, no comparability between methods will be conducted.
  - With consideration of comparability within the selected method, the following will be completed:
    - For pass/fail measurement, i.e. no numeric result, a running total of percentage pass will be maintained.
    - For methods that generate a numeric result, standard deviations will be calculated on the number measured.
    - The standard deviation will be analyzed with respect to historical data, when available, via a statistical test (f-test). This test will determine if the variation falls within expected variation considering the degrees of freedom in the sample.

- f. Sensitivity of instrumentation is selected within the constraints of the 4:1 uncertainty ratio. Uncertainties are calculated to include  $\frac{1}{2}$  the resolution of the measurement equipment or the process standard deviation whichever is greater as a parameter in the budget.

## A7.2 Quality Performance Requirements

Quality performance requirements for these performance measures are as follows:

- 1) Conformance to all specifications 1 through 15 measured on a randomly selected sample of filters before submission of qualification filters. (Prior to full Batch Production Run). Report to be submitted.
- 2) Conformance to all specifications 1 through 15 measured on a randomly selected sample of the batch (approximately 240,000) filters. Report to be submitted.
- 3) Conformance is achieved if the maximum number of rejects allowed for all physical characteristics combined does not exceed 6%. No defect is permitted for filter identification, and the maximum number allowed for the visual is 10% for defective filters and 5% for the rejected filters.
- 4) Serial Codes on Filters and Permanency of Digits.  
Serial code ranges are programmed into an electronic database and incremented automatically as each filter is printed. Prior to printing, each new code is checked automatically against the electronic database of printed codes. If a duplicate is identified, the printing run is stopped and manual intervention is required.
  - (a) Serial codes for the entire batch are printed in order. The first and last filter in each package of 25 are checked against the package label immediately after the 25 filters are printed and then placed in the package.
  - (b) Filters rejected post-printing. These serial codes are reprinted in order to deliver continuity in the established range. Rejected filters, along with the associated package of 50, are placed in a labeled plastic bag/container for reprinting.

## A7.3 Environmental Controls

The manufacturing room environmental room has controls to ensure successful production and testing of the filters. The controls specifically required and implemented are:

- 1) HEPA filtration throughout (efficient at 99.97% of airborne particles 0.3 micrometers ( $\mu\text{m}$ ) in diameter) Particle counts are recorded daily. Production is stopped when counts exceed 2500 ppm ( $.3 \mu\text{m}$ ) and resumed 1 hour after regaining control of particle count.
- 2) Clean-room Occupant Hygiene. During manufacturing, the following are required of all occupants: hair nets, latex gloves, lab coats, booties, use of tacky mats.
- 3) Static control. Finished filters and empty packages are exposed to a Polonium 210 alpha particle field for static discharge (30-60 seconds).
- 4) Temperature Control:  $21.5^{\circ}\text{C} \pm 1^{\circ}\text{C}$
- 5) Humidity Control:  $35\% \pm 5\%$  RH per 24 hrs.
- 6) Temperature Control:  $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$  for Other Operations
- 7) Humidity Control: 30% to 60% RH per 24 hours for Other Operations.
- 8) Airflow velocity at weighing location  $< 0.04 \text{ m/s}$
- 9) Tooling and work surfaces are routinely cleaned with alcohol wipes to remove contamination.
- 10) If tooling and work surfaces are adverse to solvent cleaning, dry compressed dry air will be used.
- 11) Dust Contamination during Packaging. Packaging is cleaned with compressed air and opened only in the manufacturing clean room environment.

**A7.4 Instrumentation**

The instrumentation required for each of the test methods are detailed below.

Table III. Equipment Identification, Calibration, and Traceability

| Equipment No. | Filter Characteristic  | Equipment           | Model                           | Measured Unit (sensitivity) | Calibration & Scheduled Calibration Interval  | Traceability   |
|---------------|--|---------------------|---------------------------------|-----------------------------|---|--|
| 1             | Reinforcing Ring, Outside Diameter   | Custom Fixture      | Not Applicable                  | ±0.15 mm                    | Calibrated Caliper (24 month Calibration Interval)  | NVLAP Accredited Lab (to NIST)                                   |
| 2             | Reinforcement Ring Width   | Custom Fixture      | Not Applicable                  | ±0.15 mm                    | Calibrated Caliper (24 month Calibration Interval)  | NVLAP Accredited Lab (to NIST)                                   |
| 3             | Reinforcement Ring Thickness   | Micrometer          | SPI 13-462-7 0-1"               | ±0.004 mm                   | Gauge Blocks (24 month Calibration Interval)  | NVLAP Accredited Lab (to NIST)                                   |
| 4             | Pressure Drop at 16.7 L/min  | Pressure Gauge      | ASTM 2986a Apparatus            | 0.02 cm H <sub>2</sub> O    | Transfer Standard   | Reference Filter Paper   |
| 5             | Retention of Particles 0.3 µm dia.   | Per ASTM D2986-91   | See ASTM Standard               | 0.1%                        | Whole Unit  | Reference Filter Paper   |
| 6             | Visual Test for Defective & Rejected Filters   | Light Box           | LP A45-B                        | Qualitative Only            | Not Applicable  | Not Applicable   |
| 7             | Environmental Monitoring for Laboratory 200 (used for clean room production and weighing laboratory) | Vaisala             | PTU300                          | 0.1°C<br>0.1%RH             | Transfer/ Comparison Method (24 month Calibration Interval)                               | NVLAP Accredited Lab (to NIST)                                   |
| 8             | Filter Weight Stability  | Ultra Micro Balance | Mettler Toledo UMX2, UMX5, XP2U | 0.1 µg                      | Substitution Weighing vs. Traceable Mass Std (12 month Calibration Interval) (see Note 1) | NVLAP Accredited Lab (to NIST) for calibration of Mass Standards |

Note 1: The calibration procedure for the Ultra Micro Balance(s) is via direct comparison to calibrated mass standards. Each mass measurement of a filter is determined from the difference from the traceable stainless steel mass standard. Each traceable mass standard is calibrated annually at a NVLAP (NIST Entity) Accredited Laboratory and has an uncertainty of <0.5 µg (k=2). This robust method greatly reduces the dependence upon the linearity and sensitivity errors in the design of the balance as well as the temperature dependence in the force compensation cell. All comparisons are completed using robotic automated weighing to reduce the variability caused by technician loading. The quality assurance protocol

in the automated weighing system triggers an internal calibration of the balance each 60 minutes of operation to ensure sensitivity uniformity. During this protocol external check standards of both filter and mass standard forms are recorded.

**A8. Special Training/Certification (Not required for a Category IV QAPP)**

**A9. Documents and Records (Not required for a Category IV QAPP)**

**B DATA GENERATION AND ACQUISITION**

**B1. Sampling Process Design (Experimental Design)**

The sampling process and selection of filters for test will include the following criteria:

- 1) The selection of filters will be sampled selected randomly.
- 2) When a test is destructive in nature, all selected filters will be replaced to keep numbering sequence in place.
- 3) If multiple lots are used to achieve a Batch (1 years supply, approximately 240,000 filters), each lot will be tested to conform to the detailed performance measures listed in A7.
- 4) If any Filters are marketed, identified, or advertised as compliant with the PM2.5 Filter program, the sampling process and testing protocol listed in this document will be followed.
- 5) Sampling Schedule is to be followed as noted in Table II.

**B2. Sampling Methods (Not required for a Category IV QAPP)**

**B3. Sample Handling and Custody (Not required for a Category IV QAPP)**

**B4. Analytical Methods**

All analytical methods are reference in Section E, Appendices.

This project will follow well-recognized statistical analytical methods for survey samples and uncertainty analysis and calculation.

**B5. Quality Control**

This project will undertake the following specific steps to measure/estimate the effect of data errors, consistent with MTL's Quality Management Plan.

To avert care with numbering anomalies, a database of available serial numbers is used in the printing process. Once a number is printed, it is removed from the database and not available for use. This protocol eliminates the potential of duplicate numbering. Consecutive numbering is assured by process counts on the printing apparatus, and incorporation of the packaging process directly from the printing protocol. In the event of the necessity of a reprint, this is done with manual intervention and subject to redundant checks.

**B5.1 Crosschecking data**

Data collection will be designed in such a way to allow internal crosschecking of data by comparing all technician results. Crosschecking will be automatically conducted via spreadsheet templates and graphical data analysis for electronically entered data. Further, analysis on submitted reports will be used to check for data entry errors or possible biases in technician methods.

## B5.2 Data anomalies

Procedures for handling data anomalies (such as outliers and missing data) will be handled based on guidance prepared in the project-specific statistical methodology. A student's t-test or E normal test will be used to determine anomalies. Response to results outside of acceptance or advisory limits are handled as follows:

- 1) Advisory Limit (deviation more than 75% allowable specification)
  - a. Technician observing the limit violation will inform Quality Manager who will initiate a root cause analysis.
  - b. Quality Manager will involve CTO in root cause analysis.
  - c. Quality assurance measurements will be increased per decision by CTO.
- 2) Acceptance Limit (deviation more than the specification limit)
  - a. Technician observing the limit violation will inform Quality Manager who will initiate a root cause analysis.
  - b. Quality Manager will halt all related processes.
  - c. Quality Manager will involve CTO in root cause analysis.
  - d. Quality assurance measurements will be increased per decision by CTO.
  - e. Root Cause Analysis finding will be put into action by QM, CTO, and COO.
  - f. Upon satisfaction of determination of root cause analysis, and signature of QM, CTO, and COO, production will resume.

## B5.3 Quality control statistics

The quality control statistics to be used in this project and their reporting are as follows:

- 1) Before any weighing data is collected, the ultra microbalance (0.1  $\mu\text{g}$  resolution) must be:
  - a. Calibrated internally and have traceable calibrated standards with sufficiently small uncertainties. (i.e.  $<0.5 \mu\text{g}$ , at  $k=2$ )
  - b. Precision Tested with a metal artifact and found to be operating with better than a  $0.25 \mu\text{g}$  standard deviation on 10 repetitive weighings.
- 2) During each weighing session, a metal check standard will be weighed to verify traceability within expected deviations. The  $E_{\text{normal}}$  test (form of a t-test) will be employed.
- 3) For all quantitative data generated in section A7 will be graphically plotted with linear regression analysis employed to detect drift.
- 4) All records and reports will be electronic and stored in a central backed up server (E:\EPA Filters\QC\Data, or in a location defined by the Quality Manager. Project documents, records and reports will include:
  - a. Precision Tests of Methods
  - b. Check Standard Tests (with  $E_n$  results)
  - c. Pass / Fail Ratios on qualitative tests
  - d. Calibration Reports on Equipment
  - e. Quality Control Results for each parameter in Table I.
- 5) All Filter Weighing System reports will be retrievable and reviewed weekly by the Quality Manager.

## B6. Instrument/Equipment Testing, Inspection and Maintenance (Not required for a Category IV QAPP)

**B7. Instrument/Equipment Calibration and Frequency (Not required for a Category IV QAPP)****B8. Inspection/Acceptance for Supplies and Consumables (Not required for a Category IV QAPP)****B9. Data Acquisition Requirements for Non-Direct Measurements (I.e., Secondary Data)****B10. Data Management (Not required for a Category IV QAPP)****C ASSESSMENT/OVERSIGHT****C1. Assessment and Response Actions**

Assessment tools include the following actions:

- 1) Management System Review – A management system review of this QAPP and all other related system documents will be conducted annually by the Quality Team (QM, COO, CTO). The schedule is as follows:
  - a. Completion by end of 1<sup>st</sup> quarter:
    - i. QAPP Sections A
  - b. Completion by end of 2<sup>nd</sup> quarter:
    - i. QAPP Section B
  - c. Completion by end of 3<sup>rd</sup> quarter:
    - i. QAPP Section C
- 2) Each Management System Review will generate a report of statistical success rate when measureable. This report will include:
  - a. Any corrective actions
  - b. A summary of the review, including success ratio (1 – (findings/# of requirements))
- 3) Technical System Audit, Completion by end of 4<sup>th</sup> quarter by CTO with support from QM:
  - i. Technical Data Method Review (Appendices)
  - ii. Review of Statistical QC Reports
- 4) The Quality Team has the authority to issue stop work orders upon any findings that raise concerns regarding the conformance of the product to any of the stated specifications within the QAPP.
- 5) Corrective actions and responses will be taken in accordance with Section B5.2 and documented in the Management Review.
- 6) Each Quarter, a Summary Report will be written by the QM.

**C2. Reports to Management**

The frequency and distribution of reports issued by the QM are:

- 1) A quarterly Summary Report covering topics per the schedule identified in C1, will be issued:
  - a. To the CEO.
  - b. Copied to the COO, CTO, and QM.
- 2) As needed to relay any findings or urgent action items, the QM will issue upon occurrence:
  - a. A corrective actions report to the COO, CTO, and CEO.
  - b. A stop work order report to the COO, CTO, and CEO.
- 3) An annual quality assurance improvement report for previous year by end of 1<sup>st</sup> quarter. The report is to include:

- a. Opportunities for process improvement.
- b. Significant advances in Quality Control or Quality Assurance
- c. Recommendations

**D DATA REVIEW AND EVALUATION**

**D1. Data Review, Verification and Validation (Not required for a Category IV QAPP)**

**D2. Verification and Validation of Methods (Not required for a Category IV QAPP)**

**D3. Evaluating Data in Terms of User Needs (Not required for a Category IV QAPP)**

**E ADDENDUMS**

**E1. Key Facility / Clean Room Requirements & Procedures**

**ISO  
14644-1**

**INTERNATIONAL  
STANDARD**

First edition  
1999-05-01

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**Cleanrooms and associated controlled environments —**

**Part 1:**  
**Classification of air cleanliness**

Salles propres et environnements maîtrisés apparentés —  
Partie 1: Classification de la propreté de l'air



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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and nongovernmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75% of the member bodies casting a vote.

International Standard ISO 14644-1 was prepared by Technical Committee ISO/TC 209, Cleanrooms and associated controlled environments.

ISO 14644 consists of the following parts, under the general title Cleanrooms and associated controlled environments:

- Part 1: Classification of air cleanliness
- Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1
- Part 3: Metrology and test methods
- Part 4: Design, construction and start-up
- Part 5: Operations
- Part 6: Terms and definitions
- Part 7: Enhanced clean devices

Users should note that the titles listed for parts 2 to 7 are working titles at the time of the release of part 1. In the event that one or more of these parts are deleted from the work programme, the remaining parts may be renumbered.

Annexes B and C form an integral part of this part of ISO 14644. Annexes A, D, E, and F are for information only.

## Introduction

Cleanrooms and associated controlled environments provide for the control of airborne particulate contamination to levels appropriate for accomplishing contamination-sensitive activities. Products and processes that benefit from the control of airborne contamination include those in such industries as aerospace, microelectronics, pharmaceuticals, medical devices, food, and healthcare.

This part of ISO 14644 assigns ISO classification levels to be used for the specification of air cleanliness in cleanrooms and associated controlled environments. It also prescribes the standard method of testing as well as the procedure for determining the concentration of airborne particles.

For classification purposes, this part of ISO 14644 is limited to a designated range of considered particle sizes for determination of particle concentration limits. This part of ISO 14644 also provides standard protocols for the determination and designation of cleanliness levels that are based on airborne concentrations of particles smaller or larger than the size range designated for classification.

This part of ISO 14644 is one of a series of standards concerned with cleanrooms and contamination control. Many factors besides airborne particulate cleanliness must be considered in the design, specification, operation, and control of cleanrooms and other controlled environments. These are covered in some detail in other parts of the International Standards prepared by ISO/TC 209.

In some circumstances, relevant regulatory agencies may impose supplementary policies or restrictions. In such situations, appropriate adaptations of the standard testing procedures may be required.

# Cleanrooms and associated controlled environments —

## Part 1: Classification of air cleanliness

### 1 Scope

This part of ISO 14644 covers the classification of air cleanliness in cleanrooms and associated controlled environments exclusively in terms of concentration of airborne particles. Only particle populations having cumulative distributions based on threshold (lower limit) sizes ranging from 0,1  $\mu\text{m}$  to 5  $\mu\text{m}$  are considered for classification purposes.

This part of ISO 14644 does not provide for classification of particle populations that are outside of the specified particle size range, 0,1  $\mu\text{m}$  to 5  $\mu\text{m}$ . Concentrations of ultrafine particles (particles smaller than 0,1  $\mu\text{m}$ ) and macroparticles (particles larger than 5  $\mu\text{m}$ ) may be used to quantify these populations in terms of U descriptors and M descriptors, respectively.

This part of ISO 14644 cannot be used to characterize the physical, chemical, radiological, or viable nature of airborne particles.

NOTE The actual distribution of particle concentrations within incremental size ranges normally is not predictable and typically is variable over time.

### 2 Definitions

For the purposes of this part of ISO 14644, the following definitions apply.

#### 2.1 General

##### 2.1.1

**cleanroom**

room in which the concentration of airborne particles is controlled, and which is constructed and used in a manner to minimize the introduction, generation, and retention of parti-

cles inside the room, and in which other relevant parameters, e.g. temperature, humidity, and pressure, are controlled as necessary

##### 2.1.2

**clean zone**

dedicated space in which the concentration of airborne particles is controlled, and which is constructed and used in a manner to minimize the introduction, generation, and retention of particles inside the zone, and in which other relevant parameters, e.g. temperature, humidity, and pressure, are controlled as necessary

NOTE This zone may be open or enclosed and may or may not be located within a cleanroom.

##### 2.1.3

**installation**

cleanroom or one or more clean zones, together with all associated structures, air-treatment systems, services, and utilities

##### 2.1.4

**classification**

level (or the process of specifying or determining the level) of airborne particulate cleanliness applicable to a cleanroom or clean zone, expressed in terms of an ISO Class N, which represents maximum allowable concentrations (in particles per cubic metre of air) for considered sizes of particles

NOTE 1 The concentrations are determined by using equation (1) in 3.2.

NOTE 2 Classification in accordance with this International Standard is limited to the range extending from ISO Class 1 through ISO Class 9.

NOTE 3 The considered particle sizes (lower threshold values) applicable for classification in accordance with this International

Standard are limited to the range from 0,1  $\mu\text{m}$  through 5  $\mu\text{m}$ . Air cleanliness may be described and specified (but not classified) in terms of U descriptors or M descriptors (see 2.3.1 or 2.3.2) for considered threshold particle sizes that are outside of the range covered by classification.

NOTE 4 Intermediate ISO classification numbers may be specified, with 0,1 the smallest permitted increment; i.e., the range of intermediate ISO classes extends from ISO Class 1,1 through ISO Class 8,9.

NOTE 5 Classification may be specified or accomplished in any of three occupancy states (see 2.4).

## 2.2 Airborne particles

### 2.2.1

#### particle

solid or liquid object which, for purposes of classification of air cleanliness, falls within a cumulative distribution that is based upon a threshold (lower limit) size in the range from 0,1  $\mu\text{m}$  to 5  $\mu\text{m}$

### 2.2.2

#### particle size

diameter of a sphere that produces a response, by a given particle-sizing instrument, that is equivalent to the response produced by the particle being measured

NOTE For discrete-particle-counting, light-scattering instruments, the equivalent optical diameter is used.

### 2.2.3

#### particle concentration

number of individual particles per unit volume of air

### 2.2.4

#### particle size distribution

cumulative distribution of particle concentration as a function of particle size

### 2.2.5

#### ultrafine particle

particle with an equivalent diameter less than 0,1  $\mu\text{m}$

### 2.2.6

#### macroparticle

particle with an equivalent diameter greater than 5  $\mu\text{m}$

### 2.2.7

#### fibre

particle having an aspect (length-to-width) ratio of 10 or more

## 2.3 Descriptors

### 2.3.1

#### U descriptor

measured or specified concentration, of particles per cubic metre of air, including the ultrafine particles

NOTE The U descriptor may be regarded as an upper limit for the averages at sampling locations (or as an upper confidence limit, depending upon the number of sampling locations used to characterize the cleanroom or clean zone). U descriptors cannot be used to define airborne particulate cleanliness classes, but they may be quoted independently or in conjunction with airborne particulate cleanliness classes.

### 2.3.2

#### M descriptor

measured or specified concentration of macroparticles per cubic metre of air, expressed in terms of the equivalent diameter that is characteristic of the measurement method used

NOTE The M descriptor may be regarded as an upper limit for the averages at sampling locations (or as an upper confidence limit, depending upon the number of sampling locations used to characterize the cleanroom or clean zone). M descriptors cannot be used to define airborne particulate cleanliness classes, but they may be quoted independently or in conjunction with airborne particulate cleanliness classes.

## 2.4 Occupancy states

### 2.4.1

#### as-built

condition where the installation is complete with all services connected and functioning but with no production equipment, materials, or personnel present

### 2.4.2

#### at-rest

condition where the installation is complete with equipment installed and operating in a manner agreed upon by the customer and supplier, but with no personnel present

### 2.4.3

#### operational

condition where the installation is functioning in the specified manner, with the specified number of personnel present and working in the manner agreed upon

**Table 1 — Selected airborne particulate cleanliness classes for cleanrooms and clean zones**

| ISO classification number (N) | Maximum concentration limits (particles/m <sup>3</sup> of air) for particles equal to and larger than the considered sizes shown below (concentration limits are calculated in accordance with equation (1) in 3.2) |         |         |            |           |         |
|-------------------------------|---|---------|---------|------------|-----------|---------|
|                               | 0,1 m   | 0,2 m   | 0,3 m   | 0,5 m      | 1 m       | 5 m     |
| ISO Class 1                   | 10  | 2       |         |            |           |         |
| ISO Class 2                   | 100   | 24      | 10      | 4          |           |         |
| ISO Class 3                   | 1 000   | 237     | 102     | 35         | 8         |         |
| ISO Class 4                   | 10 000  | 2 370   | 1 020   | 352        | 83        |         |
| ISO Class 5                   | 100 000   | 23 700  | 10 200  | 3 520      | 832       | 29      |
| ISO Class 6                   | 1 000 000   | 237 000 | 102 000 | 35 200     | 8 320     | 293     |
| ISO Class 7                   |   |         |         | 352 000    | 83 200    | 2 930   |
| ISO Class 8                   |   |         |         | 3 520 000  | 832 000   | 29 300  |
| ISO Class 9                   |   |         |         | 35 200 000 | 8 320 000 | 293 000 |

NOTE Uncertainties related to the measurement process require that concentration data with no more than three significant figures be used in determining the classification level

## 2.5 Roles

### 2.5.1

#### customer

organization, or the agent thereof, responsible for specifying the requirements of a cleanroom or clean zone

### 2.5.2

#### supplier

organization engaged to satisfy the specified requirements of a cleanroom or clean zone

## 3 Classification

### 3.1 Occupancy state(s)

The particulate cleanliness of air in a cleanroom or clean zone shall be defined in one or more of three occupancy states, viz. “as-built”, “at-rest”, or “operational” (see 2.4).

NOTE It should be recognized that the “as-built” state is applicable to newly completed or newly modified cleanrooms or clean zones. Once testing in the “as-built” state is completed, further testing for compliance will be performed in the “at-rest” or the “operational” state, or both.

### 3.2 Classification number

Airborne particulate cleanliness shall be designated by a classification number, N. The maximum permitted concentration of particles,  $C_n$ , for each considered particle size, D, is determined from the equation:

$$C_n = 10^N \frac{0,1}{D}^{2,08} \quad (1)$$

where

$C_n$  is the maximum permitted concentration (in particles per cubic metre of air) of airborne particles that are equal to or larger than the considered particle size.  $C_n$  is rounded to the nearest whole number, using no more than three significant figures.

N is the ISO classification number, which shall not exceed a value of 9. Intermediate ISO classification numbers may be specified, with 0,1 the smallest permitted increment of N.

D is the considered particle size, in micrometres.

0,1 is a constant, with a dimension of micrometres.

Table 1 presents selected airborne particulate cleanliness classes and the corresponding particle concentrations for particles equal to and larger than the considered sizes shown. Figure A.1 (see annex A) provides a representation of the

selected classes in graphical form. In case of dispute, the concentration  $C_n$  as derived from equation (1) shall serve as the standard value.

### 3.3 Designation

The designation of airborne particulate cleanliness for cleanrooms and clean zones shall include:

- the classification number, expressed as "ISO Class N";
- the occupancy state to which the classification applies;
- the considered particle size(s), and the related concentration(s), as determined by the classification equation (1) where each considered threshold particle size is in the range from 0,1  $\mu\text{m}$  through 5  $\mu\text{m}$ .

Example designation:

ISO Class 4; operational state; considered sizes:

0,2  $\mu\text{m}$  (2 370 particles/ $\text{m}^3$ ), 1  $\mu\text{m}$  (83 particles/ $\text{m}^3$ )

The considered particle size(s) for which the concentration(s) will be measured shall be agreed upon by the customer and the supplier.

If measurements are to be made at more than one considered particle size, each larger particle diameter (e.g.,  $D_2$ ) shall be at least 1,5 times the next smaller particle diameter (e.g.,  $D_1$ ).

e.g.:  $D_2 \geq 1,5 \times D_1$

## 4 Demonstration of compliance

### 4.1 Principle

Compliance with air cleanliness (ISO class) requirements specified by the customer is verified by performing specified testing procedures and by providing specified documentation of the results and conditions of testing, as agreed upon by the customer and the supplier.

### 4.2 Testing

The reference test method for demonstrating compliance is given in annex B. An alternative method having comparable accuracy may be specified, although if no method is specified or agreed upon, the reference method shall be used.

Tests performed to demonstrate compliance shall be conducted using calibrated instruments.

### 4.3 Airborne particle concentration limits

Upon completion of testing in accordance with 4.2, average particle concentrations and the 95% upper confidence limit

(when applicable) shall be calculated using equations shown in annex C.

Average particle concentration(s), calculated in accordance with equation (C.1), shall not exceed the concentration limit(s) determined by use of equation (1) in 3.2, as specified [3.3 c)] for the considered size(s).

In addition, for situations in which the number of sampling locations involved is at least two but not more than nine, the calculation of 95% upper confidence limits in accordance with C.3 shall not exceed the concentration limits established above.

NOTE Worked examples of classification calculations are provided in annex D.

Particle concentrations used for determination of conformance to classification limits shall be measured by the same method for all considered particle sizes.

### 4.4 Test report

The results from testing each cleanroom or clean zone shall be recorded and submitted as a comprehensive report, along with a statement of compliance or noncompliance with the specified designation of airborne particulate cleanliness classification.

The test report shall include the following:

- the name and address of the testing organization, and the date on which the test was performed;
- the number and year of publication of this part of ISO 14644, i.e., ISO 14644-1: date of current issue;
- a clear identification of the physical location of the cleanroom or clean zone tested (including reference to adjacent areas if necessary), and specific designations for coordinates of all sampling locations;
- the specified designation criteria for the cleanroom or clean zone, including the ISO classification, the relevant occupancy state(s), and the considered particle size(s);
- details of the test method used, with any special conditions relating to the test or departures from the test method, and identification of the test instrument and its current calibration certificate;
- the test results, including particle concentration data for all sampling location coordinates.

NOTE If concentrations of ultrafine particles or macroparticles are quantified, as described in annex E, the pertinent information should be included with the test report.

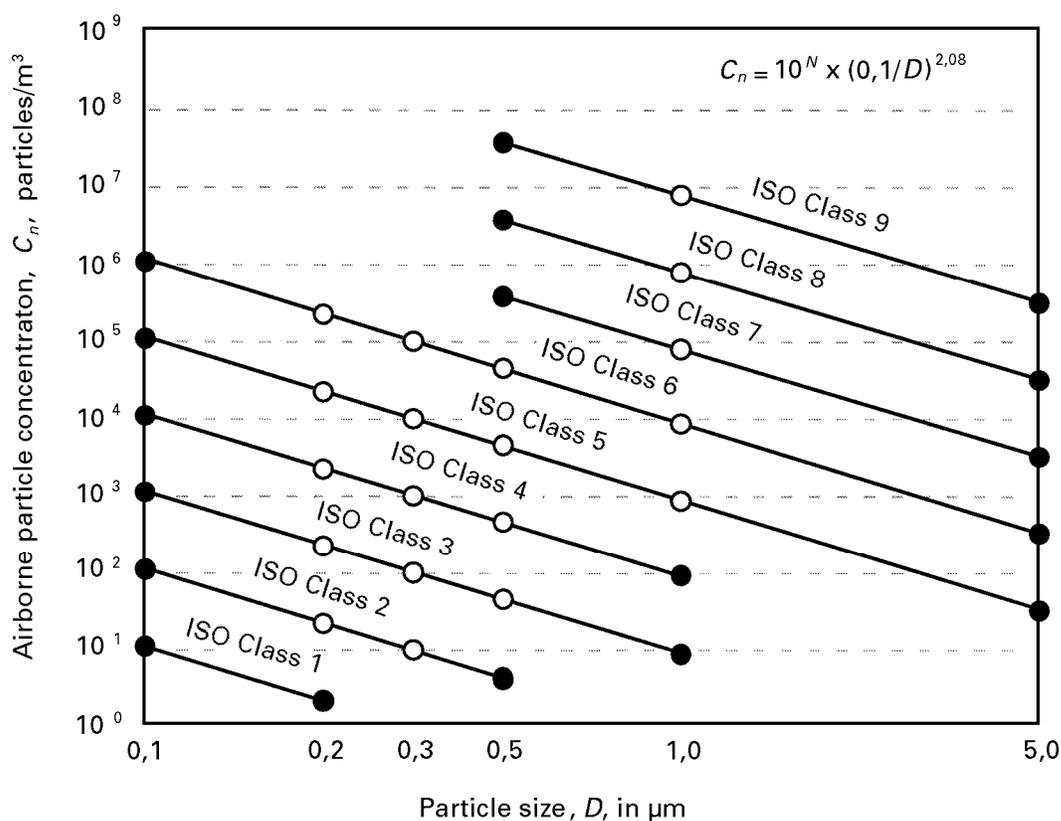
## Annex A (informative)

### Graphical illustration of the classes of Table 1

Figure A.1 depicts the air cleanliness classes of Table 1 in graphical form, for illustration purposes only. The ISO classes of Table 1 are shown as lines representing the class concentration limits for the considered threshold particle sizes. They are based on calculations using equation (1) of 3.2. As the lines only approximate the class limits, they are not to be used to define the limits. Such determinations are made in accordance with equation (1).

The classification lines shown on the graph may not be extrapolated beyond the solid circle symbols, which indicate the minimum and maximum particle size limits acceptable for each of the ISO classes shown.

The classification lines do not represent actual particle size distributions found in cleanrooms and clean zones.



**Figure A.1 — Graphical representation of ISO-class concentration limits for selected ISO classes**

NOTE 1  $C_n$  represents the maximum permitted concentration (in particles per cubic metre of air) of airborne particles equal to and larger than the considered particle size.

NOTE 2  $N$  represents the specified ISO class number.

## Annex B (normative)

### Determination of particulate cleanliness classification using a discrete-particle-counting, light-scattering instrument

#### B.1 Principle

A discrete-particle-counting, light-scattering instrument is used to determine the concentration of airborne particles, equal to and larger than the specified sizes, at designated sampling locations.

#### B.2 Apparatus requirements

##### B.2.1 Particle-counting instrument

Discrete-particle counter (DPC), a light-scattering device having a means of displaying or recording the count and size of discrete particles in air with a size discrimination capability to detect the total particle concentration in the appropriate particle size ranges for the class under consideration, and a suitable sampling system.

##### B.2.2 Instrument calibration

The instrument shall have a valid calibration certificate; the frequency and method of calibration should be based on current accepted practice.

#### B.3 Pretest conditions

##### B.3.1 Preparation for testing

Prior to testing, verify that all aspects of the cleanroom or clean zone that contribute to its operational integrity are complete and functioning in accordance with its performance specification.

Such pretesting may include, for example:

- a) airflow volume or velocity tests;
- b) air pressure difference test;
- c) containment leakage test;
- d) installed filter leakage test.

##### B.3.2 Pretest equipment setup

Perform equipment setup and pretest calibration of the instrument in accordance with the manufacturer's instructions.

#### B.4 Sampling

##### B.4.1 Establishment of sampling locations

###### B.4.1.1

Derive the minimum number of sampling point locations from equation (B.1):

$$N_L = \sqrt{A} \quad (\text{B.1})$$

where

$N_L$  is the minimum number of sampling locations (rounded up to a whole number).

$A$  is the area of the cleanroom or clean zone in square metres.

NOTE In the case of unidirectional horizontal airflow, the area  $A$  may be considered as the cross section of the moving air perpendicular to the direction of the airflow.

###### B.4.1.2

Ensure that the sampling locations are evenly distributed throughout the area of the cleanroom or clean zone and positioned at the height of the work activity.

If the customer specifies additional sampling locations, their number and positions shall also be specified.

NOTE Such additional locations may be those considered critical, based on a risk analysis.

##### B.4.2 Establishment of single sample volume per location

###### B.4.2.1

At each sampling location, sample a sufficient volume of air that a minimum of 20 particles would be detected if the particle concentration for the largest considered particle size were at the class limit for the designated ISO class.

The single sample volume  $V_s$  per location is determined by using equation (B.2):

$$V_s = \frac{20}{C_{n,m}} \quad 1\,000 \quad (\text{B.2})$$

where

$V_s$  is the minimum single sample volume per location, expressed in litres (except see B.4.2.2).

$C_{n,m}$  is the class limit (number of particles per cubic metre) for the largest considered particle size specified for the relevant class.

20 is the defined number of particles that could be counted if the particle concentration were at the class limit.

NOTE When  $V_s$  is very large, the time required for sampling can be substantial. By using the sequential sampling procedure (see annex F), both the required sample volume and the time required to obtain samples may be reduced.

### B.4.2.2

The volume sampled at each location shall be at least 2 litres, with a minimum sampling time at each location of 1 min.

## B.4.3 Sampling procedure

### B.4.3.1

Set up the particle counter (B.2.1) in accordance with the manufacturer's instructions and in compliance with the instrument calibration certificate.

### B.4.3.2

The sampling probe shall be positioned pointing into the airflow. If the direction of the airflow being sampled is not controlled or predictable (e.g., nonunidirectional airflow), the inlet of the sampling probe shall be directed vertically upward.

### B.4.3.3

Sample the volume of air determined in B.4.2, as a minimum, at each sampling location.

### B.4.3.4

Where only one sampling location is required (B.4.1), take a minimum of three single sample volumes (B.4.2) at that location.

## B.5 Recording of results

### B.5.1 Average concentration of particles at each sampling location

#### B.5.1.1

Record the result of each sample measurement as the concentration of each of the considered particle size(s) (3.3) appropriate to the relevant classification of air cleanliness.

NOTE Consideration should be given to the requirements of B.6.1 before proceeding with the calculation of the 95% upper confidence limit.

#### B.5.1.2

When only one sampling location is used, calculate and record the average value of the sample data (B.4.3.4) for each considered particle size.

#### B.5.1.3

When two or more single sample volumes are taken at a location, compute the average particle concentration for each considered particle size from the individual sample particle concentrations (B.5.1.1), according to the procedure given in C.2, and record the results.

### B.5.2 Requirement for computing the 95% upper confidence limit (UCL)

#### B.5.2.1

When the number of locations sampled is more than one and less than ten, compute the overall mean of the averages, standard deviation, and 95% upper confidence limit from the average particle concentrations for all locations (B.5.1) following the procedure described in C.3.

#### B.5.2.2

When only a single location is sampled, or when more than nine are sampled, computing the 95% upper confidence limit is not applicable.

## B.6 Interpretation of results

### B.6.1 Classification requirements

The cleanroom or clean zone is deemed to have met the specified air cleanliness classification if the averages of the particle concentrations measured at each of the locations and, when applicable, the 95% upper confidence limit calculated according to B.5.2, do not exceed the concentration limits determined in accordance with equation (1) of 3.2.

If the results of testing fail to meet the specified air cleanliness classification, testing may be performed at additional, evenly distributed sampling locations. The results of recalculation, including data from the added locations, shall be definitive.

### B.6.2 Treatment of outliers

The result of the 95% UCL calculation may fail to meet the specified ISO class designation. If the noncompliance is caused by a single, nonrandom "outlier" value resulting from an erroneous measurement (due to procedural error or equipment malfunction) or from an unusually low particle concentration (due to exceptionally clean air), the outlier may be excluded from the calculation, provided that:

- a) the calculation is repeated, including all remaining sampling locations;
- b) at least three measurement values remain in the calculation;

c) no more than one measurement value is excluded from the calculation;

d) the suspected cause of the erroneous measurement or low particle concentration is documented and accepted by both the customer and supplier.

NOTE Widely divergent values for particle concentrations among the locations sampled may be reasonable and even intentional, depending on the nature of the application of the clean installation under test.

## Annex C (normative)

### Statistical treatment of particle concentration data

#### C.1 Rationale

This statistical analysis considers only random errors (lack of precision), not errors of a nonrandom nature (e.g. bias associated with erroneous calibration).

#### C.2 Algorithm for computation of average particle concentration at a location ( $\bar{x}_i$ )

When multiple samples are taken at a location, equation (C.1) shall be used to determine the average particle concentration at the location. Calculation of the average particle concentration shall be performed for each sampling location at which two or more samples have been taken.

$$\bar{x}_i = \frac{x_{i,1} + x_{i,2} + \dots + x_{i,n}}{n} \quad (C.1)$$

where

$\bar{x}_i$  is the average particle concentration at location  $i$ , representing any location.

$x_{i,1}$  to  $x_{i,n}$  are the particle concentrations of the individual samples.

$n$  is the number of samples taken at location  $i$ .

#### C.3 Algorithms for computation of 95% upper confidence limit

##### C.3.1 Principle

This procedure is applicable only if the number of sampling locations is more than one and less than ten. In such circumstances, this procedure shall be used in addition to the algorithm of equation (C.1).

##### C.3.2 Overall mean of the averages ( $\bar{\bar{x}}$ )

Using equation (C.2), determine the overall (grand) mean of the averages.

$$\bar{\bar{x}} = \frac{\bar{x}_{i,1} + \bar{x}_{i,2} + \dots + \bar{x}_{i,m}}{m} \quad (C.2)$$

where

$\bar{\bar{x}}$  is the overall mean of the location averages.

$\bar{x}_{i,1}$  to  $\bar{x}_{i,m}$  are individual location averages, determined by using equation (C.1).

$m$  is the number of individual location averages.

All individual location averages are equally weighted, regardless of the number of samples taken at any given location.

##### C.3.3 Standard deviation of the location averages ( $s$ )

Using equation (C.3), determine the standard deviation of the location averages.

$$s = \sqrt{\frac{\bar{x}_{i,1}^2 - \bar{x}^2 + \bar{x}_{i,2}^2 - \bar{x}^2 + \dots + \bar{x}_{i,m}^2 - \bar{x}^2}{m - 1}} \quad (C.3)$$

where

$s$  is the standard deviation of the location averages.

##### C.3.4 95% upper confidence limit (UCL) for the overall mean

Using equation (C.4), determine the 95% upper confidence limit for the overall mean.

$$95\% \text{ UCL} = \bar{\bar{x}} + t_{0,95} \frac{s}{\sqrt{m}} \quad (C.4)$$

where

$t_{0,95}$  represents the 95th percentile (quantile) of the  $t$  distribution, with  $m-1$  degrees of freedom.

Values of the Student's  $t$  distribution ( $t_{0,95}$ ) for the 95% UCL are given in Table C.1. Alternatively, Student's  $t$  distributions provided in statistical computer programmes are also acceptable.

**Table C.1 — Student's  $t$  distribution for the 95% upper confidence limit**

| Number of individual averages ( $m$ ) | 2   | 3   | 4   | 5   | 6   | 7-9 |
|---------------------------------------|-----|-----|-----|-----|-----|-----|
| $t$                                   | 6,3 | 2,9 | 2,4 | 2,1 | 2,0 | 1,9 |

## Annex D (informative)

### Worked examples of classification calculations

#### D.1 Example 1

##### D.1.1

The cleanroom under consideration has an area (A) of 80 m<sup>2</sup>. Conformance with the specified airborne particulate cleanliness classification is to be determined in the operational state.

The specified air cleanliness classification of the cleanroom is ISO Class 5.

##### D.1.2

Two considered particle sizes are specified: 0,3 μm (D<sub>1</sub>) and 0,5 μm (D<sub>2</sub>).

a) Both particle sizes are within the size limitations for ISO Class 5 [see 3.3 c) and Table 1]: 0,1 μm, 0,3 μm, 0,5 μm, 1 μm, 5 μm.

b) Application of the particle size ratio requirement, D<sub>2</sub> = 1,5 × D<sub>1</sub> [see 3.3 c)], shows compliance: 0,5 μm (1,5 × 0,3 μm = 0,45 μm).

##### D.1.3

The maximum permitted airborne particle concentrations are calculated in accordance with equation (1) (see 3.2).

For particles 0,3 μm (D<sub>1</sub>):

$$C_n = \frac{0,1^{2,08}}{0,3} \cdot 10^5 = 10\,176 \quad (\text{D.1})$$

rounded to 10 200 particles/m<sup>3</sup>

For particles 0,5 μm (D<sub>2</sub>):

$$C_n = \frac{0,1^{2,08}}{0,5} \cdot 10^5 = 3\,517 \quad (\text{D.2})$$

rounded to 3 520 particles/m<sup>3</sup>

##### D.1.4

The number of sampling point locations are derived in accordance with equation (B.1) (see B.4.1.1):

$$N_L = \sqrt{A} = \sqrt{80} = 8,94 \text{ rounded to } 9 \quad (\text{D.3})$$

Therefore the minimum number of sampling locations is nine and, as the number of sampling locations is less than ten, the calculation of the 95% UCL according to annex C is applicable.

##### D.1.5

The single sample volume, V<sub>s</sub>, is calculated in litres in accordance with equation (B.2) (see B.4.2.1):

$$V_s = \frac{20}{C_{n,m}} \cdot 1\,000 = \frac{20}{3\,517} \cdot 1\,000 = 5,69 \text{ litres} \quad (\text{D.4})$$

The result is greater than 2 litres, and the sample volume selected was 28 litres over a period of 1 min (a flow rate commonly available in discrete-particle-counting light-scattering instruments).

This selection was based on:

- a) V<sub>s</sub> > 2 litres (see B.4.2.2)
- b) C<sub>n,m</sub> > 20 particles/m<sup>3</sup> (see B.4.2.1)
- c) Sampling time = 1 min (see B.4.2.2)

##### D.1.6

At each sampling location, only one single sample volume (28 litres) is taken (B.4.2.1). The counts obtained from the measurements are recorded (B.5.1.1) below.

| Sampling location | Number of particles (0,3 μm) | Number of particles (0,5 μm) |
|-------------------|------------------------------|------------------------------|
| 1                 | 245                          | 21                           |
| 2                 | 185                          | 24                           |
| 3                 | 59                           | 0                            |
| 4                 | 106                          | 7                            |
| 5                 | 164                          | 22                           |
| 6                 | 196                          | 25                           |
| 7                 | 226                          | 23                           |
| 8                 | 224                          | 37                           |
| 9                 | 195                          | 19                           |

**D.1.7**

From the raw data (D.1.6), the number of particles per cubic metre,  $x_i$ , is calculated:

| Sampling location | $x_i$ 0,3 m | $x_i$ 0,5 m |
|-------------------|-------------|-------------|
| 1                 | 8 750       | 750         |
| 2                 | 6 607       | 857         |
| 3                 | 2 107       | 0           |
| 4                 | 3 786       | 250         |
| 5                 | 5 857       | 786         |
| 6                 | 7 000       | 893         |
| 7                 | 8 071       | 821         |
| 8                 | 8 000       | 1 321       |
| 9                 | 6 964       | 679         |

Each calculated concentration value for 0,3 m and 0,5 m is less than the limits established in D.1.3. This satisfies the first part of classification (B.6.1) and therefore calculation of the 95% UCL according to annex C can proceed.

**D.1.8**

Computation of average concentration in accordance with equation (C.1) (see C.2) is not applicable, as the sample volumes taken were single volumes which represent an average particle concentration at each location. The overall means of the averages are calculated in accordance with equation (C.2) (see C.3.2).

For particles 0,3 m:

$$\begin{aligned} \bar{x} &= \frac{1}{9} \begin{matrix} 8\ 750 & 6\ 607 & 2\ 107 & 3\ 786 \\ 5\ 857 & 7\ 000 & 8\ 071 & 8\ 000 \\ 6\ 964 \end{matrix} & \text{(D.5)} \\ &= \frac{1}{9} \times 57\ 142 \\ &= 6\ 349,1 \text{ rounded to } 6\ 349 \text{ particles/m}^3 \end{aligned}$$

For particles 0,5 m:

$$\begin{aligned} \bar{x} &= \frac{1}{9} \begin{matrix} 750 & 857 & 0 & 250 & 786 \\ 893 & 821 & 1\ 321 & 679 \end{matrix} & \text{(D.6)} \\ &= \frac{1}{9} \times 6\ 357 \\ &= 706,3 \text{ rounded to } 706 \text{ particles/m}^3 \end{aligned}$$

**D.1.9**

The standard deviations of the location averages are calculated in accordance with equation (C.3) (see C.3.3).

For particles 0,3 m:

$$\begin{aligned} s^2 &= \frac{1}{8} \begin{matrix} (8\ 750 - 6\ 349)^2 & (6\ 607 - 6\ 349)^2 \\ (2\ 107 - 6\ 349)^2 & (3\ 786 - 6\ 349)^2 \\ (5\ 857 - 6\ 349)^2 & (7\ 000 - 6\ 349)^2 \\ (8\ 071 - 6\ 349)^2 & (8\ 000 - 6\ 349)^2 \\ (6\ 964 - 6\ 349)^2 \end{matrix} & \text{(D.7)} \\ &= \frac{1}{8} \times 37\ 130\ 073 \\ &= 4\ 641\ 259,1 \text{ rounded to } 4\ 641\ 259 \end{aligned}$$

$$\begin{aligned} s &= \sqrt{4\ 641\ 259} & \text{(D.8)} \\ &= 2\ 154,4 \text{ rounded to } 2\ 154 \text{ particles/m}^3 \end{aligned}$$

For particles 0,5 m:

$$\begin{aligned} s^2 &= \frac{1}{8} \begin{matrix} (750 - 706)^2 & (857 - 706)^2 \\ (0 - 706)^2 & (250 - 706)^2 \\ (786 - 706)^2 & (893 - 706)^2 \\ (821 - 706)^2 & (1\ 321 - 706)^2 \\ (679 - 706)^2 \end{matrix} & \text{(D.9)} \end{aligned}$$

$$\begin{aligned} &= \frac{1}{8} \times 1\ 164\ 657 \\ &= 145\ 582,1 \text{ rounded to } 145\ 582 \\ s &= \sqrt{145\ 582} & \text{(D.10)} \\ &= 381,6 \text{ rounded to } 382 \text{ particles/m}^3 \end{aligned}$$

**D.1.10**

The 95% upper confidence limits (UCL) are calculated in accordance with equation (C.4) (see C.3.4). As the number of

individual averages is  $m = 9$ , the  $t$  distribution taken from Table C.1 is  $t = 1,9$ .

$$\begin{aligned} 95\% \text{ UCL } 0,3 \text{ m } 6\ 349 & 1,9 \frac{2\ 154}{\sqrt{9}} \\ & 7\ 713,2 & \text{(D.11)} \\ & \text{rounded to } 7\ 713 \text{ particles/m}^3 \end{aligned}$$

$$95\% \text{ UCL} = 0,5 \text{ m} \cdot 706 \cdot 1,9 \frac{382}{\sqrt{9}} + 947,9 \quad (\text{D.12})$$

rounded to 948 particles/m<sup>3</sup>

$$\bar{x} = \frac{1}{5} (926 + 958 + 937 + 963 + 214) = \frac{1}{5} \cdot 3998 = 799,6$$

rounded to 800 particles/m<sup>3</sup> (D.13)

**D.1.11**

The interpretation of results is carried out according to B.6.1. In D.1.7, it was shown that the particle concentration of each single sample volume is less than the specified class limits. In D.1.10, it was shown that the calculated values of the 95% UCL are also less than the class limits established in D.1.3.

Therefore the airborne particulate cleanliness of the cleanroom meets the required classification.

**D.2 Example 2**

**D.2.1**

This example is constructed to show the influence of the 95% UCL calculations on the results.

A cleanroom is specified for a particulate cleanliness of ISO Class 3 in operation. The number of sampling locations has been determined to be five. As the number of sampling locations is more than one and less than ten, the calculation of the 95% UCL according to annex C is applicable.

Only one particle size (D = 0,1 m) is considered.

**D.2.2**

The particle concentration limit for ISO Class 3 at 0,1 m is taken from Table 1:

$$C_n (0,1 \text{ m}) = 1\,000 \text{ particles/m}^3$$

**D.2.3**

At each sampling location, only one single sample volume is taken (B.5.1.1). The number of particles per cubic metre, x<sub>i</sub>, is calculated for each location and recorded below:

| Sampling location | x <sub>i</sub> 0,1 m |
|-------------------|----------------------|
| 1                 | 926                  |
| 2                 | 958                  |
| 3                 | 937                  |
| 4                 | 963                  |
| 5                 | 214                  |

Each value of the concentration for D = 0,1 m is less than the limit established in D.2.2. This result satisfies the first part of classification (B.6.1) and therefore calculation of the 95% UCL according to annex C can proceed.

**D.2.4**

The overall mean of the averages is calculated in accordance with equation (C.2) (see C.3.2):

**D.2.5**

The standard deviation of the location averages is calculated in accordance with equation (C.3) (see C.3.3):

$$s^2 = \frac{1}{4} [(926 - 800)^2 + (958 - 800)^2 + (937 - 800)^2 + (963 - 800)^2 + (214 - 800)^2]$$

$$= \frac{1}{4} \cdot 429\,574 = 107\,393,5$$

rounded to 107 394 (D.14)

$$s = \sqrt{107\,394} = 327,7$$

rounded to 328 particles/m<sup>3</sup> (D.15)

**D.2.6**

The 95% UCL is calculated in accordance with equation (C.4) (see C.3.4):

As the number of individual averages is m = 5, the t distribution taken from Table C.1 is t = 2,1.

$$95\% \text{ UCL} = 800 + 2,1 \left( \frac{328}{5} \right) = 1\,108 \text{ particles/m}^3 \quad (\text{D.16})$$

**D.2.7**

The particle concentrations of all of the single sample volumes are less than the specified classification limit (D.2.2).

Calculation of the 95% upper confidence limit shows, however, that the airborne particulate cleanliness of the cleanroom does not meet the specified classification.

This constructed example demonstrates the effect of a single outlying low particle concentration (i.e. location 5) on the result of the 95% UCL test.

Because nonconformance of the air cleanliness classification results from application of the 95% UCL, and is caused by a single, low particle concentration, the procedure described in B.6.2 may be followed to determine whether the nonconformance can be waived.

## Annex E (informative)

### Considerations for the counting and sizing of particles outside the size range applicable for classification

#### E.1 Principle

In some situations, typically those related to specific process requirements, alternative levels of air cleanliness may be specified on the basis of particle populations that are not within the size range applicable to classification. The maximum permitted concentration of such particles and the choice of test method to verify compliance are matters for agreement between the customer and the supplier. Considerations for test methods and prescribed formats for specification are given in E.2 (for U descriptors) and E.3 (for M descriptors).

#### E.2 Consideration of particles smaller than 0,1 µm (ultrafine particles) — U descriptor

##### E.2.1. Application

If contamination risks caused by particles smaller than 0,1 µm are to be assessed, sampling devices and measurement procedures appropriate to the specific characteristics of such particles should be employed.

The number of sampling locations should be established in accordance with B.4.1 and the minimum sample volume  $V_s$  should be 2 litres (B.4.2.2).

##### E.2.2 U descriptor format

The ultrafine particle concentration of the U descriptor may be used alone or as a supplement to the airborne particulate cleanliness class. The U descriptor is expressed in the format

“U (x; y)”, where

- x is the maximum permitted concentration of ultrafine particles (expressed as ultrafine particles per cubic metre of air);
- y is the size in micrometres at which the applicable discrete-particle counter counts such particles with 50% counting efficiency.

**EXAMPLE** To express a maximum permitted ultrafine particle concentration of 140 000 particles/m<sup>3</sup> in the particle size range 0,01 µm, the designation would be: “U (140 000; 0,01 µm)”.

**NOTE 1** Suitable methods of test for concentrations of airborne particles smaller than 0,1 µm are given in IEST-G-CC1002 [1].

**NOTE 2** If the U descriptor designation is used as a supplement to an airborne particulate cleanliness class, the ultrafine particle concentration (x) should be not less than the particle concentration limit

(particles per cubic metre) applicable to the considered size of 0,1 µm for the specified ISO class.

#### E.3 Consideration of particles larger than 5 µm (macroparticles) — M descriptor

##### E.3.1 Application

If contamination risks caused by particles larger than 5 µm are to be assessed, sampling devices and measurement procedures appropriate to the specific characteristics of such particles should be employed.

As particle liberation within the process environment normally dominates the macroparticle fraction of the airborne particle population, the identification of an appropriate sampling device and measurement procedure should be addressed on an application-specific basis. Factors such as the density, shape, volume, and aerodynamic behaviour of the particles need to be taken into account. Also, it may be necessary to put special emphasis on specific components of the total airborne population, such as fibres.

##### E.3.2 M descriptor format

The M descriptor may be specified independently or as a supplement to the ISO classes of airborne particulate cleanliness. The M descriptor is expressed in the format

“M (a; b; c)”, where

- a is the maximum permitted concentration of macroparticles (expressed as macroparticles per cubic metre of air);
- b is the equivalent diameter (or diameters) associated with the specified method for measuring macroparticles (expressed in micrometres);
- c is the specified measurement method.

**NOTE 1** If the population of airborne particles being sampled contains fibres, they may be accounted for by supplementing the M descriptor with a separate descriptor for fibres, having the format “M<sub>fibre</sub> (a; b; c)”.

**EXAMPLE 1** To express an airborne particle concentration of 10 000 particles/m<sup>3</sup> in the particle size range of >5 µm based on the use of a time-of-flight aerosol particle counter to determine the aerodynamic diameter of the particles, the designation would be:

“M (10 000; >5 µm); time-of-flight aerosol particle counter”.

EXAMPLE 2 To express an airborne particle concentration of 1 000 particles/m<sup>3</sup> in the particle size range of 10 to 20 µm, based on the use of a cascade impactor followed by microscopic sizing and counting, the designation would be:

“M (1 000; 10 µm to 20 µm); cascade impactor followed by microscopic sizing and counting”.

NOTE 2 Suitable methods of test for concentrations of airborne particles larger than 5 µm are given in IEST-G-CC1003 [2].

NOTE 3 If the M descriptor designation is used as a supplement to an airborne particulate cleanliness class, the macroparticle concentration (a) should be not greater than the particle concentration limit (particles per cubic metre) applicable to the considered size of 5 µm for the specified ISO class.

## Annex F (informative)

### Sequential sampling procedure

#### F.1 Background and limitations

##### F.1.1 Background

If the air being sampled is significantly more or significantly less contaminated than the specified class concentration limit for the considered particle size, use of the sequential sampling procedure can reduce sample volumes and sampling times, often dramatically. Some savings may also be realized when the concentration is near the specified limit. Sequential sampling is most appropriate when air cleanliness is expected to quality as ISO Class 4 or cleaner.

NOTE For further information on sequential sampling, see IEST-G-CC1004 [3].

##### F.1.2 Limitations

The principal limitations of sequential sampling are:

- The procedure is only applicable to sampling aimed at a total of 20 particles per measurement, for particles of the considered size at the specified class or concentration limit.
- Each sample measurement requires supplementary monitoring and data analysis, which can be facilitated through computerized automation.
- Particle concentrations are not determined as

precisely as with conventional sampling procedures, due to the reduced sample volume

#### F.2 Basis for the procedure

The procedure is based on comparison of real-time cumulative particle counts to reference count values. Reference values are derived from equations for upper and lower limit boundaries:

$$\text{Upper limit: } C = 3,96 + 1,03 E \quad (\text{F.1})$$

$$\text{Lower limit: } C = -3,96 + 1,03 E \quad (\text{F.2})$$

where

$C$  is the observed count;

$E$  is the expected count.

To facilitate comparisons, helpful references have been provided in the form of a graph, Figure F.1, and in tabular form as Table F.1. Either format may be used.

As air is being sampled at each designated location, the running total particle count is continuously compared to reference count limits which are a function of the proportion of the prescribed total volume that has been sampled. If the running total count is less than the lower reference count limit corresponding to the volume that has been sampled, the air being

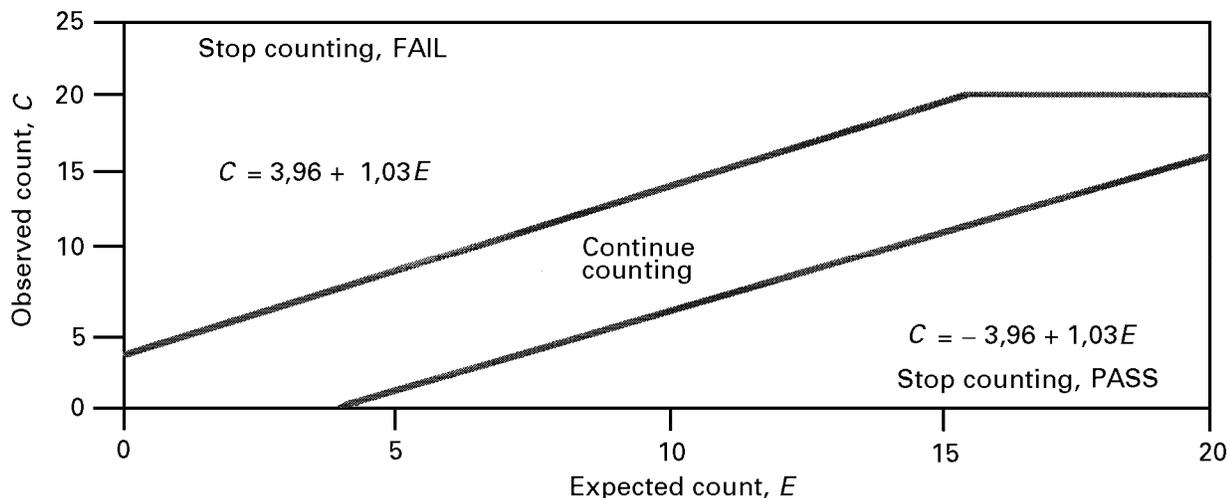


Figure F.1 — Boundaries for pass or fail by the sequential sampling procedure

Table F.1 — Upper and lower limits for time at which C observed counts should arrive

| FAILS IF COUNT, C, COMES EARLIER THAN EXPECTED |                | PASSES IF COUNT, C, COMES LATER THAN EXPECTED |                |
|--|----------------|---|----------------|
| Fractional time, t                             | Observed Count | Fractional time, t                            | Observed Count |
| 0,001 9  | 4              | 0,192 2                                       | 0              |
| 0,050 5  | 5              | 0,240 7                                       | 1              |
| 0,099 2  | 6              | 0,289 3                                       | 2              |
| 0,147 6  | 7              | 0,337 8                                       | 3              |
| 0,196 1  | 8              | 0,386 4                                       | 4              |
| 0,244 7  | 9              | 0,434 9                                       | 5              |
| 0,293 2  | 10             | 0,483 4                                       | 6              |
| 0,341 7  | 11             | 0,532 0                                       | 7              |
| 0,390 2  | 12             | 0,580 5                                       | 8              |
| 0,438 8  | 13             | 0,629 1                                       | 9              |
| 0,487 3  | 14             | 0,667 6                                       | 10             |
| 0,535 9  | 15             | 0,726 2                                       | 11             |
| 0,584 4  | 16             | 0,774 7                                       | 12             |
| 0,633 0  | 17             | 0,823 3                                       | 13             |
| 0,681 5  | 18             | 0,871 8                                       | 14             |
| 0,730 0  | 19             | 0,920 3                                       | 15             |
| 0,778 6  | 20             | 0,968 9                                       | 16             |
| 1,000 0  | 21             | 1,000 0                                       | 17             |

NOTE Fractional times are given as the fraction of total times (t = 1,000 0 at the class limit).

sampled is found to meet the specified class or concentration limit, and sampling is halted.

If the running count exceeds the upper reference count limit corresponding to the volume sampled, the air being sampled fails to meet the specified class or concentration limit, and sampling is halted. As long as the running count remains between the upper and lower limits, sampling continues until the full sample has been accumulated.

In the graph, Figure F.1, the number of observed counts, C, is plotted versus E, the expected number of counts for air being sampled at a rate (volume versus time) that would produce 20 counts in the time it would take to measure a full single sample of air if the concentration were at the specified limit for the considered particle size.

Table F.1 provides an equivalent method, in which the time of the observed count, C, is compared with incremental fractions of the time that would be required to measure a full single sample, as shown in the table. If the count occurs earlier than would be expected from the table, the air being sampled fails to meet the specified limit. If the count occurs later than

expected, the air being sampled meets the prescribed limit. At most, 21 comparisons of particle arrival times with the limiting times on the table would be required.

### F.3 Procedure for sampling

#### F.3.1 Sequential sampling references

Two optional comparison techniques are provided for judging the result as data collection proceeds. Progressive computerized analysis of the data is beneficial and recommended.

#### F.3.2 Graphical sampling comparison

Figure F.1 illustrates the boundaries established in equations (F.1) and (F.2), as truncated by the limitations of  $E = 20$ , representing the time required to collect a full sample, and  $C = 20$ , the maximum observed count allowed.

The observed count is plotted versus the expected count for air having a particle concentration precisely at the specified class level. The passage of time corresponds to increasing numbers of expected counts, with  $E = 20$  representing the time required

to accumulate a full sample volume if the particle concentration were at the class limit.

The procedure for sequential sampling using Figure F.1 is as follows:

As sampling proceeds, record the number of particles counted as a function of time, and compare the count with the upper and lower limit lines of Figure F.1. If the cumulative observed count crosses the upper line, sampling at the location is stopped and the air is reported to have failed compliance with the specified class limit. If the cumulative observed count crosses the lower line, sampling is stopped and the air passes the specified class limit. If the cumulative observed count remains between the upper and lower lines, sampling will continue.

If the total count is 20 or less at the end of the prescribed sampling period and has not crossed the upper line, the air is judged to have complied with the class limit.

### F.3.3 Tabular sampling comparison

Table F.1 provides an equivalent method for use with sequential sampling, also based on equations (F.1) and (F.2). The time,  $t$ , on the table is assigned a value of "1,000 0" to represent the duration of a complete single sample. The volume of this sample is the volume necessary to provide 20 particles, if the air contains precisely the class limit equivalent concentration of particles of the considered size. The time values listed in the table are the fractional portions of the total time required for accumulation of the entire single sample.

The procedure for sequential sampling using Table F.1 is as follows:

As sampling proceeds, record the number of particles counted as a function of time, and compare the time at which each count is observed with the times shown in the two columns of the table. If a given cumulative observed count occurs earlier than expected, as indicated by comparison with the left-hand column, sampling is stopped and the air is reported to have failed compliance with the specified class limit. If the cumulative observed count occurs later than expected, as indicated by comparison with the right-hand column, sampling is stopped and the air is reported to be in compliance with the specified limit. If the cumulative observed count continuously arrives between the times shown in the two columns, sampling will continue. If counting continues through 21 comparisons with the left-hand column and no count arrives earlier than its expected time, the air passes the specified limit for a full single sample.

## Bibliography

[1] IEST-G-CC1002, Determination of the Concentration of Airborne Ultrafine Particles. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology (1999)

[2] IEST-G-CC1003, Measurement of Airborne Macroparticles. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology (1999)

[3] IEST-G-CC1004, Sequential Sampling Plan for Use in Classification of the Particulate Cleanliness of Air in Cleanrooms and Clean Zones. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology (1999)

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**Cleanrooms and associated controlled environments —**

Part 2:

**Specifications for testing and monitoring to  
prove continued compliance with  
ISO 14644-1**

*Salles propres et environnements maîtrisés apparentés —*

*Partie 2: Spécifications pour les essais et la surveillance en vue de  
démontrer le maintien de la conformité avec l'ISO 14644-1*



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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 14644 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14644-2 was prepared by Technical Committee ISO/TC 209, *Cleanrooms and associated controlled environments*.

ISO 14644 consists of the following parts, under the general title *Cleanrooms and associated controlled environments*:

*Part 1: Classification of air cleanliness*

*Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1*

*Part 3: Metrology and test methods*

*Part 4: Design, construction and start-up*

*Part 5: Operations*

*Part 6: Terms and definitions*

*Part 7: Separative enclosures (clean airhoods, gloveboxes, isolators, mini-environments)*

Users should note that the titles listed for parts 3 to 7 are working titles at the time of the release of part 2. In the event that one or more of these parts are deleted from the work programme, the remaining parts may be renumbered.

Annexes A and B of this part of ISO 14644 are for information only.

## Introduction

This part of ISO 14644 provides a process to prove continued compliance with ISO 14644-1 and specifies minimum requirements for testing and monitoring. In any testing plan, consideration should also be given to the particular operational requirements, risk assessment of the installation, and its use.

Cleanrooms and associated controlled environments provide for the control of airborne particulate contamination to levels appropriate for accomplishing contamination-sensitive activities. Products and processes that benefit from the control of airborne contamination include aerospace, microelectronics, pharmaceuticals, medical devices, healthcare, food and others. Many factors besides airborne particulate cleanliness should be considered in the design, specification, operation and control of cleanrooms and other controlled environments.

In some circumstances, relevant regulatory agencies may impose supplementary policies or restrictions. In such situations, appropriate adaptations of the standard testing procedures may be required.

# Cleanrooms and associated controlled environments —

## Part 2:

# Specifications for testing and monitoring to prove continued compliance with ISO 14644-1

## 1 Scope

This part of ISO 14644 specifies requirements for periodic testing of a cleanroom or clean zone to prove its continued compliance with ISO 14644-1 for the designated classification of airborne particulate cleanliness.

These requirements invoke the test described in ISO 14644-1 for classification of a cleanroom or clean zone. Additional tests are also specified, to be carried out in accordance with the requirements of this part of ISO 14644. Optional tests, to be applied at the user's discretion, are also identified.

This part of ISO 14644 also specifies requirements for monitoring of a cleanroom or clean zone (hereafter referred to as an installation) to provide evidence of its continued compliance with ISO 14644-1 for the designated classification of airborne particulate cleanliness.

## 2 Normative references

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 14644. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 14644 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 14644-1:1999, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness*. ISO 14644-3:—<sup>1)</sup>, *Cleanrooms and associated controlled environments — Part 3: Metrology and test methods*.

## 3 Terms and definitions

For the purposes of this part of ISO 14644, the terms and definitions given in ISO 14644-1 and the following apply.

### 3.1 General terms

#### 3.1.1

##### **requalification**

execution of the test sequence specified for the installation to demonstrate compliance with ISO 14644-1 according to the classification of the installation, including the verification of the selected pre-test conditions

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1) To be published.

**3.1.2**

**test**

procedure undertaken in accordance with a defined method to determine the performance of an installation or an element thereof

**3.1.3**

**monitoring**

observations made by measurement in accordance with a defined method and plan to provide evidence of the performance of an installation

NOTE This information may be used to detect trends in operational state and to provide process support.

**3.2 Terms concerning frequency intervals**

**3.2.1**

**continuous**

updating that occurs constantly

**3.2.2**

**frequent**

updating that occurs at specified intervals not exceeding 60 minutes during operation

**3.2.3**

**6 months**

updating that occurs at an average interval not exceeding 183 days throughout periods of operational use, subject to no interval exceeding 190 days

**3.2.4**

**12 months**

updating that occurs at an average interval not exceeding 366 days throughout periods of operational use, subject to no interval exceeding 400 days

**3.2.5**

**24 months**

updating that occurs at an average interval not exceeding 731 days throughout periods of operational use, subject to no interval exceeding 800 days

**4 Demonstration of continued compliance**

**4.1 Principle**

Continued compliance with air cleanliness (ISO class) requirements specified for the installation is verified by performing specified tests and by documenting the results. Monitoring data is used as an indication of installation status and may determine the frequency with which tests are carried out.

**4.2 Testing for continued compliance**

**4.2.1** The reference test method and the maximum time intervals between such tests to prove continued compliance with the designated ISO class are given in Table 1.

**Table 1 — Schedule of testing to demonstrate compliance with particle concentration limits**

| Classification  | Maximum time interval | Test method                 |
|---|-----------------------|-----------------------------|
| u ISO Class 5   | 6 months              | Annex B in ISO 14644-1:1999 |
| > ISO Class 5   | 12 months             | Annex B in ISO 14644-1:1999 |
| NOTE Particle count tests will normally be performed in the operational state, but may also be performed in the at-rest state in accordance with the designated ISO classification. |                       |                             |

**4.2.2** Where the application requires them, tests as given in Table 2 shall be carried out to demonstrate compliance. The requirement for each of these tests shall be determined by agreement between the customer and supplier.

**Table 2 — Schedule of additional tests for all classes**

| Test parameter  | Maximum time interval | Test procedure            |
|---|-----------------------|---------------------------|
| Airflow volume <sup>a</sup> or airflow velocity   | 12 months             | ISO 14644-3:—, clause B.4 |
| Air pressure difference <sup>b</sup>  | 12 months             | ISO 14644-3:—, clause B.5 |
| NOTE These tests may normally be performed in either the operational or at-rest state in accordance with the designated ISO classification. |                       |                           |
| <sup>a</sup> Airflow volume may be determined by either velocity or volume measurement techniques.  |                       |                           |
| <sup>b</sup> This test will not apply to clean zones which are not totally enclosed.  |                       |                           |

**4.2.3** In addition to the normative tests given in Tables 1 and 2, other tests may be included by agreement between customer and supplier as considered appropriate to the installation, such as those listed in annex A.

**4.2.4** Where the installation is equipped with instrumentation for continuous or frequent monitoring of the airborne particle concentration, and air pressure difference, where applicable, the maximum time interval as stated in Table 1 may be extended, provided that the results of continuous or frequent monitoring remain within the specified limit(s).

**4.2.5** In those installations that require additional tests, and where the installation is equipped with instrumentation for continuous or frequent monitoring of the test parameter applicable, the maximum time interval(s) as stated in Table 2 may be extended, provided that the results of continuous or frequent monitoring remain within the specified limit(s).

**4.2.6** Where instruments are used for testing, they shall be calibrated in accordance with current industry practice.

**4.2.7** If the test results are within the limits specified, then the installation is in a condition of continued compliance. If any of the test results exceeds the limits specified, the installation is not in compliance and appropriate remedial action shall be taken. Following remedial action, requalification shall be undertaken.

**4.2.8** Requalification of the installation shall be undertaken after any of the following.

- a) Completion of remedial action implemented to rectify an out-of-compliance condition.
- b) A significant change from the current performance specification, such as a change in operational use. The significance of a change should be determined by agreement between the customer and the supplier.
- c) Any significant interruption of air movement which affects the operation of the installation. The significance of an interruption should be determined by agreement between the customer and the supplier.

- d) Special maintenance which significantly affects the operation of the installation, (e.g. change of final filters). The significance of an effect should be determined by agreement between the customer and the supplier.

### **4.3 Monitoring**

**4.3.1** Routine monitoring of airborne particle concentration and other parameters shall be performed according to a written plan.

NOTE Monitoring is normally performed with the installation in the operational state.

**4.3.2** The airborne-particle monitoring plan shall be based on risk assessment (see annex B) related to the application of the installation. The plan shall include, as a minimum, predetermined sample locations, minimum volume of air per sample, duration of measurements, number of measurements per sample location as required, time interval between measurements, particle size or sizes to be counted, and count acceptance limits, as well as count alert, action and excursion limits, if appropriate.

NOTE 1 If continuous or frequent monitoring is specified in the plan for both airborne particle counting and air pressure difference monitoring, the schedule for the particle count test may be modified by extending the time interval between tests (see 4.2.4 and 4.2.5).

NOTE 2 Monitoring of other attributes (e.g. temperature and humidity) may also be undertaken in the same manner as above.

**4.3.3** If monitoring results exceed specified action limits, the installation shall be considered non-compliant and appropriate remedial action shall be taken. Following remedial action, appropriate tests (see 4.2 and annex A) shall be performed to determine if the installation is in compliance. If compliance has been achieved, the monitoring may be resumed.

**4.3.4** Instruments used for monitoring shall be calibrated in accordance with current industry practice.

### **4.4 Documentation**

**4.4.1** The results from requalification or testing of each installation to prove continued compliance shall be recorded and submitted as a comprehensive report, along with a statement of compliance or non-compliance with the specified tests.

The test report shall include the following:

- a) name and address of the testing organization;
- b) operator(s) identification and the date on which the test was performed;
- c) reference to this part of ISO 14644, i.e. ISO 14644-2:2000;
- d) clear identification of the physical location of the installation tested (including reference to adjacent areas if necessary) and specific designations for coordinates of all sampling locations;
- e) specified designation criteria for the installation, including the ISO classification and considered particle size(s), relevant occupancy state(s), airflow volume or velocity, and air pressure difference;
- f) measuring instruments used and proof of calibration;
- g) test results, including particle concentration data for all sampling location coordinates;
- h) date of the preceding test to prove continued compliance.

Where the maximum time intervals are extended in accordance with 4.2.4 and 4.2.5, the results of the continuous or frequent monitoring shall also form part of the documentation.

**4.4.2** Documentation of the monitoring of each installation shall be as set out in the monitoring plan.

#### **4.5 Records**

Records retention shall conform to any quality control procedures in place for the installation. Records shall comply with regulatory requirements.

## Annex A (informative)

### Optional tests

In addition to the normative tests specified in Tables 1 and 2, optional tests, such as those listed in Table A.1, may be included within the testing plan.

**Table A.1 — Schedule of optional tests**

| Test parameter           | Class       | Suggested maximum time interval | Test procedure             |
|--------------------------|-------------|---------------------------------|----------------------------|
| Installed filter leakage | All classes | 24 months                       | ISO 14644-3:—, clause B.6  |
| Airflow visualization    | All classes | 24 months                       | ISO 14644-3:—, clause B.7  |
| Recovery                 | All classes | 24 months                       | ISO 14644-3:—, clause B.13 |
| Containment leakage      | All classes | 24 months                       | ISO 14644-3:—, clause B.14 |

**Annex B**  
(informative)

**Guidance on the influence of risk  
assessment on cleanroom or clean zone  
tests and monitoring**

**ISO**  
**14644-4**

First edition  
2001-04-01

The risk assessment pertaining to a particular cleanroom or clean zone application will affect the following:

- a) monitoring plan;
- b) interpretation of monitoring data;
- c) actions to be taken as a result of the monitoring data obtained;
- d) selection of parameters to ~~be measured from Table 2;~~
- e) selection of parameters to be measured from Table A.1.

**ICS 13.040.30**

Price based on 7 pages

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**INTERNATIONAL  
STANDARD**

# Cleanrooms and associated controlled environments —

## Part 4: Design, construction and start-up

*Salles propres et environnements maîtrisés apparentés —  
Partie 4: Conception, construction et mise en fonctionnement*

Reference number  
ISO 14644-4:2001(E)

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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Attention is drawn to the possibility that some of the elements of this part of ISO 14644 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14644-4 was prepared by Technical Committee ISO/TC 209, *Cleanrooms and associated controlled environments*.

ISO 14644 consists of the following parts, under the general title *Cleanrooms and associated controlled environments*:

*Part 1: Classification of air cleanliness*

*Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1*

*Part 3: Metrology and test methods*

*Part 4: Design, construction and start-up*

*Part 5: Operations*

*Part 6: Vocabulary*

*Part 7: Separative enclosures (clean air hoods, glove boxes, isolators, mini-environments)*

Users should note that the titles listed for parts 3 and 5 to 7 are working titles at the time of the release of part 4. In the event that one or more of these parts are deleted from the work programme, the remaining parts may be renumbered.

Annexes A to H of this part of ISO 14644 are for information only.

## Introduction

Cleanrooms and associated controlled environments provide for the control of airborne particulate contamination to levels appropriate for accomplishing contamination-sensitive activities. Products and processes that benefit from the control of airborne contamination include those in such industries as aerospace, microelectronics, pharmaceuticals, medical devices and healthcare.

This part of ISO 14644 specifies the requirements for the design and construction of cleanroom facilities. It is intended for use by purchasers, suppliers and designers of cleanroom installations and provides a check list of important parameters of performance. Construction guidance is provided, including requirements for start-up and qualification. Basic elements of design and construction needed to ensure continued satisfactory operation are identified through the consideration of relevant aspects of operation and maintenance.

This part of ISO 14644 is one of a series of standards concerned with cleanrooms and associated subjects. Many factors besides design, construction and start-up should be considered in the operation and control of cleanrooms and other controlled environments. These are covered in some detail in other International Standards prepared by ISO/TC 209.



# Cleanrooms and associated controlled environments

## Part 4: Design, construction and start-up

### 1 Scope

This part of ISO 14644 specifies requirements for the design and construction of cleanroom installations but does not prescribe specific technological or contractual means to meet these requirements. It is intended for use by purchasers, suppliers and designers of cleanroom installations and provides a checklist of important parameters of performance. Construction guidance is provided, including requirements for start-up and qualification. Basic elements of design and construction needed to ensure continued satisfactory operation are identified through the consideration of relevant aspects of operation and maintenance.

NOTE Further guidance in respect of the above requirements is given in annexes A to H. Other parts of ISO 14644 may provide complementary information.

Application of this part of ISO 14644 is restricted in the following:

user requirements are represented by purchaser or specifier;

specific processes to be accommodated in the cleanroom installation are not specified;

fire and safety regulations are not considered specifically; the appropriate national and local requirements should be respected;

process media and utility services are only considered with respect to their routing between and in the different zones of cleanliness;

regarding initial operation and maintenance, only cleanroom construction-specific requirements are considered.

### 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 14644. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 14644 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 14644-1:1999, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness*.

ISO 14644-2:2000, *Cleanrooms and associated controlled environments — Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1*.

ISO 14644-3:—<sup>1</sup>, *Cleanrooms and associated controlled environments — Part 3: Metrology and test methods*.

ISO 14698-1:—<sup>1</sup>, *Cleanrooms and associated controlled environments — Biocontamination control — Part 1: General principles*

ISO 14698-2:—<sup>1</sup>, *Cleanrooms and associated controlled environments — Biocontamination control — Part 2: Evaluation and interpretation of biocontamination data*.

ISO 14698-3:—<sup>1</sup>, *Cleanrooms and associated controlled environments — Biocontamination control — Part 3: Measurement of the efficiency of processes of cleaning and/or disinfection of inert surfaces bearing biocontaminated wet soiling or biofilms*.

### 3 Terms and definitions

For the purposes of this part of ISO 14644, the terms and definitions given in ISO 14644-1 and the following apply.

#### 3.1

##### **changing room**

room where people using a cleanroom may change into, or out of, cleanroom apparel

#### 3.2

##### **clean air device**

stand-alone equipment for treating and distributing clean air to achieve defined environmental conditions

#### 3.3

##### **cleanliness**

condition of a product, surface, device, gas, fluid, etc. with a defined level of contamination

NOTE Contamination can be particulate, non-particulate, biological, molecular or of other consistency.

#### 3.4

##### **commissioning**

planned and documented series of inspections, adjustments and tests carried out systematically to set the installation into correct technical operation as specified

#### 3.5

##### **contaminant**

any particulate, molecular, non-particulate and biological entity that can adversely affect the product or process

#### 3.6

##### **non-unidirectional airflow**

air distribution where the supply air entering the clean zone mixes with the internal air by means of induction

#### 3.7

##### **particle**

minute piece of matter with defined physical boundaries

NOTE For classification purposes refer to ISO 14644-1.

#### 3.8

##### **pre-filter**

air filter fitted upstream of another filter to reduce the challenge on that filter

---

<sup>1</sup> To be published.

**3.9****process core**

location at which the process and the interaction between the environment and the process occurs

**3.10****start-up**

act of preparing and bringing an installation into active service, including all systems

EXAMPLE Systems may include procedures, training requirements, infrastructure, support services, statutory under-takings requirements.

**3.11****unidirectional airflow**

controlled airflow through the entire cross-section of a clean zone with a steady velocity and approximately parallel streamlines

NOTE This type of airflow results in a directed transport of particles from the clean zone.

**4 Requirements**

**4.1** The parameters listed in 4.2 to 4.18 shall be defined and agreed between purchaser and supplier:

NOTE In the requirements stated below, references are made to annexes A to H which are for information only.

**4.2** The number, edition and date of publication of this part of ISO 14644 shall be given.

**4.3** The role of other relevant parties to the project (e.g. consultants, designers, regulatory authorities, service organizations) shall be established (see examples in annex C).

**4.4** The general purpose for which the cleanroom is to be used, the operations to be carried out therein and any constraint imposed by the operating requirements (see examples in annexes A, B and D).

**4.5** The required airborne particulate cleanliness class or demands for cleanliness in accordance with the relevant International Standard (ISO 14644-1, ISO 14698-1, ISO 14698-2 and ISO 14698-3) (see examples in annex B).

**4.6** The critical environmental parameters, including their specified set points, alert and action levels to be measured to ensure compliance, together with the measurement methods to be used, including calibration (ISO 14644-2 and ISO 14644-3) (see examples in annex F).

**4.7** The contamination control concept, including installation, operating and performance criteria, to be used to achieve the required cleanliness level (see examples in annex A).

**4.8** The methods of measurement, control, monitoring and documentation required to meet the parameters agreed (see examples in annexes C and F).

**4.9** The entry or exit of equipment, apparatus, supplies and personnel required to support the installation (see examples in annex D).

**4.10** The specified occupancy states selected from "as-built", "at-rest" and "operational" under which the required parameters shall be achieved and maintained including variations with time, and the methods of control (see examples in annex C).

**4.11** The layout and configuration of the installation (see examples in annex D).

**4.12** Critical dimensions and mass restrictions, including those related to available space (see examples in annex D).

- 4.13 The process and product requirements that affect the installation (see examples in annexes B and G).
- 4.14 The process equipment list with utility requirements (see examples in annexes D, E and H).
- 4.15 The maintenance requirements of the installation (see examples in annexes D and E).
- 4.16 The assignment of tasks for the preparation, approval, execution, supervision, documentation, statement of criteria, basis of design, detailed design, construction, testing, commissioning and qualification (including the performance and witnessing) of tests (see examples in annexes E and G).
- 4.17 The identification and evaluation of external environmental influences (see examples in annex H).
- 4.18 Additional information required by the particular application (see examples in annex H).

## 5 Planning and design

### 5.1 Planning procedure

- 5.1.1 A project plan shall be developed, in consultation with the user and all other involved parties, to define the requirements of the products, the processes and the scope of the installation.
  - 5.1.2 In order to determine the needs of an installation, a process equipment list shall be compiled, and shall include the critical requirements for each piece of process equipment.
  - 5.1.3 Diversity factors shall be defined, considering peak and average demand for each utility and environmental control system.
- NOTE A system may include multiple subsystems which require individual diversity-factor determination.
- 5.1.4 A contamination control concept shall be developed for each zone of an installation (see examples in annex A).
  - 5.1.5 The specifications as defined in clause 4 shall be reviewed and refined based on financial and timescale requirements.

5.1.6 The project plan shall include the following elements:

- a) design documentation with support calculations;
- b) cost evaluation;
- c) timescale evaluation;
- d) an outline of anticipated project complications;
- e) design options with records of advantages and disadvantages and any recommendations;
- f) a review of maintenance requirements of the installation;
- g) a review of the degree of flexibility to be included in the installation;
- h) a review of the stand-by capacities to be included in the installation;
- i) a review of the constructability of the design of the installation;
- j) a quality plan.

The use of a quality system, such as the ISO 9000 family of international standards (e.g. ISO 9000 and ISO 9001), should be considered, in conjunction with industry-specific quality assurance strategies.

**5.1.7** The completed project plan shall be reviewed and agreed upon between purchaser and supplier.

## **5.2 Design**

**5.2.1** The design shall accommodate all of the relevant product and process requirements in conjunction with the selected contamination control concept (see examples in annex A).

**5.2.2** The purchaser and supplier shall formally accept the design in accordance with predetermined acceptance criteria.

**5.2.3** The design shall conform to an agreed list of requirements, such as building, environmental and safety regulations, good manufacturing practice guidelines (e.g. ISO 14001 and ISO 14004).

The design should be reviewed at periodic stages of development, including final completion, to ensure compliance with the specifications and the acceptance criteria.

## **6 Construction and start-up**

**6.1** Construction of an installation shall comply with the drawings and specifications.

**6.2** Any changes required during the course of construction shall be checked for acceptance, approved and documented prior to implementation of the change in accordance with a change control procedure.

**6.3** Construction work, whether performed at a manufacturing location or *in situ*, shall observe the specific contamination control requirements of the quality plan.

**6.4** A clean construction protocol and cleaning procedures shall be developed as part of the quality plan and enforced to achieve the specified contamination control requirements. Security and access control is essential to maintain the clean construction protocol.

**6.5** The cleaning methods and methods to determine and approve the achieved cleanliness shall be defined and documented in the quality plan.

**6.6** The cleaning of the air systems shall be specified and shall be carried out at assembly, before initial operation and whenever rebuilding work, repair work and maintenance work are performed.

**6.7** In the case of start-up of new installations or re-starting existing installations after repair or modification, final cleaning of the cleanroom is necessary and provisions shall be made for the removal of adherent, imported or released contamination.

**6.8** Before commencing any operational activities, the complete and satisfactory function of the installation shall be determined by tests carried out in accordance with clause 7.

**NOTE** In the case of packaged units, such as clean air devices, a manufacturer's certificate of compliance with the requirements of this part of ISO 14644 may be sufficient, provided that the supplier is qualified (i.e. knowledgeable of or competent in cleanroom requirements) and the risk of damage during transport, storage and installation can be controlled adequately.

**6.9** During acceptance testing, commissioning and initial operation, the personnel in charge of the installation shall be trained. Testing, approval of the installation and training shall include all relevant practices for proper cleanroom operation, maintenance and in-process control. The responsibility for providing training shall be defined.

When training is carried out, all relevant persons such as operators, maintenance and service personnel should be included.

## 7 Testing and approval

### 7.1 General

During and upon completion of the construction of an installation, an agreed series of documented tests shall be specified and undertaken prior to operational use of the installation. Annex C gives examples of the design, testing and approval processes.

### 7.2 Construction approval

A systematic range of inspections, adjustments, measurements and tests shall be carried out to ensure that each part of the installation complies with the design requirements.

### 7.3 Functional approval

A series of tests and measurements shall be carried out to determine that all parts of the installation operate together to achieve the required conditions in the "as-built" or "at-rest" states.

### 7.4 Operational approval

A series of tests and measurements shall be carried out to determine that the complete installation achieves the required "operational" performance with the specified process or activity functioning, and with the specified number of personnel present working in the agreed manner.

## 8 Documentation

### 8.1 General

Details of a completed installation (including instrumentation calibration) and all operation and maintenance procedures shall be documented. Documents shall be made readily available to all personnel responsible for start-up, operation and maintenance of the installation.

Such personnel should fully understand the documentation.

### 8.2 Record of an installation

Details of the completed installation shall be provided and shall contain:

- a) a description of the installation and its function;
- b) a set of final and approved performance test data, derived from the tests carried out in accordance with clause 7 of this part of ISO 14644, recording the values of all conditions defined in the specification for the installation and achieved during the commissioning, testing and start-up procedures;
- c) a set of drawings, diagrams (e.g. layout of wiring, piping and instrumentation) and specifications describing the completed and approved "as-built" installation and its components;
- d) a list of parts and equipment and any recommendation for stocking spare parts.

### 8.3 Operational instructions

Each installation or system shall be provided with a clear set of operating instructions. Such operating instructions shall contain:

- a) schedules of checks and inspections to be completed prior to the start-up of an installation;

- b) schedules of the acceptance range of the critical performance parameters specified;
- c) procedures to start and stop the installation under normal and failure mode situations;
- d) procedures to be adopted in the event of alert or action levels being reached.

#### **8.4 Instructions for performance monitoring**

Performance-monitoring of an installation is essential to demonstrate satisfactory operation. Documentation shall include:

- a) test and measurement frequency;
- b) description of test and measurement methods, (or reference to standards and guidelines);
- c) action plan in the event of non-compliance;
- d) frequency required for assembly, analysis and retention of performance data to enable trends to be analysed.

#### **8.5 Maintenance instructions**

Maintenance shall be implemented in accordance with a specified method and programme.

Maintenance and repairs shall be carried out during the construction, commissioning, testing, start-up and normal operation of an installation. The following items shall be considered:

- a) definition of safety procedures prior to carrying out maintenance or repairs;
- b) specification of maintenance actions to be taken when the acceptance range of any critical performance parameter is exceeded;
- c) agreed definition of permitted adjustments;
- d) methods of making permitted adjustments;
- e) methods of checking and calibrating control, safety and monitoring devices;
- f) requirements for checking and replacing all wearing parts (e.g. driving belts, bearings, filters);
- g) specification for cleaning of the installation or components prior to, during and after maintenance work;
- h) definition of actions, procedures and tests required after maintenance is completed;
- i) inclusion of any user-specific or relevant regulatory authority requirements.

#### **8.6 Maintenance record**

A documented record of any maintenance carried out upon the installation during construction, commissioning and start-up shall be maintained. The following items shall form part of the record:

- a) definition of the maintenance tasks;
- b) identification and approval of personnel undertaking the maintenance;
- c) date of carrying out the maintenance;
- d) a condition report prior to undertaking the maintenance;

- e) a list of spare parts used;
- f) a report upon completion of the maintenance.

### **8.7 Record of operation and maintenance training**

A documented record of training shall be maintained. The following items shall form part of the record:

- a) definition of the training content;
- b) identification of personnel providing and receiving the training;
- c) training date and duration;
- d) a report upon each period of training as it is completed.

## Annex A (informative)

### Control and segregation concepts

#### A.1 Contamination control zones

For economic, technical and operational reasons, clean zones are often enclosed or surrounded by further zones of lower cleanliness classification. This can allow the zones with the highest cleanliness demands to be reduced to the minimum size. Movement of material and personnel between adjacent clean zones gives rise to the risk of contamination transfer, therefore special attention should be paid to the detailed layout and management of material and personnel flow.

Figure A.1 illustrates an example of a contamination control concept. In this configuration, the clean zone would be regarded as a more stringently controlled portion of the cleanroom.

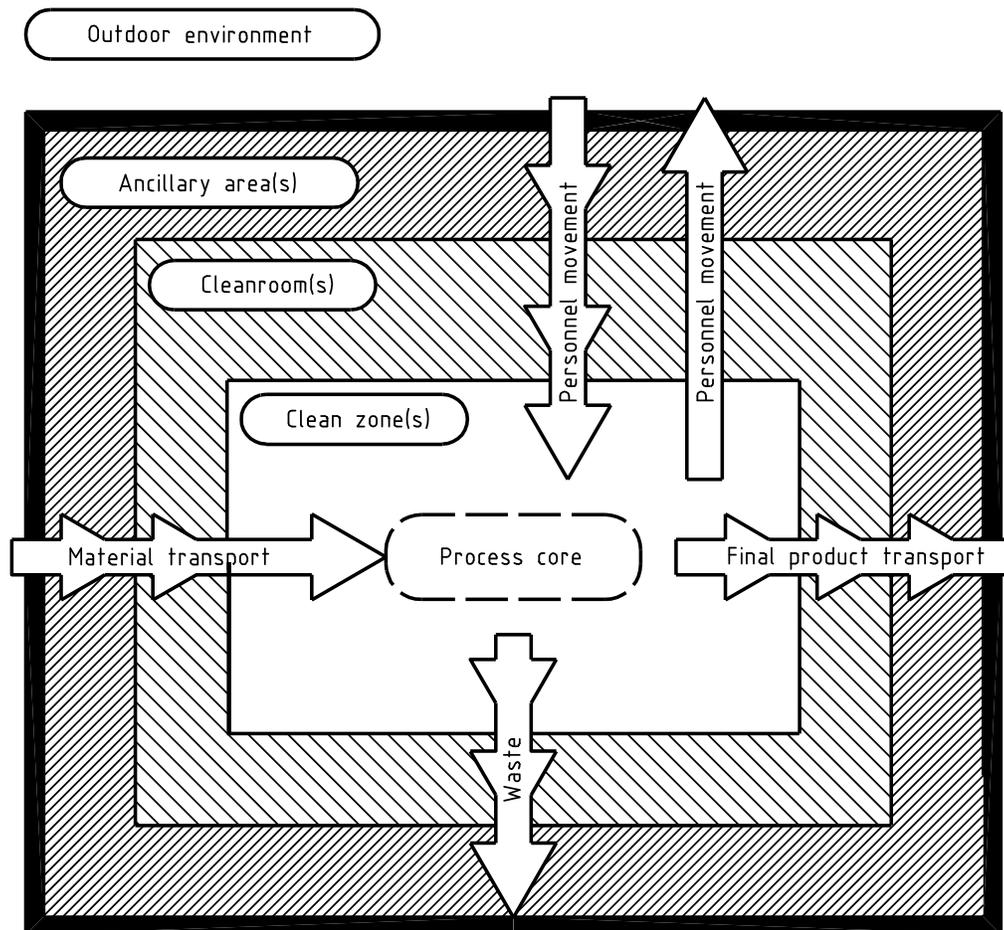


Figure A.1 — Shell-like contamination control concept

## A.2 Airflow patterns

**A.2.1** Cleanroom airflow patterns can be categorized as either unidirectional or non-unidirectional. When a combination of the two is used it is frequently called mixed airflow. Airflow patterns for cleanrooms of ISO Class 5 and cleaner in operation are often unidirectional, while non-unidirectional and mixed flow is typical for cleanrooms of ISO Class 6 and less clean in operation.

**A.2.2** Unidirectional airflow may be either vertical or horizontal (see Figure A.2). Both types of unidirectional airflow rely upon a final filtered air supply and air return inlets which are nearly opposite one another in order to maintain the airstream in as straight a flow pattern as possible. In both designs, the important design feature is the ability to ensure that the airflow pattern is disrupted as little as possible at the process core.

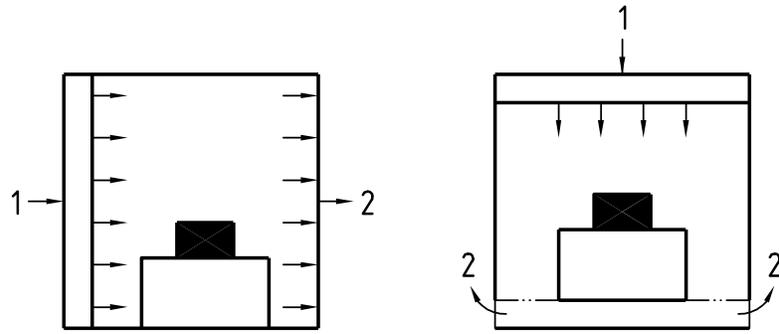
In a working plane perpendicular to the clean airflow, all positions offer the same cleanliness level. Hence, horizontally integrated or distributed processes require vertical airflow and vertically integrated processes require horizontal airflow. Working positions immediately adjacent to the clean air supply offer optimal contamination control conditions, because working positions downstream of these positions may be subject to particles generated upstream. Personnel placement should be therefore downstream of clean processing.

**A.2.3** In non-unidirectional airflow cleanrooms, air flows from filter outlets located in multiple positions distributed across the inlet plane and is returned through remote locations. Filter outlets may be distributed at equal intervals throughout the cleanroom or clean zone or grouped over the process cores. The location of filter outlets is important for the cleanroom performance. The final filter location may be remote, but special precautions should be taken to avoid contamination ingress between these filters and the cleanroom (e.g. monitoring of the surface cleanliness and airtightness of ventilation ducts and supply air inlets to avoid induction of contamination as well as the deployment of decontamination procedures). While return air locations in non-unidirectional airflow systems are not as critical as those in unidirectional applications, care should be taken to distribute the returns, as is done with the supplies, to minimize dead zones within the cleanroom.

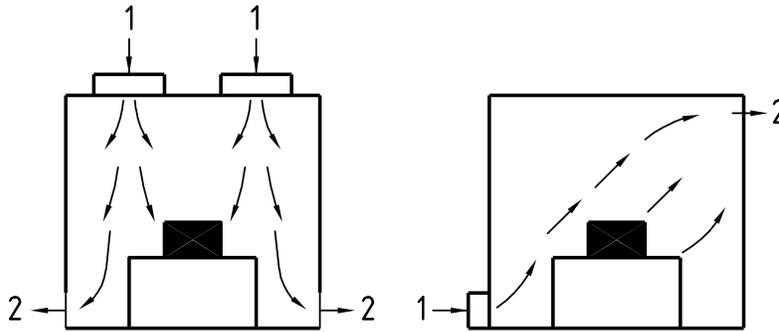
**A.2.4** Mixed-airflow cleanrooms combine both unidirectional and non-unidirectional airflow in the same room.

NOTE Some special designs are available that provide protection to specific working zones by other managed airflow techniques.

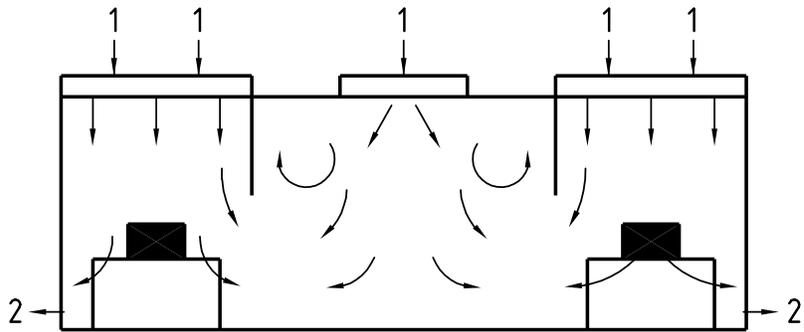
Figure A.2 gives examples that illustrate the different airflow patterns in cleanrooms. (Thermal effects are not considered.)



a) Unidirectional airflow



b) Non-unidirectional airflow



c) Mixed airflow

**Key**

- 1 Supply air
- 2 Return air

**Figure A.2 — Airflow patterns in cleanrooms**

### A.3 Disturbance of unidirectional airflow

In unidirectional airflow cleanrooms, the design of physical obstacles such as the process equipment, and the operating procedures, personnel movements and product handling, should consider basic aerodynamic requirements to prevent serious turbulence in the vicinity of the contamination-sensitive activity. Appropriate measures should be taken to avoid flow disturbances and cross-contamination between different work stations.

Figure A.3 shows the influence of physical obstacles (on the left) and appropriate measures for minimizing the impact of these (on the right).

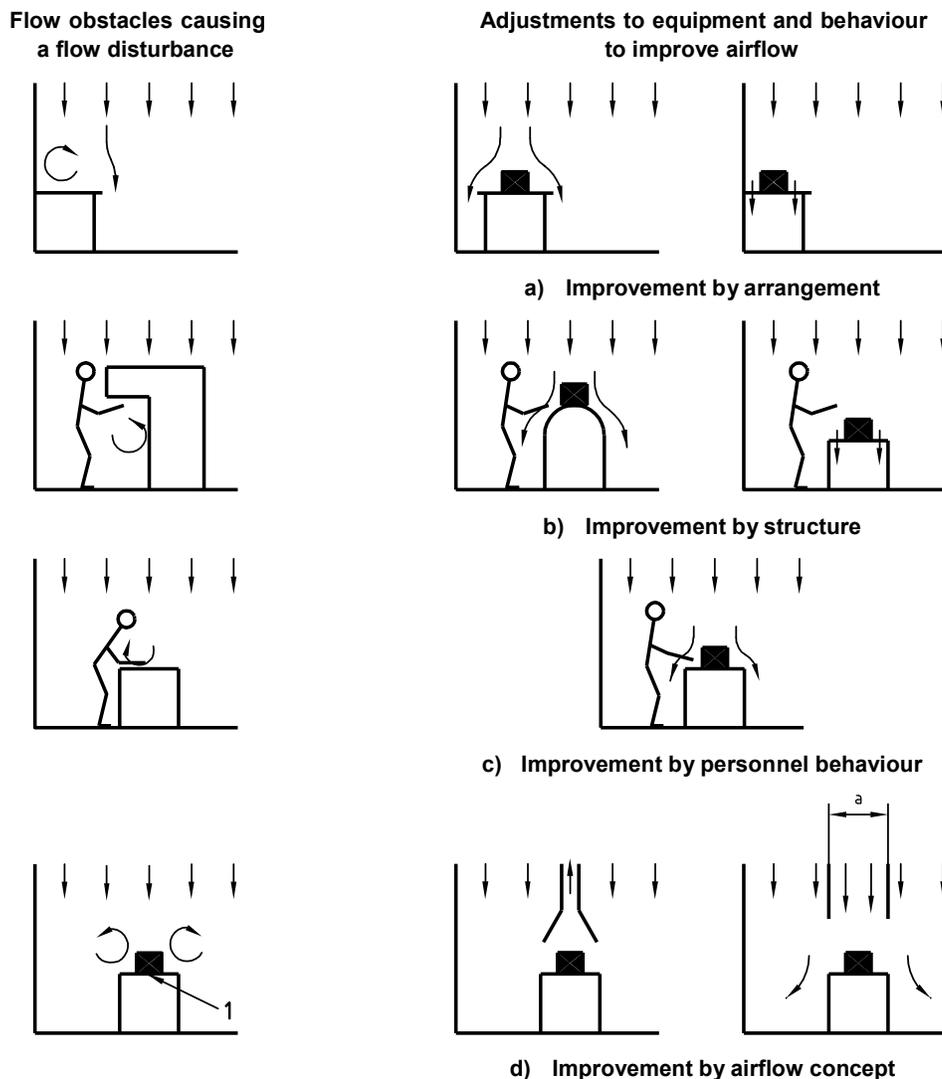


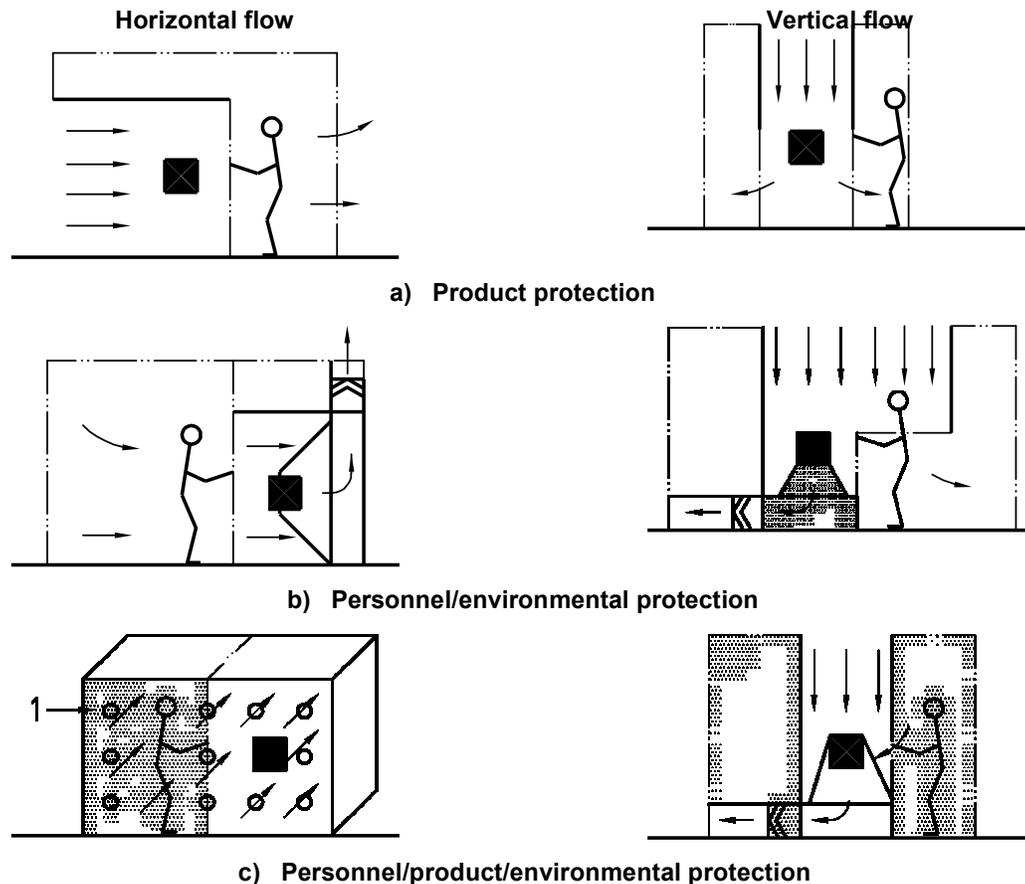
Figure A.3 — Influence of personnel and objects on unidirectional airflow

## A.4 Contamination control concepts

To select the proper technique for a given contamination control problem, Figures A.4 and A.5 show some different contamination control concepts that may be considered.

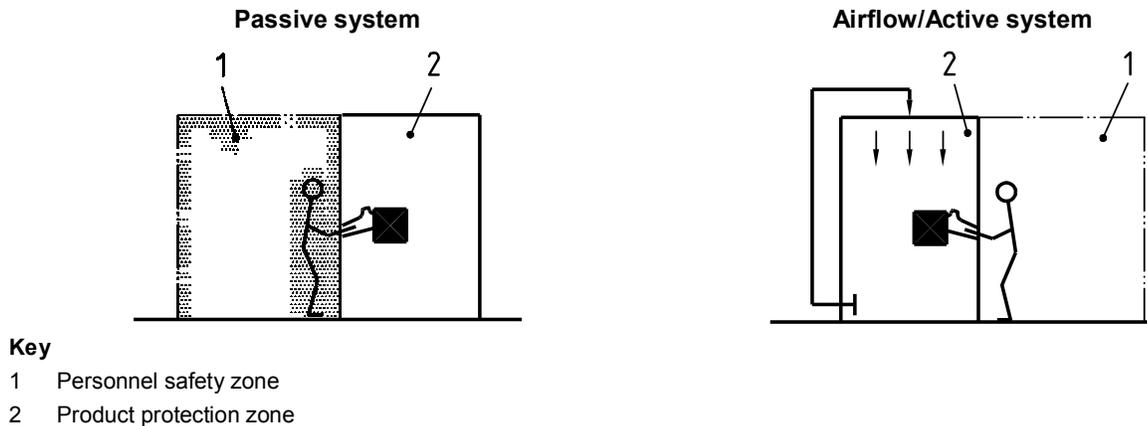
The transfer of contaminants into a zone protecting a process and/or personnel can be prevented by using aerodynamic measures, i.e. by arrangement and flow direction (Figure A.4), or by physical barriers, i.e. by both active and passive isolation (Figure A.5), if any contact between product and operator/environment is to be prevented.

If necessary, process exhaust should be treated to prevent contamination of outdoor environment.



NOTE In particular cases (e.g. dry atmosphere, shielding and protecting gas or extreme temperatures), the gas flow routing chosen should be adapted to the process.

**Figure A.4 — Contamination control concepts using aerodynamic measures**



**Figure A.5 — Contamination control concepts using physical segregation for product and personnel protection**

## A.5 Concepts to achieve segregation of cleanrooms and clean zones

### A.5.1 General

A suite of cleanrooms can consist of multiple rooms with different requirements for contamination control. The objective of the design can be to protect the product or process, or to contain the product, and in some cases a combination of these requirements. In order to protect cleanrooms from contamination from adjacent less clean spaces, the cleanroom should be maintained at a higher static pressure than the adjacent spaces, or alternatively a controlling air velocity should be established across the leakage paths between the spaces flowing from the cleaner to the less clean space. The converse can be applied to contain a hazard. In both cases, an impervious physical barrier can be used as an alternative.

The quantity of make-up air should be sufficient for ventilation purposes and to compensate for the leakage of air from the boundary of the cleanrooms or clean zones and any exhaust air for other purposes.

The following comparison of three basic concepts has been prepared to facilitate the selection of a suitable cleanroom or clean zone segregation concept.

### A.5.2 Displacement concept (low pressure differential, high airflow)

A low pressure differential can effectively separate clean and less clean adjacent zones, i.e. by means of a low turbulent "displacement" airflow, e.g. larger than 0,2 m/s (see Figure A.6).



**Figure A.6 — Displacement concept**

Displacement airflow velocity should be typically above 0,2 m/s, from the cleaner zones towards the less clean zones. The necessary airflow velocity should be selected considering important conditions such as physical obstacles, heat sources, exhausts and contamination sources.

### A.5.3 Pressure differential concept (high pressure differential, low airflow)

A pressure differential exists across the barrier between the cleaner zone towards the less clean zone. A high pressure differential between adjacent zones can be easily controlled but care is recommended to avoid unacceptable turbulence (see Figure A.7).

The pressure differential should be of sufficient magnitude and stable to prevent reversal of airflow direction from that intended. The pressure differential concept should be carefully considered, whether used alone or in combination with other contamination control techniques and concepts.

The pressure differential between adjacent cleanrooms or clean zones of different cleanliness level should lie typically in the range of 5 Pa to 20 Pa, to allow doors to be opened and to avoid unintended cross-flows due to turbulence.

The static pressure between cleanrooms of different class, and cleanrooms and unclassified areas can be established and maintained using various airflow balancing techniques. These include both active/automated and passive/manual systems that are configured to adjust the relative quantities of air that are delivered and removed from each space by the ducted air system, air transfer system and losses.

In situations when pressure differentials at the lower end of this range are accepted, special precautions should be taken to ensure accurate measurement of separating flow or pressure and to prove the stability of the installation.

NOTE Flow visualization, either experimentally or by computation, can be used to demonstrate both the effectiveness of the displacement flow concept and the pressure differential concept.

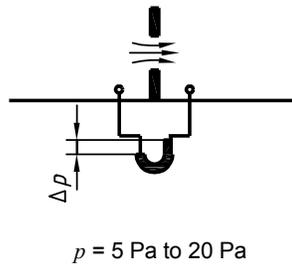


Figure A.7 — High pressure differential concept

### A.5.4 Physical barrier concept

This concept involves the use of an impervious barrier to prevent contamination transfer to a clean zone from a less clean zone.

NOTE All three concepts can be applied in the healthcare products, semiconductor, food and other industries.

## Annex B (informative)

### Classification examples

#### B.1 Healthcare products

For the manufacture of healthcare products, a frequently used correlation of typical manufacturing applications and cleanroom classification levels is given (see Table B.1). At the process core, the sterile product is filled through an aseptic assembly of components in a clean zone, controlled for particulate and microbiological contamination.

To access the process core, both the personnel and the process materials traverse several shells of increasing cleanliness (decreasing particulate concentrations). Personnel moving between various zones of different levels of cleanliness may change garments between zones, in accordance with the requirements of the zone that they are entering. Materials that enter each zone should be treated in a method appropriate to the level to be entered to remove particulate and/or microbiological contamination.

**Table B.1 — Cleanroom examples for aseptic processing of healthcare products**

| Air cleanliness class<br>(ISO Class)<br>in operation <sup>a</sup>  | Airflow<br>type <sup>b</sup> | Average,<br>airflow<br>velocity <sup>c</sup><br><br>m/s | Examples of<br>applications   |
|--|------------------------------|---|---|
| 5 (at W 0,5 m)   | U                            | > 0,2   | Aseptic processing <sup>d</sup>   |
| 7 (at W 0,5 m)   | N or M                       | na  | Other processing zones directly supporting aseptic processing               |
| 8 (at W 0,5 m)   | N or M                       | na  | Support zones of aseptic processing, including controlled preparation zones |
| NOTE 1 Application-specific classification requirements should take into account other relevant regulations.   |                              |   |   |
| NOTE 2 na = not applicable   |                              |   |   |
| <sup>a</sup> Occupancy states associated with the ISO Class should be defined and agreed in advance of establishing optimum design conditions.   |                              |   |   |
| <sup>b</sup> When airflow type is listed, it represents the airflow characteristics for cleanrooms of that class: U = unidirectional; N = non-unidirectional; M = mixed (combination of U and N).  |                              |   |   |
| <sup>c</sup> Average airflow velocity is the way that unidirectional airflow in cleanrooms is usually specified. The requirement on unidirectional airflow velocity will depend on specific application factors such as temperature, and configuration of the controlled space and the items to be protected. Displacement airflow velocity should be typically above 0,2 m/s. |                              |   |   |
| <sup>d</sup> Where operator protection is required to ensure safe handling of hazardous materials, the use of segregation concepts (see examples in annex A) or appropriate safety cabinets and devices should be considered.  |                              |   |   |

## B.2 Microelectronics

In the microelectronics industry, the minimum device feature size or film thickness dictates the target level of contamination control and the corresponding cleanliness class.

The cleanliness class with the lowest particle concentration is often selected with reference to the critical particle size. The critical particle size (often assumed to be 1/10th of the minimum feature size) is used to help select the required cleanliness classification for the cleanroom.

Determination of cleanroom or clean zone cleanliness for different process cores is based upon the probability of contamination and the potential for device failure.

For example, **photolithography** is a process which involves exposure of wafers to the environment with a high probability of contamination and also a very high potential for device failure when contamination occurs. Accordingly, protection in microelectronics for this type of risk often involves the use of physical barriers which protect process cores in order to lower particle concentrations or alter other process parameters (e.g. temperature, humidity, pressure).

**Work zones** are zones where wafers or die are handled by people and/or automated handling equipment, and the potential for contamination is high if the product is directly exposed to the environment. The most common responses for the protection of the product within work zones involve unidirectional flow, minimizing occupancy and production load per cubic meter of cleanroom, segregating personnel from exposed product(s) increasingly including barrier techniques. Work zones are most commonly separated from adjacent, less critical zones, by physical barriers and airflow.

**Utility zones** are zones where the non-operator interface portions of the wafer processing equipment are typically located. In the utility zones it is typical that work in progress is not exposed to the environment. The utility zone of a process core is usually adjacent to its corresponding work zone.

**Service zones** are zones where neither product nor process equipment are located, but service zones are sited next to work or utility zones to help separate the cleaner zones from the less clean zone (see Table B.2).

## B.3 Influence of cleanroom clothing

The number of personnel and the type of cleanroom clothing may require specific consideration with respect to particle emission (see relevant parts of this International Standard, e.g. ISO 14644-5).

Table B.2 — Examples for microelectronic cleanrooms

| Air cleanliness class <sup>a</sup><br>(ISO Class)<br>in operation | Airflow type <sup>b</sup> | Average, airflow velocity <sup>c</sup><br><br>m/s | Air changes per hour <sup>d</sup><br><br>m <sup>3</sup> /m <sup>2</sup> h | Examples of applications   |
|---|---------------------------|---|---|--|
| 2   | U                         | 0,3 to 0,5  | na  | Photolithography, semiconductor processing zone <sup>e</sup>   |
| 3   | U                         | 0,3 to 0,5  | na  | Work zones, semiconductor processing zone  |
| 4   | U                         | 0,3 to 0,5  | na  | Work zones, multilayer masks processing, fabrication of compact discs, semiconductor service zone, utility zones |
| 5   | U                         | 0,2 to 0,5  | na  | Work zones, multilayer masks processing, fabrication of compact discs, semiconductor service zone, utility zones |
| 6   | N or M <sup>f</sup>       | na  | 70 to 160   | Utility zones, multilayer processing, semiconductor service zones  |
| 7   | N or M                    | na  | 30 to 70  | Service zones, surface treatment   |
| 8   | N or M                    | na  | 10 to 20  | Service zones  |

NOTE na = not applicable

<sup>a</sup> Occupancy states associated with the ISO Class should be defined and agreed in advance of establishing optimum design conditions.

<sup>b</sup> When airflow type is listed, it represents the airflow characteristics for cleanrooms of that class: U = unidirectional; N = non-unidirectional; M = mixed (combination of U and N).

<sup>c</sup> Average airflow velocity is the way that unidirectional airflow in cleanrooms usually is specified. The requirement on unidirectional airflow velocity will depend on local parameters such as geometry and thermals. It is not necessarily the filter face velocity.

<sup>d</sup> Air changes per hour is the way that non-unidirectional and mixed airflow is specified. The suggested air changes are related to a room height of 3,0 meter.

<sup>e</sup> Impervious barrier techniques should be considered.

<sup>f</sup> With effective separation between contamination source and zones to be protected. Could be a physical or airflow barrier.

## **Annex C** (informative)

### **Approval of an installation**

#### **C.1 Test preparation and final cleaning**

Prior to carrying out any inspection, test or measurement procedure, running systems should be allowed time to reach stability; this period of time should be agreed upon in advance. Tests should be of sufficient duration to demonstrate consistent performance (see clause 4, and examples in annex H).

Prior to the fitting of filters and after cleaning as described in E.1.2/E.3.3 in annex E has been completed, all ducts, walls, ceilings, floors and installed fittings should be cleaned to remove contamination which could prejudice the classification of the cleanroom.

Following cleaning, the final filters should be fitted and the commissioning tests conducted to demonstrate compliance.

#### **C.2 Inspection, tests and approvals**

##### **C.2.1 General**

In order to demonstrate that an installation is complete in every respect and performs to meet all contamination control requirements included in clause 4, a specific range of inspections and tests should be carried out upon the installation in question. Typical activities are identified in C.2.2 to C.2.5 and Figure C.1 for graphic representation.

##### **C.2.2 Concept and design approval**

A check should be carried out to ensure that the concept, design, and developed details satisfy the agreements between the purchaser and supplier. Review should include at least:

- a) contamination control concept;
- b) layout of equipment;
- c) description of the installation;
- d) schemes and drawings;
- e) incorporation of all other agreed requirements.

##### **C.2.3 Construction and installation approval**

###### **C.2.3.1 Construction approval (at supplier's site)**

A check should be carried out to ensure that the components and assemblies comply with the design. The check should include at least the following items:

- a) inspection and testing for completeness and quality according to specification;

- b) approval for compliance with safety regulations, ergonomic requirements, relevant guidelines and normative regulations;
- c) approval of certificates.

#### **C.2.3.2 Installation approval (at the site of the installation)**

A check should be carried out to ensure that the construction of the installation complies with the design. The check should include in addition to C.2.3.1 at least the following items:

- a) completeness of the installation;
- b) interfaces with other suppliers;
- c) correct function of utilities and auxiliary equipment;
- d) calibration of all control, monitoring, warning and alarm systems;
- e) fitting and in-situ testing of final filters;
- f) proving the reserve capacity of the air treatment system;
- g) testing enclosure for leakage;
- h) confirming that the proportion of recirculation to make-up air complies with the design specification;
- i) surface cleanliness and suitability of the installation (see examples in annex E);
- j) spare parts package.

#### **C.2.4 Functional approval**

After having completed the checks and approvals according to C.2.3.2, at least the following functional tests should be performed:

- a) determine clean zone segregation;
- b) measure and record contamination control recovery time;
- c) determine ability to maintain temperature and relative humidity requirements;
- d) determine airborne particulate cleanliness class;
- e) where appropriate, determine particulate surface cleanliness and microbiological contamination levels;
- f) determine light and noise levels;
- g) demonstrate and record airflow patterns and air change rate if necessary.

#### **C.2.5 Operational approval (equipment installed in a manner agreed in advance)**

Certain of the previous tests may be repeated to determine compliance with the operational conditions, namely:

- a) confirm clean zone segregation regime;
- b) determine ability to maintain temperature and relative humidity;
- c) determine airborne particulate cleanliness class;

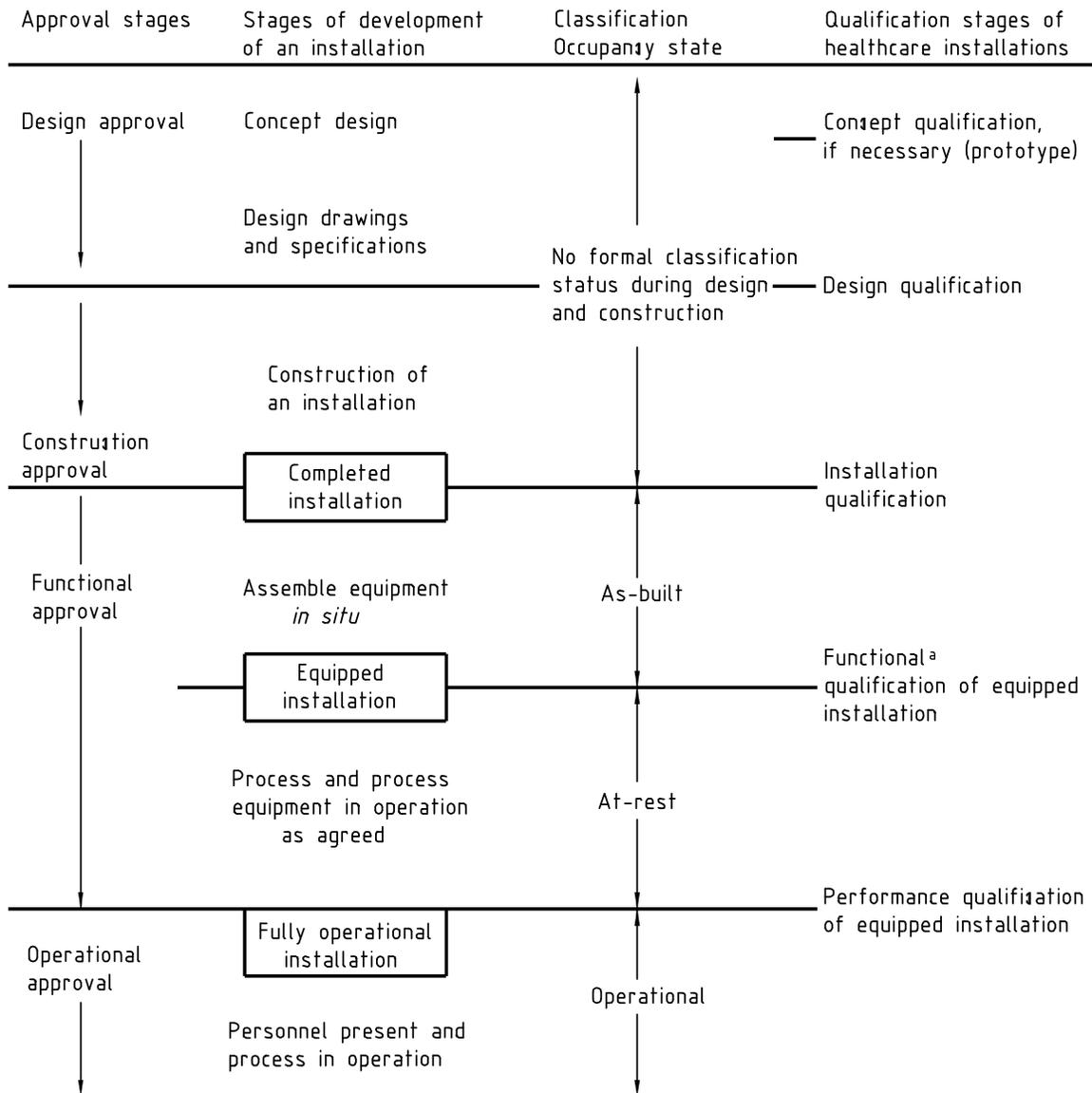
- d) where appropriate, determine particulate surface cleanliness and microbiological contamination levels;
- e) check the completeness of documentation according to clause 8.

For compliance-related issues, refer to ISO 14644-2; for microbiological-related issues, refer to ISO 14698-1, ISO 14698-2 and ISO 14698-3; for testing-related issues and for operational-related issues, refer to other relevant parts of this International Standard.

### **C.3 Reports**

The reports of the tests should be presented in a documented manual. This manual should include:

- a) supplier's test documentation;
- b) calibration certificates of instrumentation used;
- c) relevant drawings and as-installed details;
- d) witnessed verification of compliance with specification.



<sup>a</sup> Often used: operational.

**Figure C.1 — Approval of an installation**

Figure C.1 indicates a logical sequence for and relationship between approvals, stages of development and the formal classification occupancy state of an installation. Terminology may vary in specific industries, through established usage or regulatory requirement. Figure C.1 shows the qualification sequence frequently used in healthcare industry applications, in relation to the stages of construction and approval.

## Annex D (informative)

### Layout of an installation

#### D.1 General considerations

##### D.1.1 Size

The size of a cleanroom should be kept to the minimum practicable, allowing for any future requirements. In general, if a large amount of space is required, it should be divided into several zones or rooms, with or without physical barriers.

**NOTE** It is recognized that the presence of people, and activity, within a cleanroom can generate both contamination and disturbance of airflow. Annex B provides examples of installation configurations to control these phenomena. Annex A discusses contamination control concepts in which airflow and the physical configuration of a workstation or other critical discrete areas are managed to obviate or minimize exchange of contamination between product and its environment, including people in the immediate proximity.

##### D.1.2 Workstation siting and organization

Within the cleanroom, critical workstations or areas of risk should be sited away from entries and exits, major traffic pathways and other features which may cause disruption of the airflow pattern and higher levels of contamination.

In horizontal-flow cleanrooms, the siting of workstations should be such that the clean work which is to be performed receives clean air from the appropriate source, without flow disturbance or contamination from personnel movements or adjacent work.

When operations that require different degrees of cleanliness are to be carried out in an area swept by horizontal unidirectional airflow, less clean operations should be sited downstream of cleaner operations, insofar as it can be determined that this arrangement will not compromise the maintaining of target conditions for any critical points.

##### D.1.3 Ancillary areas and adjacent cleanrooms

Consideration should be given to the location and integration of ancillary areas such as service and utility, cleaning, preparation, toilet and refreshment facilities, in order to avoid compromise of the critical conditions maintained within the cleanrooms. Pressure or flow differentials, access and communication arrangements (such as airlocks, speech panels and intercoms), enclosure sealing (notably material joints, equipment and utility penetrations) should be executed so that cross-contamination from less clean zones does not compromise the cleaner zones. Layout should combine with effective training and management of personnel behaviour to minimize disturbance and cross-contamination due to movement between ancillary areas and cleanrooms.

##### D.1.4 Utility services and ancillary equipment

###### D.1.4.1 General

Utility services provided for the cleanroom should be designed, located and installed such that the cleanroom is not compromised by contamination from such services.

In general, exposed piping, tubing and cable runs within the cleanroom should be minimized, as these may present problems for adequate cleaning, and may be sources of damage by contact with cleanroom garments, wipes, etc. This should be balanced against the potential for contamination within protective housings, covers, etc., which may also hinder disinfection or fumigation. Where possible, consideration should be given to the routing of such services

in external service areas or ducts. Means should be provided for the effective removal of waste and contamination generated within such spaces.

Power take-off points, taps and connections should be designed and installed to facilitate regular cleaning, and to avoid the build-up of contamination in or behind blanking covers. Wherever possible, maintenance activities should be performed outside the cleanroom. Pressure or flow differentials, access arrangements (notably airlocks and transfer hatches), enclosure sealing (notably material joints, equipment and utility penetrations) should be executed so that cross-contamination from ancillary areas does not compromise the cleanroom.

The number, type and location of utility services should be agreed between the purchaser and supplier.

#### **D.1.4.2 Vacuum-cleaning equipment**

Vacuum-cleaning equipment, either portable or built-in, should be provided to ensure that particulate contamination can be removed during periodic cleaning, and to ensure that contamination generated by any operation that cannot reasonably be conducted outside the cleanroom can be removed efficiently, and with appropriate frequency.

Where a permanent vacuum-cleaning system is provided, the exhaust and fan should be sited outside the cleanroom. The connection sockets in the cleanroom should be blanked off when not in use. The airflow through the vacuum chamber should not compromise the differential pressure or the airflow configuration of the cleanroom.

When portable vacuum equipment is used, it should be fitted with an exhaust filter of at least the same efficiency as that filtering the environmental air supply, and care should be taken to consider the influence upon air patterns in the cleanroom.

#### **D.1.4.3 Sprinkler systems**

Fire control systems present special problems, notably in the routing of supply piping containing a fire suppressant medium, whether water, chemical substance or gas, which is a potential contaminant of the cleanrooms, and a potential source of damage to the components of the installation, in the event of accidental or deliberate release.

When sprinkler piping is to run above ceilings, careful consideration should be given to its routing, in relation to the equipment and operations sited in the cleanroom below. Adequate access should be provided for maintenance and modification, and consideration should be given to provision of means to collect and evacuate fluid leaked or released above the ceiling.

Penetration of walls or ceilings for supply to sprinkler points should be sealed as appropriate, like all other penetrations of the cleanroom. The sprinkler heads themselves should be situated and shaped for minimum intrusion into the cleanroom, and for minimal disturbance of clean airflow, insofar as this is compatible with their primary safety function. Where disturbance is inevitable, appropriate measures should be taken to avoid any undesirable effect upon the required integrity of the cleanroom conditions.

#### **D.1.5 Communication systems**

Wherever practical, communication systems should be provided in order to minimize movement of personnel into and out of the cleanroom. Windows, speech panels, intercoms, data links and telephones are suitable means of communication. They should be selected to be compatible with the cleanroom class and application considerations.

#### **D.1.6 Glazing**

Where windows to the outside are a requirement, care should be taken, in design and fitting, to avoid undue heat loss, solar gain and condensation. The use of windows to adjoining inside spaces should be considered, to allow observation of activity within the room, without entry. Windows should be non-opening and sealed. Double glazing can be used to achieve flush fitting, and also enables provision of interstitial shutters or blinds. The use of exposed blinds within a cleanroom should be avoided.

## **D.2 Access**

### **D.2.1 General**

The number of openings connecting the cleanroom to outside, or adjoining, areas should be minimized.

Effective means should be taken to minimize the contamination arising from the entry or exit of personnel or material, or from air movement. Normal (non-emergency) access to or from the cleanroom should be through airlocks for both personnel and material.

### **D.2.2 Airlocks**

In order to maintain pressure differential and integrity of the controlled space during entry and exit, airlocks or transfer hatches (pass-throughs) are normally required.

Precautions should be taken to ensure that entry and exit doors associated with an airlock are not opened simultaneously. Clear windows can be provided at both points to allow a line-of-sight view between them. Consideration should be given to the use of electrical or mechanical interlock systems including audio-visual indicators.

Barrier benches or other clear demarcation systems, together with appropriate decontamination devices and procedures, should be employed within an airlock system for the passage of material. The passage of material and personnel can be segregated.

### **D.2.3 Emergency exits**

Emergency exits should be provided with means to show that they have been opened.

### **D.2.4 Changing rooms**

#### **D.2.4.1 General**

Changing rooms are specialized airlocks for the entry and exit of personnel to and from a cleanroom. They should include sufficient space for their function, and, depending on the cleanroom quality, facilities for donning and removing specialized garments, and may include washing, disinfection facilities, etc. Special contamination control equipment such as air showers, shoe cleaners and adhesive floor materials may be provided at the point(s) of entry and exit to the cleanroom.

Separation of the personnel entering from those leaving the cleanroom via the changing room should be ensured. This can be achieved by separation in time, or by providing physically separate entry and exit routes.

Where hazardous materials are processed, a separate changing and decontamination route should be considered.

#### **D.2.4.2 Changing room control and configuration**

Changing rooms should be provided with a level of contamination control and environmental control that ensures the integrity of the cleanroom. Similarly, the methods and equipment for storage of garments and equipment for use in the cleanroom should be commensurate with the required cleanliness and contamination protection required by the contamination-sensitive operation. To provide the required protection, consideration should be given to three functional zones of the changing room:

- a) at the changing room entry: access from ancillary areas (either directly or via an airlock) appropriate for removal, storage, disposal and/or redonning of garments not permitted within the cleanroom;
- b) the transition zone: area where garments or personal equipment dedicated to the cleanroom are stored, donned or removed, as appropriate;

- c) the inspection/access zone: area where inspection of the completed gowning process is accomplished and which provides access to the cleanroom either directly or via an airlock.

The three functional zones may be separated by a physical barrier (e.g. a stepover bench or airlock) as appropriate to the operation and use of the changing room. The three zones should be established, such that the zone closest to the cleanroom provides a high degree of assurance, and that minimal adverse impact is caused by access or gowning procedures implemented in the adjacent zone.

#### **D.2.4.3 Facilities in changing rooms**

The features provided in the changing room are particular to the cleanroom that the changing room serves.

The following requirements should be defined:

- number of people passing through the gowning procedure, both in the absolute, and at any one time;

- the gowning procedure (i.e. what garments are to be taken off and put on, whether these are reusable or single-use, the required protocol to ensure garment cleanliness and to avoid cross-contamination);

- the frequency of garment replacement.

Consideration should be given to the following provisions in the changing room:

- a) storage and disposal of garments;
- b) storage before use, provision and disposal of consumable items and accessories (e.g. gloves, masks, protective glasses, overshoes);
- c) storage of personal items;
- d) hand-washing and -drying or other decontamination processes;
- e) prominent display or posting of gowning sequence, with clear instructions;
- f) full-length mirrors to check effective fit.

## **Annex E** (informative)

### **Construction and materials**

#### **E.1 Selection of materials**

##### **E.1.1 General**

The materials used in the construction of the installation should be selected and applied to meet the requirements of the installation, and should take into account the following:

- a) the cleanliness class;
- b) effects of abrasion and impact;
- c) cleaning and disinfection methods and frequencies;
- d) chemical/microbiological attack and corrosion.

Materials which may tend to break down or to shed particles should only be used when they are effectively encapsulated and protected.

Consideration should be given to the chemical compatibility of all materials used with the operating requirements of the installation. This may, for instance, influence the choice of adhesives and sealing mastics for surface-finishing work, or of materials used for filter assembly and sealing.

All surfaces which come into contact with air supplied to the interior of the cleanroom or clean zone may by their nature or condition influence the quality of the air supplied to the contamination-sensitive zones. For this reason, materials and finishes intended for the internal surfaces of the complete air-handling system should be critically assessed and specifically approved for this purpose.

All exposed surfaces of equipment, furnishings and material present within the cleanroom or clean zone should meet the same criteria as the exposed structural elements of the installation.

Further details of specific performance criteria follow.

##### **E.1.2 Surface cleanliness and cleanability of materials of construction**

All exposed materials should be suitable for effective and frequent cleaning and disinfection, and offer no surface asperities or porosity which are likely to allow retention of particulate and chemical contamination, or the development of microbiological contamination. Methods for selecting, applying and controlling suitable procedures for cleaning and disinfection are indicated in ISO 14698-1 and ISO 14698-3, and other relevant parts of this International Standard. Appropriate methods for assessing and monitoring surface cleanliness (for instance in terms of releasable particulate, biological and chemical contamination) should be selected and approved for the application. Exposed materials should be selected with due consideration of their resistance to the mechanical and chemical effects of the intended methods of cleaning and disinfection, in order to remain smooth, non-porous, abrasion- and stain-resistant (see also E.1.4 and E.3.3).

Walls, floors and ceilings in cleanrooms and in clean zones should be designed and constructed in such a way that the surfaces are accessible for cleaning. In a room, this generally includes the walls, floors, ceilings and doors, the inlet side of air diffusers and floor drain, etc. (see examples in annex G).

When it is necessary to wipe down or wash walls, floors or ceilings on a frequent basis, consideration of the selection of materials should include careful evaluation of the junction and intersection details, and in particular the avoidance of places where moisture can be trapped or lie on surfaces.

### E.1.3 Control of electrostatic charging and discharge

Accumulation of electrostatic charge, and subsequent electrostatic discharge, can present a risk of hazards such as explosion (in the presence of powders or gases), device damage (e.g. damage to electronic or optical components), or excessive attraction of particles to surfaces contributing to physical, chemical and microbiological contamination.

Where the above risks cause concern, materials used in the construction of installations should neither generate nor hold a significant static charge. This significant value will be specific to each application, and should be clearly specified by the purchaser. Certain processes may require particular conditions in terms of environmental humidity, in order to minimize the generation of electrostatic charge. Annex F provides further guidance on this technique. It should be noted that the most favourable humidity conditions for avoidance of electrostatic charge accumulation may conflict with other requirements of the process, or project objectives. A solution should be agreed, which achieves an acceptable compromise. Certain applications may require the use of conductive or static dissipative materials in order to minimize the influence of any induced static charge.

To protect electrostatically sensitive components the resistance to earth should be in the range of  $R_E = 10^4$  to  $10^7$  . Care should be taken to protect the personnel against risk of electrocution. Earthing should be considered, with a site transition resistance  $R_{ST} = 5 \cdot 10^4$  . The "ideal" range of resistance is therefore between the site transition resistance  $R_{ST} = 5 \cdot 10^4$  and the mass resistance  $R_E = 10^7$  .

The required electrical characteristics for flooring are valid for the entire structure or composite of materials used as a floor, and should be measured regularly to monitor potential loss of performance through ageing. Limit values of

2 kV (applicable to accumulated surface charge) should not be exceeded. Monitoring of wall conductivity should be carried out regularly and after modifications or repairs.

### E.1.4 Internal finishes, durability and maintainability

In the completed installation, all internal surfaces should be finished suitably smooth, non-porous and free from cracks, cavities, steps and ledges. The design and construction should be such that the number of steps, ledges, cavities and similar features where contamination could collect is minimized. The number of corners should also be kept to a minimum, particularly internal corners. Corners and junctions may be radiused, especially at floor-to-wall and wall-to-wall junctions, so that effective cleaning is facilitated. The finish should be compatible with the mechanical and chemical effects of the intended methods of cleaning and disinfection.

Materials used for internal finishes should be maintained to ensure that they consistently retain the performance qualities consistent with the cleanliness class of the installation. This may require regular maintenance procedures and repairs. Consideration of maintenance and repair methods and disruption impact should form part of the material selection criteria. Full lifecycle cost and contamination risk analysis procedures should be considered.

## E.2 Considerations for specific components

### E.2.1 Ceilings, walls and floors

#### E.2.1.1 Basic requirements

Wall, ceiling and floor elements should comply with all relevant regulations concerning fire protection, sound and thermal insulation. Surface finish and assembly details should be compatible with the specified cleaning methods. In order to avoid glare, consideration should be given to the interaction of surface colour and finish with the intended lighting conditions. Airlocks, gowning rooms and material passage points should normally have at least the same requirements as the cleaner of the zones they serve. In the case of equipment and material transfer airlocks, decontamination and "cleandown" procedures may impose special requirements.

NOTE There are many acceptable methods and materials for constructing cleanrooms ranging from *in situ* construction to fully prefabricated site-assembled systems. The basic options are summarized as follows:

a) Prefabricated site-assembled systems and *in situ* construction:

- 1) wet construction with applied surface finish,
- 2) dry construction with applied surface finish.

b) *In situ* assembly:

- 1) pre-finished engineered components,
- 2) modular pre-finished panel system.

Combinations of these basic construction options can also be used.

The choice of method of construction of an installation should take into account not only the contamination control and operational requirements, but also matters relating to the construction location (e.g. construction and finishing skills available); considerations influenced by the available building envelope in which the installation is located, such as available height, load-bearing capability, deflection of structures; maintenance constraints and requirements such as "walk-on-ceiling" capability, etc.

#### **E.2.1.2 Ceilings**

Ceilings should be sealed, to prevent ingress of air bearing particles, or other contaminants, from the ceiling void. Filters, filter frames, filter housings and diffusers mounted in the ceiling should be sealed. Penetration points (e.g. for utility services, sprinklers and lighting) should be kept to the minimum required, and be sealed. Consideration should be given to the location and configuration of components such as lights and sprinklers to avoid disturbance of the intended airflow.

#### **E.2.1.3 Walls and wall systems**

Materials and surface finishes should meet all general requirements for their application. Particular consideration should be given to impact and abrasion resistance, especially in those locations exposed to frequent passage of trolleys, carts or personnel carrying material likely to contact exposed surfaces of walls and doors. Suitable rubbing strips or protective bars may constitute satisfactory protection of otherwise vulnerable material.

Some applications may require that walls or wall panels be sealed to prevent exchange of contaminants with surrounding areas. Cover strips or seals between panels should be smooth, with rounded edges (some applications require flush fitting) to facilitate efficient cleaning and limit retention of contaminants. Particular attention should be paid to smoothness and effective sealing of utility services or other penetrations.

Where glazing is required, in walls or doors, it should be of the non-opening type. Consideration should be given to the use of double glazing, with airtight seal, which can enable flush mounting on both sides. If blinds or shutters are required, these should be fitted outside the clean zone, or between the glazed elements of double glazing. Glazing frames should be smooth. Where flush fitting is not required, rounded edges or sloping surfaces should be considered.

Doors should present as few horizontal surfaces as possible, with particular attention being paid to the minimization of steps and ledges in the door surface. Thresholds should be avoided. Consideration should be given to the minimization of abrasion in the mechanical elements of the door (e.g. latches, locks and hinges), and also between the door and its frame and the floor. Door handles, where required, should be smooth, non-snagging and easy to clean. Consideration should be given to the use of push plates, automatic openings, or appropriate door-swing direction where contamination transfer is a concern.

#### **E.2.1.4 Floors**

Floors or floor coverings should be non-porous, slip-resistant, abrasion-resistant, conductive if necessary; resistant to the chemicals they will encounter in use (both cleaning and disinfection products, and accidental spillage of process fluids) and easy to clean. The floor should support the specified static and dynamic loads with the required durability. The floor complex should provide the appropriate electrostatic characteristics.

#### **E.2.2 Air-handling systems**

Attention should be paid to minimizing the contamination generated, retained and released throughout the air-handling system, in all components and surfaces in contact with the system air, in order that an excessive load is not placed on the filtration system. Ducts should be manufactured from materials with corrosion-resistant and non-flaking properties, or should be given suitable surface treatment to prevent release of contaminants from the duct to the air passing through. If there is no terminal filter outlet provided, the quality and integrity of the system downstream of the final filter is more important. The effects of leakage from air-handling systems should be considered.

#### **E.2.3 Fittings in airlocks**

Fittings in airlocks and gowning rooms should present as few horizontal surfaces as possible. For example consideration should be given to the use of hanging rails and perforated shelf boards rather than closed lockers. Exposed surfaces should satisfy criteria similar to those specified for the interior of the cleanroom and clean zone, and may require additional specifications to ensure durability in this application.

#### **E.2.4 Ancillary areas**

These should have no direct connection to the cleanroom, except for emergency exits. Exposed surfaces in these areas should be chosen with a particular concern for durability and ease of maintenance.

### **E.3 Construction and assembly**

#### **E.3.1 General**

Construction work should comply with the drawings and specifications, and the agreed quality plan. Any changes required during the construction phase should be checked for acceptance, approved and documented prior to their implementation (see also examples in annex C).

#### **E.3.2 Material management during construction**

All components and materials for use in the construction and subsequent maintenance of the installation should be manufactured, packed, transported, stored and inspected before use in such a manner as to ensure their suitability for their intended use.

#### **E.3.3 Cleanliness and cleaning during construction and start-up**

Many tasks involved in construction and assembly intrinsically generate contamination. A clean construction protocol should be developed and enforced to satisfy and achieve the specified contamination control objectives. Particular attention should be paid to the scheduling of tasks which are the greatest sources of contamination, such that those tasks are accomplished before tasks which are lesser sources of contamination or more contamination-sensitive.

During construction, measures should be taken to ensure that contamination generated in the course of assembly and construction work is contained and removed, so as to limit undue contamination of surrounding areas. Appropriate means of containment may include the use of temporary screens and walls, and pressurization of critical zones, with provisional use of temporary "sacrificial" filters in the air-handling system(s). Such filters, installed to protect clean volumes (clean environment and air-handling systems) from outside contaminants, and to permit their initial pressurization and operation, are intended to be removed and replaced by filters of the appropriate grade at the agreed stage or stages of start-up, before construction approval and subsequent operational use of the installation.

Continual or frequent cleaning should be planned, undertaken and controlled as specified, with the aim of preventing undue build-up of contaminants in any part of the installation, and so facilitating the essential final cleaning before start-up (see also clause 6 and E.1.2).

It may be useful to effect initial cleaning of components, and those preparation or assembly tasks which it is not absolutely necessary to perform as part of definitive construction *in situ*, in a separate or intermediate zone between the point of reception on-site, and the final point of construction. Such procedures can contribute significantly to the reduction of contamination in all parts of the installation, though they are of special value where subsequent access and cleaning would be difficult or impossible.

#### E.4 Materials of construction

Typical surface materials are:

a) For walls and ceilings:

sheets of stainless steel;

anodized aluminium;

polymer sheets or coating.



mounted on appropriate substrates or construction

b) For floors:

polymer coating or sheets;

tiles with appropriate sealed joints.

Selection of materials should include consideration of the chemical, thermal and mechanical stresses during operation (production, setup, cleaning and decontamination as well as conductivity and outgassing characteristics). Additionally, flexibility, functionality, durability, aesthetics and maintainability should be considered by customer and supplier.

## **Annex F** (informative)

### **Environmental control of cleanrooms**

#### **F.1 Design**

**F.1.1** Requirements for environmental control vary with each application. Therefore the purchaser should state which criteria are important when specifying a cleanroom. The lists given in this annex are not exhaustive and should be supplemented as required.

**F.1.2** The design of the environmental systems should take into account the following:

- a) the contamination control concept chosen;
- b) product quality requirements;
- c) capital and operating costs (life cycle costing);
- d) energy conservation;
- e) safety;
- f) health and comfort of personnel;
- g) needs and constraints imposed by equipment and processes;
- h) reliability, ease of operation and maintenance;
- i) environmental issues (e.g. handling of waste and packaging);
- j) regulatory requirements.

#### **F.2 Temperature and humidity**

**F.2.1** The set point and variation limits of temperature (in degrees Celsius) and relative humidity (in percent saturation) which may depend on special process requirements should be specified for the performance of the cleanroom.

**F.2.2** Temperature control should be provided for:

- a) processes;
- b) equipment and materials;
- c) stable conditions for personnel wearing cleanroom garments selected to suit the class of cleanliness specified.

In general terms, heat loads from lighting are high and stable; personnel loads vary; the heat generated by process operations (e.g. heat-sealing, soldering, welding, heat-treating and heating pressure vessels) is usually high and variable.

**F.2.3** The large quantities of air required for contamination control facilitate the offsetting of internal heat gains at an acceptable rate of response from the temperature control system. However, areas of concentration of heat-

producing equipment and supply-air patterns should be analysed to determine the acceptability of resulting temperature gradients and contamination control.

**F.2.4** Humidity control should be provided for:

- a) manufacturing processes;
- b) equipment and materials;
- c) the reduction of electrostatic charges;
- d) personnel comfort in conjunction with temperature control mentioned above.

**F.2.5** In cleanroom installations, humidity control is affected more by external influences (such as weather changes) than by variations in moisture generation within the space. If processes involving evaporation should take place within the cleanroom installation, they should be confined within ventilated enclosures. Precautions should be taken to control static electricity effects. Some manufacturing processes (such as vacuum tube manufacture and tableting) require relative humidities (R.H.) lower than 35 %. As indicated in annex E, consideration should be given also to selection of materials which minimize electrostatic effects. If the humidity in a confined space is low, static charges may be higher than in an area with higher humidity.

**F.2.6** Temperature and humidity levels for personnel comfort should be defined for these specific installations. A typical set range for relative humidity is < 65 % R.H. to > 30 % R.H. Outside this range, suitable measures should be considered to meet process and personnel requirements. Specific guidance to adjust temperature specifications to cleanroom garments used is given in ISO 7730.

**F.2.7** The locations at which temperatures and relative humidities require to be measured should be specified.

**F.2.8** The outside conditions under which the system is required to operate should be specified taking into account the intended operational mode.

**F.2.9** The amount of heat and moisture generated in the cleanroom, the location of sources and the nature of their dynamic variation should be specified.

### **F.3 Lighting**

**F.3.1** The lighting levels and uniformities required within the various parts of the installation should be specified, together with the methods used to assess them.

**F.3.2** The colour rendering of light should be specified by the purchaser, as it has a significant effect on the comfort of personnel and, in many cases, the processes being carried out, especially photosensitive processes.

**F.3.3** The lighting system should be consistent with the effective operation of the cleanroom. Light fittings should have no areas from which contamination may be released. The use of sealed or flush fittings should be considered. For unidirectional airflow applications, the design and positioning of the light fitting and associated diffuser should be such as to minimize or negate turbulence. The light fittings should be serviceable in a manner such that the integrity of the cleanroom is not violated and excessive contamination is not produced. The effect of glare should be considered within the context of the work being carried out.

### **F.4 Noise and vibration**

#### **F.4.1 General**

Noise and vibration limits should be specified, if required, according to a specific process or other requirements. Consideration should be given to

- a) site selection: vibration, soils, and future site developments;
- b) structural design: cleanroom floor support, stiffness, isolation joints;
- c) mechanical design: equipment selection, system design, performance specifications, vibration isolation systems, noise control systems (internal and external);
- d) architectural layout: building and installation layout, plant areas, service systems.

#### **F.4.2 Sound pressure level**

The selected sound pressure level should be based on the requirements with regard to both the comfort and safety of the personnel and consideration of the background sound pressure level created in the environment (e.g. other equipment). A typical A-weighted sound pressure level range for cleanroom installations lies between 55 dB and 65 dB. Some applications may require lower levels or may tolerate higher levels. Noise control measurements should be carried out in accordance with ISO 3746.

#### **F.4.3 Mechanical vibration**

**F.4.3.1** Vibration is an important consideration in cleanroom installations, since it can have an adverse influence on processes, human comfort and service life of equipment and systems.

**F.4.3.2** Vibration in cleanrooms should be minimized, or the source isolated, using methods such as high quality fans and vibration control equipment.

**F.4.3.3** When vibration control is required, the permissible levels should be defined using ISO 1940-1 and ISO 10816-1.

#### **F.5 Energy conservation**

Consideration may be given to incorporating in the design energy conservation considerations, such as provisions to reduce or close down temperature and humidity control and to reduce airflow during periods in which there is no activity. The ability to recover operating conditions in a defined recovery period should be demonstrated.

## **Annex G** (informative)

### **Control of air cleanliness**

#### **G.1 Air filtration systems**

Air filtration systems including filter elements, mounting frames, housings, gaskets, sealants and clamping systems should be selected to suit both the cleanliness level required and the conditions associated with their use and installation test requirements in the system. Specific air filtration standards should be used for filter selection. Three basic stages of air filtration are recommended:

- a) prefiltering of the outside air to ensure adequate quality of air supply to the air conditioning plant;
- b) secondary filtering in the air conditioning plant to protect the final filters;
- c) final filtering before cleanroom supply.

#### **G.2 Secondary filtration**

It should be understood that unless adequate secondary filtration is provided before the final filters supplying cleanrooms, several problems may arise. These problems include the following:

- a) the desired class of air cleanliness may not be achieved;
- b) the high frequency of final filter changing may become unacceptable;
- c) undesirable particulate and microbiological contamination of the product may occur.

#### **G.3 Application**

The designer should evaluate the performance of the primary and secondary air filters used in cleanroom air conditioning systems to suit each application. Consideration should be given to the use of filters for chemical and molecular decontamination (e.g. activated carbon) and configurations for exhaust air filtration to protect the outdoor environment.

#### **G.4 Energy conservation**

For energy conservation reasons, airflow of the ventilation systems may be reduced to low levels during non-operating periods. If, however, they are turned off, the potential for unacceptable room contamination to occur should be considered.

#### **G.5 Temporary filters**

The installation of temporary filters should be considered to protect the air cleanliness of air-handling systems during construction and commissioning.

## **G.6 Packaging and transportation**

High-efficiency air filters should be packaged in a manner that adequately protects the element from mechanical damage during handling and transportation from the supplier. The filters should be inspected and be free from damage prior to fitting into the installation.

## **G.7 Fitting**

The fitting of high-efficiency filters should be delayed until they are required for commissioning purposes. Whilst awaiting fitting, filters should be stored in accordance with the supplier's instructions. Immediately prior to fitting, the air ducting system should be visibly clean and free from contamination. The filters should be fitted in accordance with the manufacturer's instructions.

## **G.8 Testing**

All air filtration equipment installed in an installation should allow for leak-testing of the final filters and integrity-testing of the seals between filter and mounting arrangements. Consideration should be given to the materials used for such testing to ensure that materials themselves do not become contaminants or cause contamination.

## Annex H (informative)

### Additional specification of requirements to be agreed upon between purchaser/user and designer/supplier

#### H.1 General

This annex is intended to assist the purchaser/user and designer/supplier to communicate and agree on additional requirements. It is intended that the checklist be used to define known requirements and identify aspects where further development is required.

#### H.2 Checklists

Checklists are given in the form of tables.

Table H.1 suggests a check for process requirements which affect the installation.

Table H.2 suggests a check for contaminants which detrimentally affect the process.

Table H.3 suggests a check for all pieces of equipment to be utilized in the process.

Table H.4 suggests a check for all external factors affecting the process.

Table H.5 suggests a check for environmental requirements affecting the process.

Table H.6 suggests a check to identify requirements for safe operation.

Table H.7 suggests a check to evaluate the requirements for systems redundancy (standby/backup).

Table H.8 suggests a check for the scope of equipment maintenance required.

Table H.9 suggests a check for miscellaneous requirements not previously defined that affect design, construction, operation and maintenance.

Tables H.10, H.11 and H.12 suggest checks for factors affecting future developments, cost requirements and scheduling, respectively.

**Table H.1 — Process requirements**

| Number | Item               | Description  | Specified value | Achieved performance |
|--------|--------------------|--|-----------------|----------------------|
| 1      | Direct processes   | Those which directly affect the end product or service.              |                 |                      |
| 2      | Indirect processes | Those which support or indirectly affect the end product or service. |                 |                      |

Table H.2 — Process contaminants

| Number     | Item                         | Description  | Specified value | Achieved performance |
|------------|------------------------------|--|-----------------|----------------------|
| <b>1</b>   | <b>Matter as contaminant</b> | Non-viable or viable matter  |                 |                      |
| <b>1.1</b> | <b>Particulate</b>           | Particles of different shape   |                 |                      |
| 1.1.1      | Class                        | In accordance with ISO 14644-1   |                 |                      |
| 1.1.2      | Size(s)                      | Particle size(s), M- and U-Descriptors (see annex E in ISO 14644-1:1999)/Basic, ultrafine, macroparticles and fibres |                 |                      |
| 1.1.3      | Recovery time                |  |                 |                      |
| <b>1.2</b> | <b>Chemical</b>              | Molecular, ionic, gaseous, condensable, metallic   |                 |                      |
| 1.2.1      | Amount                       | Quantity of chemical contamination/weight, layer(s), concentration   |                 |                      |
| 1.2.2      | Class                        | In accordance with ISO 14644-1 or other standard   |                 |                      |
| 1.2.3      | Recovery time                |  |                 |                      |
| <b>1.3</b> | <b>Biological</b>            | Viable, aerobic or non-viable pathogenic organisms/organisms capable of reproducing                                  |                 |                      |
| 1.3.1      | General type                 | Bacteria, fungi, other   |                 |                      |
| 1.3.2      | Contamination type           | Aggressive to surfaces, resistant to disinfection, pathogenicity   |                 |                      |
| 1.3.3      | Propagation                  | Duration from upset to steady state  |                 |                      |
| <b>2</b>   | <b>Energy as contaminant</b> | Energy sources which interfere   |                 |                      |
| <b>2.1</b> | <b>Vibration</b>             | Extent of motion   |                 |                      |
| 2.1.1      | Amplitude                    | Greatest displacement  |                 |                      |
| 2.1.2      | Frequency                    | Rate of motion   |                 |                      |
| <b>2.2</b> | <b>Magnetic</b>              | Electromagnetic fields   |                 |                      |
| 2.2.1      | Field strength               |  |                 |                      |
| <b>2.3</b> | <b>Radio frequency</b>       |  |                 |                      |
| 2.3.1      | Field strength               |  |                 |                      |

Table H.3 — Process equipment specification

| Number | Item                                       | Description   | Specified value | Achieved performance |
|--------|--|---|-----------------|----------------------|
| 1      | <b>Input utilities</b>                     | Matter and energy required to be delivered to each process equipment  |                 |                      |
| 1.1    | <b>Solids — Supply requirements</b>        | List equipment solids to be utilized in the process   |                 |                      |
| 1.1.1  | Solids supply purities/<br>Concentrations  | List, for each piece of equipment, for purities/concentrations required for all solids to be utilized in the process  |                 |                      |
| 1.1.2  | Solids supply quantities                   | List, for each piece of equipment, the quantities of all solids to be utilized in the process, including the maximum, minimum, and nominal rates of introduction and utilization            |                 |                      |
| 1.2    | <b>Gases — Supply requirements</b>         | List, for each piece of equipment, all gases to be utilized in the process  |                 |                      |
| 1.2.1  | Gases supply purities                      | List, for each piece of equipment, the purities required for all gases to be utilized in the process  |                 |                      |
| 1.2.2  | Gases supply quantities                    | List, for each piece of equipment, the quantities of all gases to be utilized in the process, including the maximum, minimum, and nominal rates of introduction and utilization             |                 |                      |
| 1.2.3  | Pressures                                  | List, for each piece of equipment, the pressures of all gases to be utilized in the process, including the maximum, minimum, and nominal rates of introduction and utilization              |                 |                      |
| 1.3    | <b>Liquids — Supply requirements</b>       | List, for each piece of equipment, all liquids to be utilized in the process  |                 |                      |
| 1.3.1  | Liquids supply purities/<br>Concentrations | List, for each piece of equipment, the purities/concentrations required for all liquids to be utilized in the process   |                 |                      |
| 1.3.2  | Liquids supply quantities                  | List, for each piece of equipment, the quantities required for all liquids to be utilized in the process, including the maximum, minimum, and nominal rates of introduction and utilization |                 |                      |

| Number     | Item  | Description   | Specified value | Achieved performance |
|------------|---|---|-----------------|----------------------|
| 1.3.3      | Liquids supply pressures                            | List, for each piece of equipment, the pressures for all liquids to be utilized in the process, including the maximum, minimum, and nominal rates of introduction and utilization           |                 |                      |
| <b>1.4</b> | <b>Electric power requirements</b>                  | List, for each piece of equipment, the electric power requirements  |                 |                      |
| 1.4.1      | Voltage   |   |                 |                      |
| 1.4.2      | Phase   |   |                 |                      |
| 1.4.3      | Frequency   |   |                 |                      |
| 1.4.4      | Load  |   |                 |                      |
| 1.4.5      | Allowable electrical power fluctuation requirements | List, for each piece of equipment, the maximum allowable fluctuation in electrical service that can be accepted without electrical power filtration   |                 |                      |
| <b>2</b>   | <b>Output utilities</b>                             |   |                 |                      |
| <b>2.1</b> | <b>Solid waste requirements</b>                     | List, for each piece of equipment, all solids to be rejected in the process   |                 |                      |
| 2.1.1      | Solids waste purities/<br>Concentrations            | List, for each piece of equipment, the purities/concentrations of all solids to be rejected in the process  |                 |                      |
| 2.1.2      | Solids waste quantities                             | List, for each piece of equipment, the quantities of all solids to be rejected in the process, including the maximum, minimum, and nominal rates of rejection                               |                 |                      |
| <b>2.2</b> | <b>Exhaust flow requirements</b>                    | List, for each piece of equipment, all types of exhaust to be utilized in the process   |                 |                      |
| 2.2.1      | Exhaust flow characteristics                        | List, for each piece of equipment, the types of exhaust flows (e.g. acid, solvent, heat, general, etc.) to be utilized in the process and their respective concentrations, and temperatures |                 |                      |
| 2.2.2      | Exhaust flow quantities                             | List, for each piece of equipment, the quantities of all exhaust flows to be utilized in the process, including the maximum, minimum, and nominal rates of introduction and utilization     |                 |                      |

| Number     | Item                                       | Description   | Specified value | Achieved performance |
|------------|--|---|-----------------|----------------------|
| 2.2.3      | Exhaust flow pressures                     | List, for each piece of equipment, the pressures of all exhaust flows to be utilized in the process, including the maximum, minimum, and nominal rates of introduction and utilization                  |                 |                      |
| <b>2.3</b> | <b>Liquid waste requirements</b>           | List, for each piece of equipment, all liquids to be rejected in the process  |                 |                      |
| 2.3.1      | Liquid waste quantities                    | List, for each piece of equipment, the quantities of all liquids to be rejected in the process, including the maximum, minimum, and nominal rates of introduction and utilization                       |                 |                      |
| <b>3</b>   | <b>Environmental parameters</b>            | To allow intended use of the process equipment  |                 |                      |
| <b>3.1</b> | <b>Temperature requirements</b>            | List, for each piece of equipment, the maximum, minimum, and optimum temperature requirement, both internal and external to the equipment. Further list by equipment component separately, as required. |                 |                      |
| 3.1.1      | Rate of temperature rise                   | List, for each piece of equipment, the maximum allowable rate of temperature rise   |                 |                      |
| 3.1.2      | Rate of temperature fall                   | List, for each piece of equipment, the maximum allowable rate of temperature fall   |                 |                      |
| <b>3.2</b> | <b>Humidity requirements</b>               | List, for each piece of equipment, the maximum, minimum, and optimum humidity requirement, both internal and external to the equipment components as required separately                                |                 |                      |
| 3.2.1      | Rate of humidity rise                      | List, for each piece of equipment, the maximum allowable rate of humidity rise  |                 |                      |
| 3.2.2      | Rate of humidity fall                      | List, for each piece of equipment, the maximum allowable rate of humidity fall  |                 |                      |
| <b>3.3</b> | <b>Vibration requirements/ Limitations</b> | List, for each piece of equipment, the maximum, minimum, and nominal vibration energy level   |                 |                      |
| <b>3.4</b> | <b>Physical barrier applied</b>            | Are they required?  |                 |                      |

| Number | Item                                | Description   | Specified value | Achieved performance |
|--------|-------------------------------------|---|-----------------|----------------------|
| 4      | <b>Physical attributes</b>          | Equipment dimensions and mass                                 |                 |                      |
| 5      | <b>Installation considerations</b>  | How to install  |                 |                      |
| 6      | <b>Operational considerations</b>   | How to operate  |                 |                      |
| 7      | <b>Maintenance considerations</b>   | How to maintain   |                 |                      |
| 8      | <b>Pre-process</b>                  | Status of incoming product or starting materials              |                 |                      |
| 9      | <b>Post-process</b>                 | Description of subsequent manufacturing steps                 |                 |                      |
| 10     | <b>Process throughput</b>           | The amount of product passing through the equipment over time |                 |                      |
| 11     | <b>Communication considerations</b> | Describe  |                 |                      |
| 12     | <b>Ergonomic considerations</b>     | Describe  |                 |                      |

Table H.4 — External factors

| Number | Item                                 | Description  | Specified value | Achieved performance |
|--------|--------------------------------------|--|-----------------|----------------------|
| 1      | <b>Regulatory requirements</b>       | List all regulatory factors affecting site selection and operations, including local zoning laws and ordinances, local tax structures, and permitting requirements |                 |                      |
| 2      | <b>Utility resources and factors</b> | List utility resources, including availability, quality, and quantities  |                 |                      |
| 2.1    | Site water supply                    | List the characteristics of local ground or municipal water supply, including toxicity, turbidity, etc.  |                 |                      |
| 2.2    | Site air quality                     | List existing site air quality characteristics   |                 |                      |
| 2.3    | Site electrical power factors        | List the local electrical power supply characteristics, i.e. capacity, voltage, number of phases, frequency, and intensity and frequency of fluctuations, etc.     |                 |                      |
| 2.4    | Site waste systems factors           | List the local waste system characteristics  |                 |                      |

|   |                                       |   |  |  |
|---|---------------------------------------|---|--|--|
| 3 | <b>Site vibration characteristics</b> | Evaluate the ambient site vibration level and its variations. Evaluate for potential impacts on planned processes and facilities                            |  |  |
| 4 | <b>Proximity factors</b>              | List all proximate and adjacent site structures, processes, pollutants, etc. Evaluate for potential impacts on planned processes, facilities, and personnel |  |  |
| 5 | <b>Site geotechnical factors</b>      | List all geotechnical factors, i.e. soils toxicity, soils expansion, characteristics, etc. Evaluate affect on planned installation                          |  |  |
| 6 | <b>Security and access factors</b>    | List all security and accessibility factors. Evaluate for affect on installation.   |  |  |

Table H.5 — Environmental requirements

| Number | Item                                     | Description   | Specified value | Achieved performance |
|--------|--|---|-----------------|----------------------|
| 1      | <b>Ambient requirements</b>              | Consider for process, equipment, and personnel requirements. List initially by cleanliness hierarchy. List each process area by cleanliness classification only if the design process is substantially developed. |                 |                      |
| 1.1    | Cleanliness                              | Required cleanliness classification   |                 |                      |
| 1.2    | Air pattern type                         | List the cleanroom air pattern type, i.e. unidirectional, non-unidirectional, or mixed  |                 |                      |
| 1.3    | Airflow direction                        | List the cleanroom airflow direction, i.e. vertical or horizontal   |                 |                      |
| 1.4    | Air velocity                             | List the cleanroom air velocity within the process area   |                 |                      |
| 1.5    | Air circulation system and configuration | Evaluate the cleanroom air circulation system configuration. Consider process, regulatory, personnel and budgetary factors  |                 |                      |
| 1.6    | Dry bulb temperature                     | Evaluate the cleanroom dry bulb temperature requirement, including the maximum, minimum and nominal value   |                 |                      |
| 1.6.1  | Rate of dry bulb temperature rise        | List the cleanroom maximum allowable rate of dry bulb temperature rise  |                 |                      |

| Number   | Item                                | Description   | Specified value | Achieved performance |
|----------|-------------------------------------|---|-----------------|----------------------|
| 1.6.2    | Rate of dry bulb temperature fall   | List the cleanroom maximum allowable rate of dry bulb temperature fall  |                 |                      |
| 1.7      | Humidity                            | Evaluate the cleanroom humidity requirement, including the maximum, minimum, and nominal value                        |                 |                      |
| 1.7.1    | Rate of humidity rise               | List the cleanroom maximum allowable rate of humidity rise  |                 |                      |
| 1.7.2    | Rate of humidity fall               | List the cleanroom maximum allowable rate of humidity fall  |                 |                      |
| 1.8      | Pressurization                      | List the cleanroom pressure   |                 |                      |
| 1.8.1    | Pressurization differential         | List the cleanroom pressurization differential from zone of higher space pressure to adjacent zone of lesser pressure |                 |                      |
| 1.8.2    | Pressurization rate of change       | List the cleanroom maximum allowable rate of change in space pressure   |                 |                      |
| <b>2</b> | <b>Sound pressure level (noise)</b> | List the cleanroom maximum allowable and nominal sound pressure levels  |                 |                      |
| <b>3</b> | <b>Vibration</b>                    | List the cleanroom maximum allowable and nominal vibration energy level   |                 |                      |
| <b>4</b> | <b>Lighting</b>                     | List the minimum and nominal cleanroom lighting requirements, and any wavelength restrictions                         |                 |                      |
| <b>5</b> | <b>Physical geometry</b>            | List the dimension/size requirements  |                 |                      |
| 5.1      | Ceiling-to-floor height             | List the cleanroom ceiling-to-floor height requirement  |                 |                      |
| 5.2      | Floor area requirement              | List the cleanroom floor area requirement, i.e. length and breadth  |                 |                      |
| 5.3      | Floor loading                       | Maximum mass loading  |                 |                      |
| <b>6</b> | <b>Ionization</b>                   | Charge balance (air)  |                 |                      |

Table H.6 — Safety requirements

| Number | Item  | Description  | Specified value | Achieved performance |
|--------|---|--|-----------------|----------------------|
| 1      | Cleanroom life-safety requirements                                | Identify all safety codes and regulations that affect the installation     |                 |                      |
| 2      | Separation of air circulation zones                               | Evaluate specific requirements for individual zone control and segregation |                 |                      |
| 3      | Storage and transport of toxic, flammable and hazardous materials | Evaluate specific process and overall storage requirements                 |                 |                      |
| 4      | Exiting requirements  | Evaluate maximum exit distance requirements                                |                 |                      |
| 5      | Physical requirements   | Evaluate requirements for fire resistive materials and assemblies          |                 |                      |
| 6      | Purge system  | Is one required?   |                 |                      |
| 6.1    | Flowrate  | At what rate?  |                 |                      |

Table H.7 — Standby/backup requirements

| Number | Item                            | Description                  | Specified value | Achieved performance |
|--------|---------------------------------|------------------------------|-----------------|----------------------|
| 1      | System duplication              | 100 % replacement capability |                 |                      |
| 2      | System oversizing               | More available than required |                 |                      |
| 3      | Largest component backup        | Replace 100 % of single      |                 |                      |
| 4      | Alternative source              | Switch over to alternative   |                 |                      |
| 5      | Failure detection and reporting |                              |                 |                      |
| 6      | Change-over methodology         | Manual or automatic          |                 |                      |

Table H.8 — Operations and maintenance factors

| Number | Item                     | Description                | Specified value | Achieved performance |
|--------|--------------------------|----------------------------|-----------------|----------------------|
| 1      | MTBF                     | Mean time between failures |                 |                      |
| 2      | MTTR                     | Mean time to repair        |                 |                      |
| 3      | Maximum time to repair   | How long to fix?           |                 |                      |
| 4      | Spare parts availability | How many, what type?       |                 |                      |

Table H.9 — Personnel factors affecting people and productivity

| Number | Item                                      | Description   | Specified value | Achieved performance |
|--------|---|---|-----------------|----------------------|
| 1      | Personnel and materials flow requirements | Evaluate product and process flow requirements and personnel flow requirements. Evaluate distances between individual processes and their functional interdependencies. Evaluate personnel communications and access needs. |                 |                      |
| 1.1    | Airlocks                                  | Required?   |                 |                      |
| 1.2    | Gowning requirements                      | What type of gown(s)  |                 |                      |
| 2      | Operating frequency                       | List the operating frequency of the cleanroom, i.e. continuous versus intermittent. If intermittent, specify frequency of operation, e.g. 5 days per week, 8 h per day  |                 |                      |
| 3      | Ergonomics                                | Any requirements  |                 |                      |
| 4      | Aesthetics                                | Any requirements  |                 |                      |

Table H.10 — Future developments

| Number | Item        | Description               | Specified value | Achieved performance |
|--------|-------------|---------------------------|-----------------|----------------------|
| 1      | Future      | Planning to consider now? |                 |                      |
| 2      | Flexibility | Planning to consider now? |                 |                      |

Table H.11 — Cost requirements

| Number | Item              | Description                             | Specified value | Achieved performance |
|--------|-------------------|---|-----------------|----------------------|
| 1      | Capital cost      | First cost                              |                 |                      |
| 2      | Operating cost    |   |                 |                      |
| 2.1    | Energy use        | Identify ways to reduce operating costs |                 |                      |
| 2.2    | Maintenance costs |   |                 |                      |
| 3      | Life cycle cost   | Owning cost                             |                 |                      |

Table H.12 — Schedule

| Number | Item                | Description   | Specified value | Achieved performance |
|--------|---------------------|---|-----------------|----------------------|
| 1      | Task definition     | Project tasks shall be agreed between the user and supplier           |                 |                      |
| 2      | Identify milestones | Identify or define key project milestones and the acceptance criteria |                 |                      |

### H.3 Specification checklist of basic requirements for cleanroom projects

Purpose: The purpose of this form is to help the user and supplier of the cleanroom project to document the essential and non-essential aspects of the cleanroom project. This form should be used in conjunction with the normative and informative clauses of this part of ISO 14644.

Project Name: \_\_\_\_\_ Project Location: \_\_\_\_\_  
Customer Name: \_\_\_\_\_ Supplier Name: \_\_\_\_\_  
Customer Contact: \_\_\_\_\_ Supplier Contact: \_\_\_\_\_  
Customer Phone No. \_\_\_\_\_ Supplier Phone No. \_\_\_\_\_  
Date: \_\_\_\_\_

## H.4 Relation to clause 4

Table H.13 — Relation to clause 4

| Clause 4 reference | Description of requirement  | Response, requirement, specification |
|--------------------|---|--------------------------------------|
| 4.2                | What is the number of the International Standard being referenced?  |                                      |
| 4.2                | What is the date of publication of this International Standard?   |                                      |
| 4.4                | What is the general purpose for which the controlled space is to be used?   |                                      |
| 4.4                | What are the operations to be carried out in the cleanroom?   |                                      |
| 4.4                | Are there any constraints imposed by the operating criteria (see examples in annexes A, B and D)?   |                                      |
| 4.5                | What are the required classes or demands for cleanliness in accordance with the relevant parts of this International Standard (ISO 14664-1, ISO 14698-1, ISO 14698-2, ISO 14698-3) (see examples in annex F)?   |                                      |
| 4.6                | What environmental parameters will be measured for validation purposes? What are the allowable variations, measurement method(s), and calibration method(s) (ISO 14644-2 and ISO 14644-3) (see examples in annex F)?  |                                      |
| 4.7                | Describe the contamination control concept to be used to achieve the required cleanliness level (including operating and performance criteria) (see examples in annex A for description of control concepts).   |                                      |
| 4.9                | What is the material flow through the cleanroom (see examples in annex D)?  |                                      |
| 4.10               | What are the occupancy state(s) under which the required conditions shall be achieved and maintained, including variations with time, and the methods of control of occupants, including e.g. gowning, sanitation techniques, personnel flow and access control to all clean areas (see examples in annex C)? |                                      |
| 4.11               | Provide layout and configuration drawings of the installation (see examples in annex D).  |                                      |
| 4.12               | Provide all critical dimensions and mass restrictions, including those related to available space (see examples in annex D).  |                                      |
| 4.13/4.14          | The process and product equipment to be installed in the cleanrooms or clean zones, including usage, method of gaining access for construction and maintenance, emissions, size and mass, and utility requirements (see examples in annexes B, D, E, G and H).  |                                      |

| Clause 4 reference | Description of requirement  | Response, requirement, specification |
|--------------------|---|--------------------------------------|
| 4.15               | The maintenance requirements of the system components creating the cleanroom or clean zone shall be supplied in a timely manner (see examples in annexes D and E).  |                                      |
| 4.16               | Provide the definition of all responsibilities for statement of criteria, basis of design, detailed design, construction, testing, commissioning and qualification (including the performance and witnessing) of tests (see examples in annexes E and G). |                                      |
| 4.17               | Identify all external environmental influences, such as chemical and particle contamination, noise and vibration (see examples in annex H).   |                                      |

## Bibliography

- [1] ISO 1940-1:1986, *Mechanical vibration — Balance quality requirements of rigid rotors — Part 1: Determination of permissible residual unbalance.*
- [2] ISO 3746:1995 + Technical Corrigendum 1:1995, *Acoustics — Determination of sound power levels of noise sources using sound pressure — Survey method using an enveloping measurement surface over a reflecting plane.*
- [3] ISO 7730:1994, *Moderate thermal environments — Determination of the PMV and PPD indices and specification of the conditions for thermal comfort.*
- [4] ISO 9000:2000, *Quality management systems — Fundamentals and vocabulary.*
- [5] ISO 9001:2000, *Quality management systems — Requirements.*
- [6] ISO 9004-1:1994, *Quality management and quality system elements — Part 1: Guidelines.*
- [7] ISO 10816-1:1995, *Mechanical vibration — Evaluation of machine vibration by measurements on non-rotating parts — Part 1: General guidelines.*
- [8] ISO 14001:1996, *Environmental management systems — Specification with guidance for use.*
- [9] ISO 14004:1996, *Environmental management systems — General guidelines on principles, systems and supporting techniques.*
- [10] EN 779:1993, *Particulate air filters for general ventilation — Requirements, testing, marking.*
- [11] EN 1822-1:1998, *High efficiency air filters (HEPA and ULPA) — Part 1: Classification, performance testing, marking.*
- [12] EN 1822-2:1998, *High efficiency air filters (HEPA and ULPA) — Part 2: Aerosol production, measuring equipment, particle counting statistics.*
- [13] EN 1822-3:1998, *High efficiency air filters (HEPA and ULPA) — Part 3: Testing flat sheet filter media.*
- [14] EN 1822-4:1997, *High efficiency air filters (HEPA and ULPA) — Part 4: Testing filter elements for leaks (scan method).*
- [15] EN 1822-5:1996, *High efficiency air filters (HEPA and ULPA) — Part 5: Testing the efficiency of the filter element.*
- [16] IEST-RP-CC001.3:1993, *HEPA and ULPA filters.* Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [17] IEST-RP-CC007.1:1992, *Testing ULPA filters.* Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [18] IEST-RP-CC012.1:1993, *Considerations in cleanrooms design.* Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [19] IEST-RP-CC021.1:1995, *Testing HEPA and ULPA filter media.* Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [20] IEST-RP-CC024.1:1994, *Measuring and reporting vibration in microelectronics facilities.* Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.



[21] US Pharmacopeia 23-NF 18 (1995) Supplement 8 (May 15, 1998) P4426 (1116), *Microbiological evaluation of cleanrooms and other controlled environments*.

[22] VDI 2083 part 2:1996, *Cleanroom technology — Construction, operation and maintenance*. Berlin: Beuth Verlag GmbH.

[23] VDI 2083 part 4:1996, *Cleanroom technology — Surface cleanliness*. Berlin: Beuth Verlag GmbH.

#### **Major multi-national cleanroom relevant standards/recommendations**

[24] *EC Guide to GMP for medicinal products*. Brussels: European Commission, 1995.

[25] ISO 13408-1:1998, *Aseptic processing of health care products — Part 1: General requirements*.

#### **Surveys of contamination control standards/recommendations**

[26] IEST-RD-CC009.2:1993, *Compendium of standards, practices, methods, and similar documents relating to contamination control*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.

#### **Major contamination control handbooks**

[27] TOLLIVER, D.L. (ed.): *Handbook of contamination control in microelectronics*. Park Ridge (New Jersey): Noyes Publications, 1988, 488 pp.

[28] WHYTE, W. (ed.): *Cleanroom design*. Wiley, Chichester, 1991, 357 pp.

[29] HAUPTMANN-HOHMANN (eds.): *Handbook of cleanroom practice*. Ecomed Verlag, Landsberg, 1992.

[30] LIEBERMANN, A: *Contamination control and cleanrooms*. Van Nostrand Reinhold, New York, 1992, 304 pp.

#### **Dictionaries for contamination control terms**

[31] IEST-RD-CC011.2:1996, *A glossary of terms and definitions relating to contamination control*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.

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## **DRAFT INTERNATIONAL STANDARD ISO/DIS 14644-5**

ISO/TC 209

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

# Cleanrooms and associated controlled environments —

## Part 5: Operations

*Salles propres et environnements maîtrisés apparentés —*

*Partie 5: Exploitation*

ICS 13.040.30

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 14644 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 14644-5 was prepared by Technical Committee ISO/TC 209, *Cleanrooms and associated controlled environments*.

ISO 14644 consists of the following parts, under the general title *Cleanrooms and associated controlled environments*:

- *Part 1: Classification of air cleanliness*
- *Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1*
- *Part 3: Metrology and test methods*
- *Part 4: Design, construction and start up*
- *Part 5: Operations*
- *Part 6: Vocabulary*
- *Part 7: Separative enclosures (clean air hoods, glove boxes, isolators and minienvironments)*

Users should note that the titles listed for parts 3, 4, 6 and 7 are working titles at the time of the release of part 5. In the event that one or more of these parts are deleted from the work programme, the remaining parts may be renumbered.

Annexes A to F to this International Standard are for information only.

## Introduction

Industries and organizations of all kinds utilize cleanrooms to help provide quality products and processes. Operational procedures have a profound effect on the cleanliness levels achieved during the operation of the cleanroom and equipment. Consistent quality is cleanliness dependent. Operational cleanliness can only be attained and maintained through a deliberate program established to specify, measure and enforce defined operational procedures. Regulatory agencies that have authority over processes and products produced in the cleanroom may require additional procedures and measures of cleanliness not covered in this general operating standard.

This part of ISO 14644 addresses normative and informative operational requirements related to:

- a) providing a system that defines policies and operational procedures;
- b) the clothing used to isolate the human element from the cleanroom environment;
- c) the training of personnel inside the cleanroom and monitoring their compliance to specified procedures and disciplines;
- d) the transfer, installation and maintenance of stationary equipment (selection criteria is not discussed);
- e) the selection and use of materials and portable equipment in the cleanroom;
- f) maintaining the cleanliness of the cleanroom through systematic cleaning and monitoring procedures.



# Cleanrooms and associated controlled environments — Part 5: Operations

## 1 Scope

This part of ISO 14644 specifies basic requirements for cleanroom operations. It is intended for those planning to use and operate a cleanroom. Aspects of safety that have no direct bearing on contamination control are not considered in this part of ISO 14644 and national and local safety regulations must be observed. This document considers all classes of cleanrooms used to produce all types of products. Therefore, this document is broad in application and does not address specific requirements for individual industries. Methods and programmes for routine monitoring within cleanrooms are not covered in detail in this part of ISO 14644 but reference should be made to ISO 14644-2 and ISO 14644-3 for monitoring particles and ISO 14698-1 and ISO 14698-2 for monitoring micro-organisms.

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 14644. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 14644 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 14644-1:1999, *Cleanrooms and associated controlled environments — Part 1: Classification of air cleanliness*.

ISO 14644-2:2000, *Cleanrooms and associated controlled environments — Part 2: Specification for testing and monitoring to prove continued compliance with ISO 14644-1*.

ISO 14644-3:—<sup>1)</sup>, *Cleanrooms and associated controlled environments — Part 3: Methods for evaluation and measurement*.

ISO 14644-4:—<sup>1)</sup>, *Cleanrooms and associated controlled environments — Part 4: Design, construction and start-up*.

ISO 14698-1:—<sup>1)</sup>, *Cleanrooms and associated controlled environments — Part 1: Biocontamination control—General principles*.

ISO 14698-2:—<sup>1)</sup>, *Cleanrooms and associated controlled environments — Part 2: Biocontamination control—Evaluation and interpretation of biocontamination data*.

## 3 Term(s) and definition(s)

For the purposes of this part of ISO 14644, the following terms and definitions apply.

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<sup>1)</sup> To be published.

### 3.1 General

#### 3.1.1

**airlock**

intermediate room or area that is usually ventilated, and used to minimise the transfer of airborne contamination from one area to another

#### 3.1.2

**biocleanroom**

cleanroom used for products and processes that are sensitive to microbiological contamination

#### 3.1.3

**changing room**

room where people entering or leaving a cleanroom put on or take off cleanroom clothing

[adapted from ISO 14644-4:—<sup>1</sup>), 3.1]

#### 3.1.4

**cross-over bench**

bench that is used as an aid to changing of cleanroom clothing and which provides a barrier to the tracking of floor contamination

#### 3.1.5

**disinfection**

removal, destruction or de-activation of micro-organisms on objects or surfaces

#### 3.1.6

**fibre**

particle having an aspect (length-to-width) ratio of 10 or more

[ISO 14644-1:1999, 2.2.7]

#### 3.1.7

**operator**

person working in the cleanroom performing production work or carrying out process procedures

#### 3.1.8

**particle**

minute piece of matter with defined physical boundaries

NOTE For classification purposes refer to ISO 14644-1:1999.

#### 3.1.9

**personnel**

persons entering the cleanroom for any purpose

#### 3.1.10

**unidirectional airflow**

controlled airflow through the entire cross section of a clean zone with a steady velocity and approximately parallel airstreams

NOTE This type of airflow results in a directed transport of particles from the clean zone

[ISO 14644-4:—<sup>1</sup>), 3.11]

## 3.2 Occupancy states

### 3.2.1

#### **as-built**

condition where the installation is complete with all services connected and functioning but with no production equipment, materials, or personnel present

[ISO 14644-4:—<sup>1</sup>), 2.4.1]

### 3.2.2

#### **at-rest**

condition where the installation is complete with equipment installed and operating in a manner agreed upon by the customer and supplier, but with no personnel present

[ISO 14644-4:—<sup>1</sup>), 2.4.2]

### 3.2.3

#### **operational**

condition where the installation is functioning in the specified manner, with the specified number of personnel present and working in the manner agreed upon

[ISO 14644-4:—<sup>1</sup>), 2.4.3]

## 4 Specification of requirements

### 4.1 Operational systems

**4.1.1** A system of operational procedures shall be established and documented, that will provide a framework for producing the quality products and processes for which the cleanroom was designed.

**4.1.2** A set of risk factors, appropriate for the use of the cleanroom, shall identify the areas where there is a risk of contamination to the process. A method for monitoring these risks shall be instituted so action can be taken when conditions violate the contamination limits for the cleanroom classification.

NOTE Although not covered in detail in this part of ISO 14644, it is important to routinely monitor the operation of a cleanroom. Guidance for monitoring particles is given in ISO 14644-2 and ISO 14644-3. Guidance for monitoring biocontamination is given in ISO 14698-1 and ISO 14698-2.

**4.1.3** A system for training personnel in cleanroom procedures shall be instituted. A method for monitoring compliance to those training procedures shall be specified.

**4.1.4** A documentation system shall be maintained to provide evidence that all personnel have received suitable levels of training for their assignments.

**4.1.5** A set of procedures shall be documented to describe how the cleanroom mechanical systems are to be operated, maintained, repaired and monitored (see ISO 14644-4).

**4.1.6** All activities that modify, supplement, or enlarge the cleanroom shall be planned and include all relevant personnel. Any significant change of operational use may be subject to re-qualification of the installation in compliance with ISO 14644-2.

**4.1.7** A system shall be documented that encourages and enforces safety for personnel in the cleanroom that may influence aspects of contamination control.

NOTE Informative guidance concerning the operational systems requirements listed in 4.1.1 to 4.1.7 can be found in annex A.

## 4.2 Cleanroom clothing

**4.2.1** Cleanroom clothing shall protect the environment and products from contamination generated by the personnel and their everyday clothing. To maximise this containment, the choice of barrier fabric, the clothing style and extent of coverage of personnel by the cleanroom clothing shall be established.

**4.2.2** Cleanroom clothing shall be made of fabrics and materials that will resist breakdown (minimal linting) and therefore not shed contamination.

**4.2.3** The frequency of changing into fresh clothing before entering the cleanroom shall be determined in accordance with the product and process cleanliness requirements.

**4.2.4** Reusable cleanroom clothing shall be processed at regular intervals to remove contamination.

**4.2.5** The necessary cleaning, processing (including sterilization or disinfection where required) and packaging of clothing shall be defined.

**4.2.6** Cleanroom clothing shall be transported and stored in a specified manner to minimize contamination.

**4.2.7** Cleanroom clothing (clean packaged or dirty) shall not be removed beyond the confines of the storage area and cleanroom except for laundering purposes.

**4.2.8** Cleanroom clothing shall be put on and taken off in such a way that the spread of contamination is avoided or minimized.

**4.2.9** If clothing is to be reused, it shall be removed and stored to ensure that contamination is minimized.

**4.2.10** Cleanroom clothing shall be checked at regular intervals to ensure that it retains acceptable contamination control characteristics.

**4.2.11** Consideration shall be given for the comfort of personnel wearing the cleanroom clothing.

**4.2.12** Consideration shall be given to special (e.g. chemical, physical or microbiological) properties of the clothing that may be necessary for specific applications.

**4.2.13** Consideration shall be given to special concerns for cleanroom clothing during and after emergency evacuations.

NOTE Informative guidance concerning cleanroom clothing requirements listed in 4.2.1 to 4.2.13 can be found in annex B.

## 4.3 Personnel

**4.3.1** Personal and other items not intended for cleanroom use shall not be allowed inside the cleanroom, unless approved.

**4.3.2** Personnel shall be instructed in hygiene-related issues that will prepare them for properly working in the cleanroom environment.

**4.3.3** A policy concerning jewelry, cosmetics and similar materials that can cause contamination problems shall be determined.

**4.3.4** Cleanroom personnel shall be trained to conduct themselves in a manner that minimizes the possibility of contamination being generated or stirred up, and transferred or deposited on or into the product.

**4.3.5** Personnel shall be protected against hazards. They shall receive safety training for all known health and safety risks associated with their work.

NOTE Informative guidance concerning personnel requirements listed in 4.3.1 to 4.3.5 can be found in annex C.

## 4.4 Stationary equipment

**4.4.1** All equipment, with its associated moving and rigging equipment, shall be thoroughly cleaned or decontaminated, or both, before being transported into the cleanroom environment.

**4.4.2** Procedures relating to the entry of equipment into a controlled environment shall be specified to ensure that all equipment undergoes the necessary cleaning and decontaminating.

**4.4.3** Installation of equipment shall be planned and carried out to minimize the impact on the cleanroom environment.

**4.4.4** Equipment maintenance, repairs and calibration procedures shall be performed in such a way as to control and minimize contamination of the cleanroom.

**4.4.5** Procedures relating to maintenance work and repairs shall be specified to control contamination.

**4.4.6** Preventive maintenance schedules shall be established and timed to renew and replace components before the components become contamination sources.

NOTE Informative guidance concerning stationary equipment requirements listed in 4.4.1 to 4.4.6 can be found in annex D.

## 4.5 Materials and portable and mobile equipment

**4.5.1** All materials, and portable and mobile equipment shall be appropriate for the level of cleanroom cleanliness, and in use, shall not compromise the product and process.

**4.5.2** Procedures shall be established to ensure materials and portable and mobile equipment entering the cleanroom are not contaminated.

**4.5.3** Procedures shall be established to minimise the quantities of materials stored in the cleanroom. Consideration shall be given to shelflife limitations, if applicable.

**4.5.4** Materials stored in the cleanroom shall be subject to defined procedures and, where necessary, shall be held in protective storage or isolation. The risk of contamination, arising from the storage and subsequent use of materials and portable and mobile equipment in the cleanroom, shall be considered.

**4.5.5** All used and waste materials shall be collected, identified and removed in accordance with defined procedures. Waste materials shall be removed frequently and in a manner that does not compromise the cleanliness of the product or process. Procedures for hazardous materials shall conform to standards set by local and other regulatory agencies.

NOTE Informative guidance concerning materials and portable equipment requirements listed in 4.5.1 to 4.5.5 can be found in annex E.

## 4.6 Cleanroom Cleaning

**4.6.1** Cleaning methods and procedures shall be specified and routinely followed to maintain cleanroom surfaces at acceptable cleanliness levels.

**4.6.2** Personnel responsible for the cleaning operation shall be designated and receive specific training for accomplishing the task.

**4.6.3** Cleaning schedules shall be defined and carried out at effective frequencies to ensure that specified cleanliness levels are maintained.

**4.6.4** Appropriate contamination checks shall be carried out on a routine basis to ensure the cleanroom is maintained at specified levels.

**4.6.5** An assessment shall be made to identify any cleaning procedures that will place products or processes at risk during the performance of such cleaning tasks so preparations are made to remove or cover work-in-process before cleaning begins.

**4.6.6** Special cleaning procedures and techniques shall be defined for unavoidable accidents or system failures that create contamination that places the cleanroom, products, processes or personnel at risk.

NOTE Informative guidance concerning cleaning requirements listed in 4.6.1 to 4.6.6 can be found in annex F.

## Annex A (informative)

### Operational systems

#### A.1 General

It is essential that management provides leadership that will focus the attention of its staff on generating and maintaining systems that will encourage good cleanroom practices. A management structure should be defined and published to ensure all parties are aware of their responsibilities. Good cleanroom practices will have a significant impact on the quality of products being produced and the processes performed in the cleanroom. This annex is provided to assist management in identifying those systems.

#### A.2 Assessing contamination risks

##### A.2.1 Methods for assessing risks

A risk assessment should be made to determine any relevant contamination control factors that may affect the products or processes performed in the cleanroom.

Some examples of methods used for determining and managing these factors include:

- a) HAZOP (HAZard and OPerability studies of the International Social Security Association)<sup>[1]</sup>;
- b) HACCP (Hazard Analysis Critical Control Point)<sup>[2]</sup>;
- c) FMEA (Failure Mode Effects Analysis)<sup>[3][4]</sup>;
- d) FTA (Fault Tree Analysis)<sup>[5]</sup>.

##### A.2.2 Determining operational risks

Improper control of the critical elements of an operational cleanroom can pose a risk to the cleanliness of the cleanroom and the quality of the product. An assessment of these risks should be carried out and plans formulated to remedy out-of-control situations. The list in A.2.2.1 identifies some of the risks that may prove important. Each organisation should determine which of these factors to include in its own risk assessment program. Information on cleanroom support parameters including heating, ventilation and air conditioning functions, pressure, temperature, humidity, air change failure and filter failure are discussed in ISO 14644-2, ISO 14644-3 and ISO 14644-4.

###### A.2.2.1 Cleanroom clothing

Risk factors that may influence the operation or environmental quality of the cleanroom may include:

- a) required human containment (coveralls, frocks, hoods, gloves, boots, masks, etc);
- b) material performance (weave characteristics, filament types, sterility, antistatic, calendaring, etc);
- c) design and construction (special tailoring requirements);
- d) comfort;

- e) usage (launderable vs disposable);
- f) choice of personal clothing worn under cleanroom clothing;
- g) time interval or number of wearings before laundering is required;
- h) choice of cleanroom clothing laundry;
- i) renewing, packaging, storage, and distribution.

#### **A.2.2.2 Personnel**

Risk factors that may influence the operation or environmental quality of the cleanroom may include:

- a) selection of personnel;
- b) education and training;
- c) safety (including emergency procedure);
- d) personnel practices, hygiene, and behavior, (including behavior prior to entering the cleanroom);
- e) chronic and acute medical conditions;
- f) who is allowed to enter;
- g) special procedures for visitors;
- h) maximum occupancy;
- i) entry and exit procedures;
- j) the movement of personnel within the cleanroom.

#### **A.2.2.3 Stationary equipment**

Risk factors that may influence the operation or environmental quality of the cleanroom may include:

- a) entry and exit procedures;
- b) installation;
- c) cleaning techniques;
- d) contamination generation;
- e) generation of heat, humidity and electrostatic charge;
- f) maintenance and repair;
- g) cleanliness of process material and utilities delivery systems;
- h) potential equipment failures.

#### **A.2.2.4 Materials and portable and mobile equipment**

Risk factors that may influence the operation or environmental quality of the cleanroom may include:

- a) compatibility and selection;
- b) entry, exit and movement procedures;
- c) storage factors while in the cleanroom;
- d) contamination factors during use;
- e) generation of electrostatic charges;
- f) fluid and gas purity supplied by delivery systems;
- g) waste disposal;
- h) packaging.

#### **A.2.2.5 Cleanroom cleaning**

Risk factors that influence the operation or environmental quality of the cleanroom may include:

- a) routine environmental contaminating factors (airflows, airborne particles, outgassing, hazardous gas, viables, vibration, electrostatic charges, molecular contamination, etc.);
- b) personnel and material flow;
- c) service, maintenance, and repair;
- d) cleaning methodology;
- e) emergency and planned shutdown;
- f) facility expansion and modification;
- g) frequency for monitoring the results.

### **A.3 Monitoring and corrective action**

A routine monitoring programme encompassing personnel, cleaning and other operational systems should be followed. Monitoring should be sufficiently frequent and comprehensive to detect actual or emerging unacceptable conditions in a timely manner. Exceeding specified action levels should result in a prompt response, including investigative and corrective action. Investigative and corrective action should include the effect on product quality as a potential result of the out-of-specification condition. Further information can be found in ISO 14644-2 and ISO

14644-3 for particle monitoring. Information on microbiological monitoring can be found in ISO 14698-1 and ISO 14698-2.

### **A.4 Education and training**

#### **A.4.1 Involvement**

General personnel activity within the cleanroom has a profound effect on the integrity of the clean environment. Failure to properly train anyone entering, using or maintaining the facility will compromise the effectiveness of the cleanroom. Management is therefore responsible for implementing a comprehensive program to train all personnel with regard to their responsibilities and how those responsibilities interact with the clean environment. Certification should be based on successful completion of testing to demonstrate understanding and compliance. The program should ensure each of the following groups of personnel is educated and trained appropriately:

- a) operators;
- b) technicians;
- c) engineers and scientists;
- d) supervisors and managers;
- e) facilities personnel;
- f) contractors;
- g) field service personnel;
- h) visitors.

#### **A.4.2 Training course contents**

Subjects that can be included in the training course include:

- a) how the cleanroom works (design, airflow, and air filtration);
- b) cleanroom standards;
- c) sources of contamination;
- d) personal hygiene;
- e) cleaning;
- f) cleanroom clothing procedures;
- g) maintenance procedures;
- h) how a cleanroom is tested and monitored;
- i) how to act in a cleanroom;
- j) explanation of the work process and technologies or sciences employed;
- k) safety and emergency response.

#### **A.4.3 Monitoring of cleanroom personnel and corrective action**

The cleanroom training program provides an explanation of requirements and actions that minimise the risk factors important to the cleanroom, identified in A.2.2.2. The ability of personnel to incorporate all elements of cleanroom training into practice is essential to the continuous, effective operation of the cleanroom. Although properly trained, personnel may not fully understand or fall into poor cleanroom habits. Therefore, actions of personnel, listed in A.4.1, should be monitored to ensure personnel carefully comply with correct cleanroom disciplines. Consideration should be given to a system that will monitor the cleanroom personnel. Monitoring programs can be formal or informal depending on the level of empowerment given to each person that is part of the cleanroom staff. Internal auditors can monitor the actions of those in the cleanroom based upon the written procedures. Reports can be issued to management on a regular basis detailing deficiencies and can be used for determining corrective action<sup>[6]</sup>.

An effective program should be a positive influence on all personnel to follow proper cleanroom procedures.

#### **A.4.4 Training documentation**

A concise, comprehensive system that documents the training progression and level of each individual should be used. The management team should identify each job and set of jobs or responsibilities. This documentation system should be easily accessible to management and periodically reviewed. Basic documentation should include course contents, personnel identification information, training and certification dates, and schedules for retraining that may be required at future intervals.

### **A.5 Cleanroom support services**

Management is responsible for ensuring cleanroom support services consistently function as designed on a day-to-day basis. Support services may include clean and conditioned air systems, compressed air and gasses, water and other utilities, and other aspects required for standard cleanroom operation. Failure of any mechanical support system can seriously affect the cleanliness and operation of the cleanroom. Records and procedures documenting the operation of the systems that provide and maintain the cleanroom should be readily available. Some of the information required to establish such systems is given in A.5.1 to A.5.5. More thorough coverage of the subjects listed in A.5.1 to A.5.5 is found in ISO 14644-4.

#### **A.5.1 Record of the installation**

This record should contain installation drawings, cleanroom classification including acceptance test results to original specifications, and recommended spare parts lists.

#### **A.5.2 Operating and maintenance instructions**

The mechanical and electrical systems within the installation should have a clear set of operating and maintenance instructions. These instructions should describe procedures used to check and inspect all critical components prior to start-up. Emergency shutdown procedures and start-up procedures after unplanned shutdowns should be documented.

#### **A.5.3 Performance monitoring**

Performance monitoring of the installation is essential to demonstrate satisfactory operation. Documented schedules and procedures that specify the required tests and the frequency of testing are needed to demonstrate compliance with specified cleanroom classifications. Action plans for out-of-control situations should be defined.

#### **A.5.4 Maintenance procedures**

Unplanned downtime can adversely affect productivity and introduce contamination to the cleanroom. Ongoing performance checks and preventive maintenance should be performed to minimize contamination that may be caused by unanticipated equipment failures. Repair and maintenance procedures should contain precautions that will help minimise and contain contamination. Tests may also be necessary to ensure that reactivated equipment is clean and in specification before being accepted for reuse.

#### **A.5.5 Maintenance records**

Evidence of effective maintenance requires a documented record involving all maintenance activities. Problem diagnoses, parts replaced, dates, times and personnel performing the maintenance should be documented. Preventive maintenance schedules and charts should be updated as required. Periodic analysis of such records may help in making improvements to the program and help optimize preventive maintenance schedules.

### **A.6 Upgrading and modifying the cleanroom**

All upgrades or modifications, including the addition of stationary equipment and changes to floor plans, can affect the cleanroom. Management should make certain these changes are planned and carried out in a controlled and

thorough manner and that requalification of the installation is in compliance with ISO 14644-2 and ISO 14644-4. A record of all changes or modifications should be documented after requalification. All appropriate personnel with responsibilities affected by these changes should be involved and kept informed of progress. Such personnel can include but are not limited to:

- a) facility engineers;
- b) manufacturing engineers;
- c) equipment engineers;
- d) contamination control engineers;
- e) process engineers and scientists;
- f) quality assurance engineers and scientists;
- g) manufacturing managers;
- h) contractors.

## **A.7 Safety**

Normal operation of cleanroom facilities often includes the use of hazardous, toxic or infectious materials. Personnel should be protected from exposure to these agents. Management should implement and monitor effective systems for protecting the health and welfare of personnel. Good programs should include the following:

- a) centralised, readily-available safety data sheets that describe hazardous materials;
- b) evacuation plans and practice evacuations;
- c) accident reporting system;
- d) feedback suggestion systems for personnel;
- e) appropriate monitoring of potentially hazardous conditions or materials;
- f) rapid response to emergencies by trained personnel;
- g) documentation that supports improvements and corrections to safety related issues.

## **Annex B** (informative)

### **Cleanroom clothing**

#### **B.1 Function of cleanroom clothing**

Personnel disperse fragments from their skin and particles from their normal indoor clothing. This airborne dispersion will vary from person to person and from time to time but can be several million particles per minute and several hundred bacteria-carrying particles per minute. The prime function of cleanroom clothing is to act as a barrier filter that protects product and process from human contamination. Therefore, cleanroom clothing should be made from a fabric that filters the contamination dispersed. Cleanroom clothing should also be designed to envelop a person and not allow significant amounts of unfiltered body emissions to be dispersed into the cleanroom. An effective cleanroom undergarment in combination with cleanroom clothing can give an additional reduction in dispersion.

Although the majority of contamination originates from the skin and non-cleanroom clothing, contamination is also dispersed from the surface of cleanroom clothing fabrics. The fabric used to manufacture cleanroom clothing should not add to the contamination burden.

Personnel emit inert and microbe-carrying particles from the mouth through sneezing, coughing and talking. Touching will transmit contamination from the hands to surfaces in the cleanroom. Depending on the cleanroom function and class, it may be necessary to wear facemasks, helmets and gloves to minimize transmission of these types of contamination.

The choice of cleanroom clothing will vary according to the product cleanliness and process requirements but will normally, but not exclusively, consist of hoods, caps, helmets, coveralls, overboots, facemasks and goggles or safety glasses.

#### **B.2 General choice of cleanroom clothing**

The best design of cleanroom clothing will completely envelop the person and have good closures at the wrist, neck and ankle. The choice will depend on the class of cleanroom but cleanrooms with higher cleanliness requirements are typified by a one-piece coverall, overboots and a hood with yoke or skirt that tucks under the neck of the garment. Increasing technical requirements on cleanroom clothing may result in increasing personal restrictions or discomfort. Therefore, consideration should be given to what is necessary for the standard of room cleanliness. Where cleanliness and process requirements permit, clothing of lesser coverage may be acceptable<sup>[7]</sup> <sup>[8]</sup><sup>[9]</sup><sup>[10]</sup>. Certain enhanced devices with built-in clean air systems (e.g. minienvironments or isolators) may allow for the simplification of required cleanroom clothing.

There are two broad categories of clothing used in cleanrooms: 1) disposable (or limited use) and 2) reusable. In general, disposable or limited use clothing usually is made from a non-woven materials and is used either once or a few times, and then discarded. Reusable clothing is processed at regular intervals and usually made from tightly woven synthetic fabrics. More critical applications may require the use of membrane barrier technology. Natural fabrics made from fibres, such as cotton, would not normally be used in cleanrooms as they easily break up and disperse contamination.

## B.3 Properties of fabric

### B.3.1 Barrier properties

The fabric used in cleanroom clothing should prevent personnel-generated contamination from being dispersed into the cleanroom. Woven fabric acts as a filter; effectiveness is related to the tightness of the weave of the fabric. The effectiveness of the weave can be tested by measuring the pore size and the efficiency of the fabric in removing particles. In the case of barrier-type fabrics such as non-wovens and laminate membranes the effectiveness to contain contamination is a function of the barrier characteristics. Fabric effectiveness can be assessed by a measurement of air permeability, particle retention and pore size<sup>[8][11][12]</sup>. As air permeability decreases, there is a corresponding increase in pressure within the garment as personnel move about. This can result in an outward pumping action of unfiltered air through the closures of the cleanroom clothing.

### B.3.2 Durability

Cleanroom clothing should be resistant to breakdown and tearing. The fabric should disperse the minimum of particles. Information is available on tests used to assess these types of fabric properties<sup>[7][10][13][14]</sup>.

### B.3.3 Electrostatic properties

In some types of cleanrooms (e.g. microelectronics or rooms with flammable or explosive chemicals) the electrostatic charges that build up on the surface of clothing will be harmful to the components being manufactured or hazardous to operators. Fabrics are available with woven-in, static-dissipative threads to discharge any induced voltage potentials on the fabric surface. The effectiveness of a fabric to dissipate an electrostatic charge can be indirectly measured by checking the fabric's surface resistivity. Such methods are described in other sources<sup>[15][16]</sup>.

In a more effective test, a static charge of known voltage level is applied to the fabric. Static dissipative performance can then be determined by the time it takes for the voltage to decrease by a given percentage of the original voltage. Such methods are described in other sources<sup>[15][16]</sup>.

### B.3.4 Other physical properties

The effectiveness of a fabric will deteriorate due to aging, wear, washing, drying, sterilization, etc. This deterioration should be monitored. Another physical property that should be considered is the resistance of the fabric to chemicals, such as those used during the manufacturing process and in the cleaning and disinfection of the cleanroom and clothing.

NOTE The tests referenced in B.3.1 to B.3.4 will help to verify that the clothing remains effective.

## B.4 Design and construction of cleanroom clothing

### B.4.1 Construction of clothing

Cleanroom clothing should be constructed to minimise contamination in the cleanroom. Cutting the fabric prior to sewing produces raw edges that will generate particles if left unfinished. Methods used to finish these edges are as follows: all raw edges of the fabric should be covered, interlocked and heat sealed or laser cut to prevent fraying. Seams should be double-needle stitched, bound, or taped to provide a good barrier and not produce fibres. Threads should be synthetic continuous filament. Zippers, clips and fasteners, and shoe soles should not shed, chip or corrode, and should stand up to multiple launderings and where necessary, sterilisation.

### B.4.2 General design

The selection of the design of clothing should be considered with respect to the type of cleanroom<sup>[8][9][10]</sup>.

Cleanroom clothing should incorporate a large selection of sizes to provide comfort and fit. To minimise the retention of contamination pockets, pleats, darts, hook and pile fasteners, and action backs should not be used. Elasticised or knitted cuffs should not trap or shed contaminants and should not build up electrostatic charges. Garment closures should provide a tight yet comfortable closure. Other design parameters that should be considered are:

- a) zipper material (e.g. covered plastic zip-fasteners), type, and location;
- b) placement and effectiveness of snap adjusters and stays;
- c) sleeve construction (set-in or raglan);
- d) collar style;
- e) ability to don over various shoe or boot styles;
- f) hood style (open or closed face, snap or pull-over);
- g) passive or active adjustment and fit of hoods;
- h) type and placement of straps on boots.

#### **B.4.3 Dispersal chamber (body box)**

This simulation procedure can be used to demonstrate the combined effect of fabric, construction and design of clothing. A person will enter the box, which is ventilated at a known flow rate of filtered air, and exercise to a given routine. The number of particles or bacteria dispersed can be measured. Different types of clothing that have to be assessed can be compared. A description of this test is available<sup>[8]</sup>.

### **B.5 Thermal comfort**

Whenever possible, the comfort of people working in the cleanroom should be considered when choosing cleanroom clothing materials<sup>[21]</sup>. Air and moisture permeability specifications of the fabrics under consideration can help in this determination<sup>[18][19]</sup>. A simple but effective approach is to obtain a selection of suitable clothing of different fabrics and try them in the cleanroom. Feedback, solicited from personnel who will be expected to wear the clothing, may provide valuable information that will aid in the selection process.

The use of relevant personnel parameters and environmental parameters can be used to derive theoretical clothing comfort levels to determine cleanroom clothing requirements for the cleanroom. Personal clothing or underclothing worn by personnel (clo=clothing factor), as well as physical activities (met=metabolic rates) in the cleanroom are determined. Additionally, air temperature, velocity, turbulence, mean radiant temperature and humidity within the cleanroom are used to calculate the Predicted Mean Vote (PMV) index leading to the Predicted Percentage of Dissatisfied (PPD). The resultant PPD index indicates the percentage of people that are likely to be too hot (or cold) in given conditions. ISO 7730<sup>[20]</sup> provides guidance and tables that will assist in making this determination.

### **B.6 Processing of clothing and frequency of change**

During use, cleanroom clothing will become contaminated. If it is to be reused it should be cleaned. Suggestions as to how this cleaning process should be carried out are available in other sources<sup>[8][17]</sup>. Final treatment and packaging operations for cleanroom clothing should be carried out in cleanroom conditions that match the standards of the cleanroom in which they will be used. Clothing similarly becomes contaminated with bacteria. In cleanrooms where bacteria are an important consideration, the processing cycle in the cleanroom laundry should include, as appropriate:

- a) disinfection;

- b) hot water cycles;
- c) sterilisation.

Cleaning procedures should be followed by sample testing at the laundry for the appropriate type and level of contamination.

The frequency of clothing change will vary according to the intended use of the cleanroom. The more sensitive the process is to contamination, the more frequently the clothing should be changed and cleaned. Guidance is also available to help in the decision making process<sup>[8]</sup>.

## B.7 Gloves

Cleanroom gloves are required in most cleanrooms. They cover that part of the human body that is often closest to the product and critical surfaces. Consideration should therefore be given to whether gloves are necessary. If they are used, consideration should be given to what properties are best suited, as well as how often they should be changed or cleaned (and where appropriate, disinfected).

Properties of cleanroom gloves that should be considered with respect to the type of cleanroom in which they will be used are as follows: surface contamination, outgassing, sterility, tactility, strength, comfort, fit as well as the method of packaging. Various tests can be performed to help in selecting the proper gloves for each particular cleanroom application<sup>[22]</sup>.

Gloves can be constructed of latex, vinyl, or other materials such as nitrile rubber. The choice of construction should be considered with respect to the required properties and application of the glove, and the cost. Undergloves, made from non-linting materials, may also be needed by some employees to provide a level of comfort or isolation from inner glove surfaces that can cause, or aggravate, contact dermatitis.

## B.8 Facemasks and other headgear

Face masks and exhaust headgear provide a barrier against saliva and contamination dispersed from the mouth, nose, face and, in the case of headgear, the head. Masks and veils are passive barrier elements commonly used in cleanrooms. The masks can be surgical-style masks with elasticised straps and loops and the veils are snapped into hoods or permanently sewn into the hood at manufacture. Materials used are washable and disposable fabrics. Care should be taken to select the proper material and style that is appropriate to the risk from emissions from the mouth. This selection should also consider the acceptability of the facemask to the personnel.

Headgear is available that provides an active barrier to contamination from the mouth and head. A helmet with hood and clear face-shield encloses the head and is provided with a filtered exhaust system that prevents contamination from escaping into the cleanroom.

Glasses or goggles can help provide an additional barrier to help retain skin flakes and eyelashes and keep them from falling onto critical surfaces. Glasses or goggles should be constructed from materials that are cleanroom compatible and should conform to accepted personal safety standards.

## B.9 Storage of clothing

**B.9.1** If the cleanroom clothing is to be reused, it should be stored or hung using appropriate techniques that will maintain the cleanliness of the clothing. Clothing elements may require physical separation when stored. Launderable or disposable bags can be used to help avoid cross-contamination. Several methods are effective for storing clothing. These may include:

- a) clothing racks with high efficiency self-contained, filtered air supply;
- b) fixed and portable racks utilising hangers;

- c) locking and non-locking hooks mounted to walls or frames in the changing area or room. These can be in a locker or in the room;
- d) bins or storage slots.

**B.9.2** The space required to store cleanroom clothing to supply all personnel working in the cleanroom is dependent upon the number of people working in the cleanroom and the frequency at which the cleanroom clothing is changed.

**B.9.3** An area large enough to contain the packaged cleanroom clothing should be set aside for storage purposes. Lockers can be obtained for this purpose. These lockers should be placed on the cleaning schedule to ensure that they do not contribute to contamination.

**B.9.4** Cleaned clothing should be packaged in clean, non-shedding bags to avoid contamination during handling, storage and distribution. Shelflife for sterilised products should be defined. It is recommended that storage should be in a controlled environment that is adjacent to or in the changing area. This allows better control of the inventory and reduces the risk of clothing being removed from the cleanroom environment and becoming soiled.

## **Annex C** **(informative)**

### **Personnel**

#### **C.1 Training**

Only trained personnel should be allowed to enter and work in a cleanroom. All personnel should be given an introductory course when initiated into the cleanroom and further periodic retraining (see A.2).

#### **C.2 Access by personnel**

People generate contamination; the more people in a cleanroom the greater the potential for elevated contamination levels. Therefore, only essential personnel should enter the cleanroom. If the total number of people allowed in the cleanroom is controlled, access should be documented and enforced. Visitors and maintenance people can be allowed into the room by permission and under supervision. They should be given an appropriate level of training.

#### **C.3 Clothing and personal items**

The type of clothing worn under cleanroom clothing will affect the dispersion of airborne particles and fibres. Personal indoor clothing manufactured from natural fibres such as wool or cotton and worn underneath cleanroom clothing will shed contamination. The provision for special cleanroom undergarments should be considered. If these are provided, they should be made from closely woven, artificial fibres such as polyester for effective filtration of body contamination.

Personal items should be left outside the cleanroom in a secure area. Jewelry, such as rings, watches, and chains, can puncture cleanroom gloves or dangle outside facemasks, hoods, or sleeves of the clothing and should be avoided.

Cosmetics, talcum powder, hair sprays, nail polish or similar materials are undesirable in a cleanroom. An assessment should be made of the risk to the product or process from these items. Cosmetics can generate particles that contaminate cleanroom clothing, the cleanroom and products being produced and may be prohibited.

#### **C.4 Hygiene**

Cleanroom personnel are expected to have good personal hygiene. Personnel should keep dandruff controlled, and, as necessary, use specially formulated skin lotion to replace skin oils after washing and showering.

Personnel arriving for work should report problems that might increase contamination in the cleanroom, including the following:

- a) conditions such as flaking skin, dermatitis, sunburn or bad dandruff;
- b) a cold, flu or chronic coughing;
- c) allergic conditions which cause sneezing, itching or scratching;
- d) in a biocleanroom—high microbial bioburden on personnel.

Depending on the seriousness of the condition with respect to the process or product being produced, it may be necessary to reassign personnel with such conditions to work outside the cleanroom until the condition is in abeyance.

In some cleanrooms, it may be required that personnel refrain from smoking for a defined period of time before entering.

## C.5 Cleanroom clothing changing procedures

Cleanroom personnel will change into cleanroom clothing before proceeding into a cleanroom. A method should be adopted to put on and remove clothing to minimise contamination of the outside of the cleanroom clothing and to ensure contamination is not spread from the changing area. Several methods are acceptable depending on the design of the changing area and the standard of cleanliness of the cleanroom. Further information is described in other sources<sup>[6][23]</sup>. A typical procedure is outlined in the following list, but many variations exist. Usually the process begins at the head and proceeds downward to the feet:

- 1) Remove contamination from shoes by use of a shoe cleaner, cleanroom mat or cleanroom flooring.
- 2) Remove unnecessary street clothing.
- 3) Remove jewelry, etc. if required.
- 4) Remove cosmetics and put on moisturiser, if required.
- 5) Put on hair cover, if applicable.
- 6) Wash hands and put on suitable moisturiser, if applicable.
- 7) Put on cleanroom underclothing, if applicable.
- 8) Put on cleanroom-dedicated under-shoes, or shoe covers.
- 9) Select cleanroom clothing.
- 10) If required, put on gloves for handling cleanroom clothing.
- 11) Put on face and head covering.
- 12) Put on coverall or gown.
- 13) Put on shoe coverings or special cleanroom shoes, using a crossover bench.
- 14) Gloves used for putting on cleanroom clothing can now either be removed, or left on, so that process gloves can be put on.
- 15) Using a full-length mirror, ensure that all items of clothing are properly adjusted.
- 16) Enter the cleanroom.

The way cleanroom clothing should be taken off when leaving the cleanroom will depend on whether fresh clothing is used on each entry or whether the clothing is to be reused. Methods for removing cleanroom clothing that will be reused is described in other sources<sup>[23]</sup>. Special storage methods can be used if the clothing is to be reused and are described in other sources<sup>[5][21]</sup>. Cleanroom clothing should not be removed from the controlled environment except for transfer to the laundry for cleaning.

## C.6 Discipline and conduct

Cleanroom personnel should conduct themselves in a cleanroom in such a way as to minimize the possibility of contaminating the product. The following are minimum disciplines that should be considered (more information can be found in other sources<sup>[6][23][24][25][26]</sup>).

- Doors should not be opened and closed quickly, nor left open.
- When entering an airlock, the first door should be allowed to close before the next one is opened.
- Personnel should not position themselves between clean air supplies and product or process surfaces. Doing so will increase the risk of dispersing particles onto product or process surfaces. In general, the correct positioning sequence should be: air supply to exposed product to personnel and then to the general cleanroom area and the air return or exhaust.
- Methods should be devised for moving or manipulating the product. 'No-touch' techniques should be used where appropriate.
- Personnel should not support material against their body, or contamination may be transferred.
- Personnel should not talk when working close to the product.
- Personnel should not allow anything to trail over the product.
- Nose blowing should be done outside the cleanroom. Gloves should always be changed afterwards.
- Personnel should also refrain from touching, scratching or wiping any skin areas while in the cleanroom.
- Glove and garment surfaces can easily become contaminated. Personnel should not touch surfaces and transfer contamination to critical areas.
- A cleanroom wipe should be used as specified and then discarded.
- All personnel movements should be deliberate and methodical. Over-exuberant behavior should not be allowed. Contamination generation is proportional to personnel activity.
- The room should be kept neat and tidy.
- Products stored or left standing in a cleanroom should be protected from contamination and kept in an identifiable closed cabinet, container or unidirectional cabinet.
- Waste material should be placed into easily identifiable containers and not allowed to collect unnecessarily.

## C.7 Safety

**C.7.1** Personnel should be protected against hazards that may occur or may be in use in the cleanroom, such as microbes, radioactivity and chemicals. Containment cabinets, cupboards or isolators may address these concerns and information on such methods is discussed in ISO 14644-4 and ISO 14644-7. Suitable protective clothing such as eye splash shields, gloves and aprons may be required. Other authorities may recommend or require additional measures to protect the safety of personnel in cleanrooms.

**C.7.2** Emergency situations may arise and emergency response personnel, trained in all aspects of potential emergencies can minimise the effects of mishaps that may occur. All employees should be trained for an orderly evacuation. If an evacuation is necessary, provisions should be made for the orderly return to the cleanroom once the situation is cleared. An emergency procedure for supplying fresh cleanroom clothing should be implemented.

## C.8 Personnel initiatives

Elements for formal monitoring and corrective action programs are described in A.7. However, personnel should understand that they could have a positive influence on the effectiveness of the cleanroom. Helpful coaching of one to another can have a positive effect on conformance to personnel procedures. Personnel should be encouraged and empowered to immediately report any observed deficiencies, whether personnel or facility related, to individuals responsible for cleanroom integrity. Such action will allow otherwise unnoticed contamination sources to be corrected before the problem becomes serious enough to place products or process at risk.

## Annex D (informative)

### Stationary equipment

#### D.1 General

Equipment that is large enough to be stationary or relatively immovable, once located within the cleanroom, is discussed in this section. Usually, extensive efforts are required to remove or relocate this equipment once installation is completed.

#### D.2 Clean Entry Process

When possible, equipment to be used in the cleanroom should be manufactured under clean conditions and packaging procedures should be adapted for the requirements of the intended cleanroom.

The process of bringing equipment into the cleanroom should not add contamination to the environment.

Equipment entering a cleanroom that is "as-built" or "at-rest" should be properly unpacked and cleaned. Failure to do so will require extensive cleanup afterwards. However, special considerations should be made before bringing equipment into an "operational" cleanroom. Failure to do so will expose not only the cleanroom to contamination risks but may affect products in process. This will also necessitate additional cleaning and may require the cleanroom to be requalified under ISO 14644-2. An appropriate strategy should be developed to avoid problems. Guidance is given in D.2.1 and in other sources<sup>[6]</sup>.

##### D.2.1 Unpacking

**D.2.1.1** All equipment should be checked for damage in transport. Suspected or damaged goods should be isolated or protected outside the cleanroom pending appropriate actions. Whenever possible, shipping crates and packaging should be removed in the uncontrolled environment adjacent to the cleanroom. All cardboard and heavily shedding materials should be removed before being transported into the controlled environment. When not pre-packaged, all surfaces of the equipment should be pre-cleaned prior to entry of the equipment into a cleanroom area. This cleaning is best carried out within the airlock used for equipment entry.

If the equipment is so large that special installation procedures are required, the area should be isolated from surrounding cleanrooms or other controlled environments through the use of temporary walls. Unpacking of equipment should be done in steps to control contamination entering the cleanroom. The airlock, or a temporary room built for this purpose and attached to the cleanroom, can be used for the removal of exterior film packaging materials and surface cleaning before cleanroom entry.

**D.2.1.2** The following is an example of the steps that should be taken during unpacking:

- 1) The outer protective covering should be vacuumed, beginning at the top surface and then proceeding to the sides.
- 2) The protective cover should be wiped, using the appropriate cleaning agent.
- 3) The outer layer of packaging film should be slit at the top in an "I" form and peeled from the top to the bottom edge. The bottom edge of the packaging film should then be lifted and joined to the sides of the packaging film.

- 4) The unpacking procedures in steps 2 and 3 should be repeated for each additional layer. All exterior surfaces of the equipment should be thoroughly cleaned.
- 5) All personnel should be wearing the proper cleanroom clothing prior to entering the airlock.
- 6) All moving and handling equipment should be cleaned, within the airlock, in accordance with procedures described in D.2.1.1.
- 7) The airlock should be cleaned before opening the doors to the cleanroom for transferring the equipment inside.

### D.3 Transporting equipment

Large equipment should be dismantled (if possible) to a size that will enable safe entry, minimizing risk to personnel and the existing cleanroom. Physical damage and contamination can result when these large units come into contact with fixed surfaces and other tools.

Any special equipment used for lifting, hauling or positioning large equipment should be thoroughly cleaned before being allowed into the cleanroom. Often this equipment may not be designed or maintained for cleanroom use and should be thoroughly inspected for chipping and flaking surfaces or for materials unsuitable for transfer into the cleanroom. These tools can often be made acceptable by means such as wrapping and sealing the tool with cleanroom-compatible plastic films and tape. Soft rubber wheels can be coated with cleanroom tape to avoid leaving trails of rubber or plastic particles on the flooring.

### D.4 Installation Procedures

An effective way to install equipment is to isolate the equipment from the rest of the cleanroom environment. Surrounding the equipment with a temporary isolation wall or partition can do this. An area should be left around the equipment to complete the installation unhindered.

- a) Access to the isolation area should be from a service aisle or other non-critical area, if possible. If access is not possible, measures should be taken to minimise the effects of construction-generated contamination. Airflow to this isolation area should be maintained at a neutral or negative pressure to reduce the possibility of contamination being forced outside the work area.
- b) A completely sealed isolation area should not be pressurised from within or the possibility of contaminating the surrounding cleanroom exists if a penetration of the barrier should occur. The clean-air supply inside the isolated area should be blocked to avoid pressurising the surrounding cleanroom. When entry to the isolated area is only accessible through an adjacent cleanroom, sticky mats should be installed to remove shoe-borne contamination. Once inside, disposable boots or shoe covers and coveralls may be required to avoid contaminating cleanroom clothing. These disposables should be taken off before leaving the isolated area.
- c) A method and frequency for monitoring the areas surrounding the isolated area should be instituted to ensure that any contamination that may leak into the adjacent cleanroom areas is detected.
- d) All facility services, such as electricity, water, gas, vacuum, compressed air, and waste piping, will then be attached. Care should be taken to ensure fumes and debris generated by this operation are controlled and contained as completely as possible to avoid inadvertent release to the surrounding cleanroom and facilitate effective cleaning before removal of the isolation barriers.
- e) Accepted cleaning procedures (see annex F) should then be used to decontaminate the entire isolation area.  
All surfaces should be vacuumed, wiped, and mopped; including all walls, both fixed and portable, equipment and floors.
- f) Special care should be taken to clean areas behind equipment panels and under equipment.

- g) Some internal preparation and preliminary performance testing of the equipment is now possible, but final acceptance may require full cleanroom conditions before final testing can be completed.
- h) The isolation walls can now carefully be removed and filtered air sources returned to service if deactivated. This step should be scheduled to minimise interruptions in the regular operation of the cleanroom. Particle measuring or testing may also be required.
- i) Equipment interiors and critical processing chambers should be cleaned and prepared for use under normal cleanroom conditions.
- j) All appropriate inner chambers and all surfaces coming into contact with the product or involved in the handling of the product should be wiped to achieve a desired cleanliness level. The cleaning procedure should be carried out, by working from the top to the bottom of the equipment, as once particles are disturbed, gravity will force larger particles to fall to the bottom of the equipment or to the floor.
- k) Clean the outer surfaces of the equipment, working from the top to the bottom surfaces.
- l) If necessary, surface particle checks should be performed in areas critical to product or process requirements.

## D.5 Maintenance and repair

Maintenance and repair of equipment should proceed without contaminating the cleanroom<sup>[27][28]</sup>. Successful completion of such repairs should include decontamination of external surfaces. Decontamination of internal surfaces may also be needed, if required by the process. The equipment should not only be in working condition, but steps should be taken to decontaminate internal and external surfaces consistent with processing requirements.

**D.5.1** The following measures can help to control contamination generated by maintenance of stationary equipment.

- a) Equipment being repaired should be removed from the area whenever possible before making repairs to reduce the possibility of generating contamination.
- b) If necessary, stationary equipment should be suitably isolated from the surrounding cleanroom operations before proceeding with major repairs or maintenance. Alternatively, steps should be taken to ensure that all products under manufacture have been removed to a suitable location.
- c) Adjacent cleanroom areas near the equipment being repaired should be suitably monitored to insure that contamination is being effectively controlled.
- d) Maintenance personnel working in the isolated areas should not come into contact with personnel performing manufacturing or processing procedures.
- e) All personnel repairing and maintaining equipment in cleanrooms should follow the appropriate practices defined for the area, including wearing appropriate garments, and cleaning the area and equipment after repairs are completed.
- f) A determination of conditions should be made before technicians lie or crawl under equipment to make repairs. Conditions caused by chemicals, acids, or biohazards should be effectively neutralised before proceeding.
- g) Steps should be taken to protect the cleanroom garment from undue contact with contamination from lubricating oils or processing chemicals. Rips and tears from sharp edges should also be avoided.
- h) All tools, boxes and carts used for maintenance or repair work should be thoroughly cleaned before being exposed to the cleanroom environment. No rusted or corroded tools should be allowed. Sterilisation or disinfection may be necessary in a biocleanroom.

- i) Technicians should refrain from setting tools; spare, damaged parts; or cleaning materials on adjacent or nearby work surfaces used for product and process materials.
- j) Care should be taken to clean as repairs proceed so that contamination does not build up.
- k) Gloves should be changed regularly so they do not deteriorate and permit bare skin to touch clean surfaces.
- l) When gloves other than cleanroom gloves (e.g. acid-, heat- or cut-resistant types) are required, they should be either cleanroom compatible or covered with a pair of cleanroom gloves.
- m) Vacuum cleaners should be used during all drilling or sawing operations. Maintenance and construction operations often require drilling or sawing. Special shrouds can be used to contain the tool and area being drilled or sawn.
- n) Open spaces remaining after holes are drilled in floors, walls, the sides of equipment or other such surfaces should be properly sealed afterwards to prevent contamination from entering the cleanroom. Methods for sealing may include use of caulks, adhesives and specially fabricated plates.

**D.5.2** With time, equipment wears out and becomes dirty or emits contamination unless it is maintained. Preventive maintenance should be carried out to ensure equipment is not allowed to become a source of contamination.

**D.5.3** When maintenance is complete, it may be necessary to verify the surface cleanliness of the equipment that was repaired or maintained.

## **D.6 Equipment removal**

Removing stationary equipment from the cleanroom often stirs up or loosens contamination from internal or other inaccessible surfaces that have not been routinely cleaned. This is especially true when the equipment must be disassembled before removal. Steps should be taken to isolate, clean, and contain such equipment before and during removal to avoid contaminating the surrounding cleanroom (see D.6.1).

Regulatory considerations may be involved if the contamination is of a hazardous nature.

## **Annex E** (informative)

### **Materials and portable equipment**

#### **E.1 General**

Items that can easily be transported into and out of the cleanroom can compromise the cleanliness of the cleanroom if they are not properly selected and handled. This includes consumable and disposable supplies, and production and cleaning materials as well as hand tools and portable equipment.

The physical attributes for materials and portable tools typically used in cleanrooms and or clean zones are discussed in E.5. These materials and portable tools should be selected to control cleanroom contamination and protect the process. The quantity of materials used in the cleanroom should also be limited to the minimum with due consideration of timing of delivery. The ability to sterilise reusable materials and portable equipment should be considered in biocleanroom applications.

#### **E.2 Criteria for Selection**

Basic criteria for selecting cleanroom-compatible materials that are allowed to enter the cleanroom are described in E.2.1 and E.2.2.

##### **E.2.1 Characteristics**

To protect a cleanroom from contamination, materials should:

- have surfaces and moving parts that shed or generate as little contamination as possible;
- have unbroken, impervious, and clean surfaces;
- have properties that minimise generation of contamination by shedding and cutting;
- be supplied in suitable cleanroom packaging;
- be evaluated for compatibility with the cleanroom environment.

##### **E.2.2 Other criteria**

The following additional criteria should be determined according to the purpose and usage within a cleanroom:

- free from chemicals (e.g. acid, alkali, organic);
- acceptable anti-static properties;
- low outgassing properties;
- free from micro-organisms;
- compatible with sterilisation procedures in biocleanrooms.

### **E.3 Preliminary testing**

Preliminary testing and auditing should be performed as agreed upon between customers and suppliers. Testing procedures performed by the supplier may be deemed sufficient for entry and use in the cleanroom.

Additional testing may be required for some materials before being used in the cleanroom. Incoming inspection criteria and sampling methods should be fully documented. A secure storage location may be necessary to avoid unauthorised use while materials are waiting for acceptance. Strict quarantine measures may be necessary for biologically sensitive materials. Test equipment and methods should be fully documented. Acceptance limits and authorised personnel should be identified for final approval or disposition of non-conforming materials. A procedure for communicating problems to the supplier should be instituted. The supplier should be expected to react with plans to improve its quality and avoid further shipment of non-conforming materials. The supplier should notify the customer prior to making critical changes to approved materials or supplies used in the cleanroom.

Evaluation methods and technologies should be reviewed periodically. Some incoming inspections may be eliminated when data show that the supplier has a proven quality record.

### **E.4 Entry and exit procedures**

Carrying materials into the cleanroom should not contribute contamination to the cleanroom. Materials and supplies that are carried into the cleanroom are subject to procedures similar to those described in annex D.

#### **E.4.1 Unpacking and entry procedures**

Only materials and portable equipment that are compatible with the cleanroom classification and use should be brought into the cleanroom. Outer contamination-generating packaging, such as wood, cardboard, paper and other materials, should be removed before entry to any part of the controlled or cleanroom environments. Inner plastic wrappers should not be removed at this time. Any interior packaging should be wiped with appropriately moistened cleanroom wipers to remove any gross contamination from the outer packaging before being carried into the controlled environment or specific area used for removing cleanroom packaging. Various types of non-wrapped portable equipment require careful cleaning before entering the cleanroom and are discussed in E.5. A designated area, such as an airlock, should be used for final wiping procedures. The changing area should be avoided for this purpose to avoid contaminating cleanroom clothing. A working surface and wiping materials should be readily available in this location for the task of cleaning all outer surfaces of the object to be transported into the cleanroom. The outer wrappers of double-wrapped packaging can now be removed and placed in an appropriate rubbish receptacle. Final packaging should only be removed prior to use of the material or object.

Any wheeled, portable equipment should be thoroughly cleaned before being allowed back into the cleanroom. Cleaning efforts should not overlook the surfaces of wheels that can transfer excess contamination directly onto the cleanroom floors. Sticky mats or flooring will help prevent this from occurring.

Cleanroom personnel, correctly dressed in cleanroom clothing, may then carry such items into the cleanroom via the airlock. A clean cart (trolley) may be used to transport many items from the airlock into the cleanroom.

#### **E.4.2 Entry through pipes**

Materials such as bulk chemicals, compressed gasses and water generally enter the cleanroom through pipes. Such materials are subject to the procedures that govern the introduction and intended use of those materials to the facility.

#### **E.4.3 Exit procedures**

Many items used by personnel are routinely removed when personnel leave the cleanroom. These items may include notebooks, pens, hand tools and other types of small portable equipment. These items should be protected from becoming contaminated through the use of approved plastic bags or other suitable means. This procedure will facilitate re-entry to the cleanroom at a future time.

Certain waste materials and portable equipment may have a higher risk of transferring contamination to personnel and their clothing. Steps should be taken to completely contain any such materials before transport and arrangements made to thoroughly clean such areas before personnel or processing is allowed to continue. Preferably, such materials should be removed through airlocks and not through changing areas.

## E.5 Types of materials and portable equipment

Materials designated for use in the cleanroom should conform to desired cleanliness levels. Considerations vary according to the desired use in the cleanroom. Many items typically used in cleanrooms are listed in E.5.1–E.5.16.

### E.5.1 Cleanroom clothing materials

Cleanroom clothing materials are described in annex B.

### E.5.2 Solutions and finishes used in cleaning

Cleaning solutions are used to aid in the removal of contamination from surfaces in the cleanroom. Some particles are floated off by the cleaning solution and others are pushed off through use of a wiper. After cleaning, certain finishes are also used to protect or preserve characteristics of surfaces in the cleanroom. These solutions and finishes should be as clean as required to meet the particle requirements of the cleanroom. The filtration of prepackaged solutions should be considered. The following are types of cleaning solutions and finishes:

- a) Clean-filtered, distilled, or deionised water has many desirable properties but such water can corrode certain types of surfaces and may be ineffective in cleaning without the addition of a surfactant or disinfectant.
- b) Surfactants and detergents are the most reasonably priced, nontoxic, nonflammable and effective cleaning agents. However, non-ionic surfactants are generally preferred for cleaning cleanrooms as this group is the least reactive and does not contain metallic ions.
- c) Organic solvents can also be used for removing contamination on hard surfaces. Organic films are best removed with organic solvents or detergents (detergents tend to leave behind a film).
- d) Disinfectants are used to kill micro-organisms. Care should be taken to select an appropriate material that will not contaminate the process or become harmful to personnel or equipment<sup>[38]</sup>.
- e) Synthetic sealers that are highly resistant to wear can be used on certain cleanroom floors. Antistatic floors require special care and sealers should not compromise the surface or electrical characteristics. Any sealing operations should only be done when cleanroom manufacturing is stopped or during general maintenance periods.

### E.5.3 Wipers

Wipers are used to remove contamination from surfaces in the cleanroom. Unfortunately, there is no single perfect wiper that suits every application within the cleanroom. Some wipers are absorbent but shed particles; others don't shed but do not absorb. Information on the selection of wipers is described in other sources<sup>[29]</sup>. The needs of the application should be considered and an appropriate evaluation should be performed. The following characteristics should be considered when selecting wipers for cleanroom use:

- a) wiper material;
- b) solution or solvent compatibility;
- c) absorption rate of liquids;
- d) particle generation (both wet and dry);
- e) extractable molecular contamination;

- f) sterilisation compatibility, if necessary;
- g) packaging.

#### **E.5.4 Vacuum cleaners, hoses and handles**

The selection and use of cleanroom-compatible vacuum cleaning equipment is important for an effective contamination control program.

- a) Portable vacuum cleaners are constructed of stainless steel or plastic. Exhaust must flow through a HEPA or ULPA filter before being allowed to escape to the surrounding environment. Vacuum cleaners capable of handling damp and liquid materials are also available for the cleanroom.
- b) Built-in vacuum cleaner systems employ a large, centralised vacuum pump, usually in a service area outside the cleanroom environment, that is connected by a system of plastic piping to wall outlets in each area of the cleanroom.
- a) Hoses, handles and tools should be matched to the application and constructed of cleanroom-compatible materials.
- b) Arrangements should be made for routine inspection and maintenance of all equipment used in the vacuum cleaning process. The HEPA or ULPA filters of the vacuum cleaning equipment should be tested and/or replaced on a regular basis to ensure that they do not become a source of airborne contamination in the cleanroom.

#### **E.5.5 Mops**

Standard commercial or industrial grade mops and handles should not be used within the cleanroom environment (including changing and other controlled areas). Mops should be carefully selected to resist the effects of sterilization, if so required. Floor mop heads should be constructed of polyester fibres or open-celled hydrophilic (synthetic) materials. Block or sponge mop heads should be constructed of open-celled hydrophilic (synthetic) sponge material. Handles and fittings should be made of stainless steel, anodized aluminum, fiberglass coated with polypropylene, or other non-shedding plastics and should be compatible with the operation of the cleanroom mops. Roll mops (similar to paint rollers), with a slightly sticky surface, may be used when appropriate for removing contamination from wall surfaces without applying any moisture. These are available in both renewable and disposable forms.

**NOTE** When purchasing synthetic mops or handles, one should be cognisant of the intended cleaning application. Polyvinyl acetate (or equivalent) mop heads are acceptable when used with aqueous cleaning solutions. However, the mop heads will prematurely deteriorate when used with cleaning agents containing high levels of isopropyl alcohol. Some materials used in handles or mop heads are not compatible with steam sterilisation. Polyester offers better resistance to autoclaving than polyvinyl acetate.

#### **E.5.6 Buckets and wringers**

Buckets or containers with wringers that are compatible with the cleanroom operation are required for wet or damp cleaning operations.

Buckets and containers should be constructed of plastic or stainless steel (not galvanised). Stainless steel buckets can be repeatedly autoclaved.

The wringer system used in mopping should be compatible with the style and material of the mop head.

#### **E.5.7 Floor scrubbers, buffing and waxing machines**

Standard commercial floor scrubbers or buffers should never be used within an operating cleanroom, as the process would contaminate the environment.

Special machines designed for scrubbing cleanroom floors are available. These machines have special shrouds and built-in HEPA filtered vacuum cleaners to control contamination redistribution. They also have HEPA filtered exhaust casings for the motor chambers. Careful evaluation for compatibility should precede the use of such equipment in the cleanroom.

Cleanrooms should never have waxes or non-permanent sealers applied to the floors since these materials flake off from traffic, causing contamination. For specific types of flooring refer to ISO 14644-4.

#### **E.5.8 Stepladders**

Stepladders should be of anodised aluminum or reinforced fiberglass and should not leave the cleanroom controlled area. They should be thoroughly cleaned (disinfected or sterilised if necessary) before entry.

#### **E.5.9 Brooms or brushes**

Brooms, brushes, or similar tools should not be used in an operational cleanroom, as they will cause gross particle generation. The bristles themselves are very large fibres that are also a contaminant.

#### **E.5.10 Receptacles for rubbish and recycling**

Used materials, by-products and other waste generated inside the cleanroom should be removed as soon as possible. A means for the collection, containment and storage of wastes should be provided to protect the cleanroom from these contamination sources while waiting for removal. Removal procedures are discussed in F.4.10.

The following criteria should be considered when selecting receptacles for collection of these materials:

- a) nature of the materials to be discarded or recycled;
- b) safety requirements;
- c) environmental hazards;
- d) lining materials and how they will be installed;
- e) floor space available;
- f) size required, based on frequency of collection;
- g) material of construction;
- h) cleanroom compatibility.

#### **E.5.11 Cleanroom mats and sticky flooring**

Cleanroom mats and sticky flooring can be used as a barrier to help control foot-borne contamination from entering the cleanroom. The size (particularly the length) and location of the mats/flooring are the major factors governing the effectiveness for the removal of foot-borne contamination. Two major varieties of available mats/flooring are:

- 1) Disposable — Multiple layers of adhesive, plastic film with the sticky surface facing up. Layers are removed and discarded as they get dirty.
- 2) Reusable — Resilient polymeric mat with a naturally sticky surface, to be cleaned when it becomes dirty.

### **E.5.12 Clean containers and packaging**

Clean containers can be used for transporting or isolating sensitive materials and products to and from the cleanroom, while waiting to be used or processed. Surface cleanliness and isolation characteristics should be consistent with the intended use of the enclosed materials. Entry procedures as stated in E.4 should be followed. Frequent cleaning may be necessary to avoid contamination build up during use. Special cleaning and verification of cleanliness may be required before reuse.

Materials that may be used to protect or package finished products made in the cleanroom should be clean and compatible with the cleanroom. Selection should be based on particle generation, microbial contamination, electrostatic properties, outgassing and other concerns. Tapes that are used within the cleanroom should have adhesives that leave minimal residues when removed.

### **E.5.13 Hand tools, boxes and maintenance equipment**

Hand tools should be compatible with the cleanroom classification, products, stationary equipment and processes with which they will come into contact. They should be kept clean and free from contamination of all kinds.

Boxes or cases that contain tools and other repair or diagnostic equipment are often overlooked sources of contamination. They should be made of stainless steel or synthetic materials that resist or protect against the generation or transfer of contamination. Any use of molded inserts or dividers that can generate contamination such as open-cell foam, vinyl-covered wood or pressboard (wood-chip board) should be avoided. Boxes should be thoroughly cleaned on a regular and scheduled basis (with tools and instruments removed) to ensure cleanliness. Tools and instruments should be cleaned before being replaced inside the toolbox or case. Tool boxes and cases should remain inside the cleanroom whenever possible. If removed from the environment, toolboxes and cases should never be opened outside the cleanroom. Thorough external cleaning should be required before being allowed back inside the cleanroom.

Carts and trolleys routinely used for transferring maintenance and other supplies into and out of the cleanroom must be thoroughly cleaned before re-entry.

Initial and routine sterilization or disinfection of the above items may be required when used in the biocleanroom.

### **E.5.14 Safety equipment**

Safety goods and equipment used in the cleanroom such as chemical gloves, aprons, face and arm shields, self-contained breathing apparatus, chemical absorbing pads and fire extinguishers should be selected for their intended safety requirements as well as compatibility with the intended cleanroom.

### **E.5.15 Documentation**

Contamination from documents, within the cleanroom, should be controlled. Methods for documentation will depend largely on the use and classification of the cleanroom.

#### **E.5.15.1 Electronic methods**

The use of computers for work in progress will eliminate the need for many sources of contamination such as, log books, log sheets, process documentation and others. Installation and use of computers and peripherals should be compatible with the classification of and intended location inside the cleanroom.

Computers often employ internal cooling fans. Consideration should be given to how the exhaust air may affect the cleanroom and critical surfaces surrounding the computer. Methods may need to be devised to duct such exhaust directly to air returns or through portable filtration units, depending on cleanliness requirements. Keyboards have recesses around the pushbuttons that may trap and release particles. Use of flexible continuous films or covers placed over the keyboard will facilitate cleaning and reduce contamination. Printers interfaced with such computers should be appropriately contained or isolated and exhausted in a similar manner. Printer maintenance should be performed carefully to avoid dispersal of residual contamination generated by the printing operation.

### **E.5.15.2 Paper documents**

Paper and paper products will contaminate the cleanroom. All documents should be printed on lint-free, cleanroom-compatible media or thermally laminated between plastic films. Information on the selection of such substrates is given in other sources<sup>[30]</sup>. Use of such media in the form of labels, log sheets, equipment repair manuals, reports and notebooks should be controlled and kept to a minimum. Label adhesives should leave minimal residues when removed from surfaces.

### **E.5.15.3 Writing instruments**

Writing instruments can become sources of contamination to the cleanroom, products or processes.

Pencils and felt-tipped pens should be avoided. Pens should be of the non-retractable, ballpoint style with inks that are permanent and compatible.

### **E.5.15.4 Other materials**

Many other materials, including those directly used in the production process, are taken into the cleanroom. They should exhibit the lowest possible contamination properties for the application intended and the classification of the cleanroom. They should enter and be controlled in an appropriate manner as described above and be compatible with products and processes.

## **E.6 Storage**

Materials can become contaminated or ineffective if improperly stored while waiting to be used. Proper storage and controlled storage methods are critical to preserve their effectiveness. They should be stored in an environment that protects them from degradation and contamination. If not properly stored the accumulation of unused materials in the cleanroom presents a risk of contamination.

Certain classes and types of waste materials are stored in the cleanroom until specified limitations are reached. Often these limits are regulated by agencies or by recycling programs set up for the cleanroom. The use of specialised containers may also be required.

## Annex F (informative)

### Cleanroom cleaning

#### F.1 Overview

Cleanrooms are designed to be as free from contamination as possible. Facility and maintenance operations, manufacturing processes, the presence and activity of personnel and other factors may cause contamination to be generated and dispersed on surfaces in the cleanroom. Therefore, all surfaces should be cleaned frequently enough to prevent this from becoming a risk to the manufacturing process. Procedures should be specified to ensure that thorough and complete cleaning operations are performed in a manner that is consistent with recommended cleanroom practices for the facility. When feasible, cleaning should be avoided during manufacturing operations. If this is not possible, special cleaning procedures should be devised to minimise risks.

NOTE Some processes generate contamination as a by-product. It is better to identify and attempt to contain contamination from such operations than to rely on cleaning to control the contamination.

Information is available in a number of documents that will assist in effective cleaning of a cleanroom<sup>[29][30][31][32]</sup>.

#### F.2 Surface classification

The cleanliness of areas and surfaces should be classified and designated based upon how they may affect the products and processes performed in the cleanroom. Effective application of this classification will be useful in developing the proper cleaning strategy for the cleanroom.

##### F.2.1 Critical surfaces

Surfaces classified as critical are located at and around the point of manufacture or production where contamination can gain direct access to the product or process. Unidirectional airflow equipment, clean benches or workstations usually help control the cleanliness of these surfaces. These surfaces are the most critical and should be kept the cleanest.

##### F.2.2 General cleanroom surfaces

All surfaces within the cleanroom that are not at the point of production or localised by unidirectional airflow are considered "general." They should be cleaned on a regular basis to prevent transfer of contamination onto critical surfaces.

##### F.2.3 Surfaces of changing rooms and airlocks

Surfaces of changing rooms and airlocks can become highly contaminated due to the high level of activity. Frequent cleaning is necessary to minimise the level of contamination and to reduce the transfer of contamination into the cleanroom.

#### F.3 Basic cleaning

Maintaining the cleanliness of a cleanroom is a meticulous set of tasks. Cleaning levels should be defined and basic methods for attaining those levels should be developed. Approved methods can then be applied to every surface within the cleanroom to achieve the desired result<sup>[10][31][32][33][34]</sup>.

### F.3.1 Basic cleaning categories

The act of cleaning can be divided into three different categories depending on the current state and desired cleanliness of the surface once cleaning is completed. These are **gross**, **intermediate** and **precision**.

**Gross cleaning** involves the removal of large particles of contamination usually greater than 50 µm in diameter. Contamination of this size is usually found on floors and is typical of the type carried into changing areas and airlocks. Broken or spilled materials resulting from the production operation or process are additional sources of contamination that end up on work surfaces and floors. Construction and equipment maintenance activities also can often generate gross particle contamination.

**Intermediate cleaning** involves the removal of smaller particles of contamination, typically ranging from 10 µm to 50 µm in diameter. Performed on general cleanroom surfaces, intermediate cleaning is usually associated with walls, benches and clean hallways. This size of contamination remains after gross cleaning methods are used. Intermediate cleaning provides the next level of cleanliness.

**Precision cleaning** is needed to remove remaining particulate contamination that is generally less than 10 µm in diameter. Precision cleaning is generally employed on or near critical surfaces where product is stored and processed.

### F.3.2 Vacuum cleaning

Vacuum cleaning can be used in gross and intermediate cleaning operations as a basic first step to cleaning both general and critical areas. Vacuum cleaning is a prerequisite, not an alternative, to mopping or wet wiping. Vacuum cleaning is effective in removing larger particles and other debris such as glass fragments. Vacuum cleaning should be performed in deliberate, unidirectional strokes to minimize air turbulence at the floor level and at operator height.

The use of HEPA/ULPA vacuum cleaners or in-house central vacuum systems is employed in vacuum cleaning. Systems that can accommodate wet materials are helpful for removing excess water and suspended particles during and after the mopping process. Vacuum cleaning can also be useful in helping to speed the drying process once mopping is completed.

### F.3.3 Wet cleaning

Wet cleaning methods can be employed in all stages of cleaning.

**Scrubbing** is a gross cleaning method that employs machine or manual methods to remove stains or heavily soiled areas. Scrubbing is followed by mopping or wet vacuuming procedures. Care should be taken to control any contamination that may be generated by the equipment or materials used in scrubbing.

**Mopping** is an effective method in gross or intermediate cleaning for removing particulate contamination. Mopping can also be used for removing residues from spilled liquids left after wet vacuuming is completed. Wet wipers may be used in small or localised areas. Mops are used for floors and other large areas. The mop bucket should be filled with clean-filtered de-ionised or distilled water and changed frequently to avoid recontamination. The more critical the surface, the more frequently the water should be changed. Water discoloration indicates the bucket should be cleaned and refilled in the gross cleaning mode. Intermediate and critical areas should show little or no discoloration, so cleaning procedures for these areas should define the allowable surface area to be cleaned before changing the water. Two (or multiple) bucket systems can be used to reduce the frequency of rinse water changes. Non-ionic detergents or surfactants can be added if necessary. Mops should be well squeezed to avoid puddles. A damp mop will produce a damp surface that will dry more quickly. A systematic method, using overlapping strokes should be employed to ensure complete cleaning of floor surfaces. Frequent rinsing and turning of mop surfaces help to avoid recontamination of previously cleaned sections of the floor. Mop heads should be rinsed frequently to avoid recontamination of the mop head. Specialty mops are also available for removing intermediate-sized contamination from walls, and floors (see E.5.5).

### **F.3.4 Damp cleaning**

Wiping techniques are used in most phases of cleaning. Wiping produces results that support intermediate and precision cleanliness for general and critical surfaces. The chosen wiper should be dampened with the appropriate cleaning solution. The solution is dependent upon the type of contaminant being removed. Wiping should always be done in unidirectional, overlapping strokes, proceeding from most critical to least critical areas, following the direction of unidirectional airflow. As wiping proceeds, wipers should be folded to provide an unused surface area. The wiper should be replaced as frequently as needed to avoid transferring contaminants to other parts of the cleanroom surface.

## **F.4 Cleaning specific surfaces**

All surfaces within the cleanroom that become contaminated should be cleaned. It is important that all surfaces be identified according to criticality to the product or process performed in the cleanroom. Cleaning techniques can then be developed and specified to ensure that the required level of cleanliness is attained.

### **F.4.1 Floors and subfloors**

Gross contamination can be removed first by vacuum cleaning e.g. glass or product fragments. Areas with stubborn stains should then be identified and addressed with predetermined scrubbing procedures. The floor should be wet mopped according to predetermined procedures. Water or cleaning solutions should be changed frequently enough to minimise the spread of dissolved or suspended contamination as the cleaning process continues. Larger floor areas should be divided into manageable segments so that work can proceed in an orderly manner. Cleaning should begin in critical areas and proceed through general areas, but certain cleanroom applications may require a different routine. Repeating the mopping procedure will produce cleaner surfaces if greater cleanliness levels are required.

During operational hours, it may be necessary to cordon off the area and redirect traffic flow to avoid dangerous falls by unwary personnel. Damp mopping or wet vacuuming after mopping will speed the drying process.

Wet washer/scrubber systems, followed by wet vacuum cleaning, can be used to remove stubborn stains and floor stains. These systems are described in E.5.7 and should be thoroughly cleaned before and after each use.

### **F.4.2 Walls, doors, return grilles, windows and vertical surfaces**

Surfaces, upstream from product exposure, should never be cleaned in the operational state. Upstream surfaces should only be cleaned in the at-rest state or after products have been removed from the area or covered. Contamination should be removed using wiping methods or special purpose or roll mops. Choice of method should be determined based on state of cleanliness desired and configuration of the area being cleaned.

### **F.4.3 Ceilings, diffusers and lamp fixtures**

Ceilings and other fixtures upstream of work areas should not be cleaned in the operational state but should wait for at-rest conditions. Diffuser and ceiling grids should be carefully wiped using damp cleaning techniques. Some diffusers may require removal for washing or replacement. Lamp fixtures should be thoroughly wiped whenever bulbs are changed.

### **F.4.4 Tables and other critical horizontal surfaces**

These critical surfaces should be cleaned using appropriate wiping techniques described above. Acceptable cleaning solutions may be used to aid in contamination removal. Damp wipers can be used to remove contamination, working in unidirectional strokes from most to least critical areas.

### **F.4.5 Cleanroom chairs, furniture and ladders**

Wipe these surfaces from top to bottom. Include cushions, supports, and wheels if appropriate.

#### **F.4.6 Stationary equipment**

See annex D.

#### **F.4.7 Carts and trolleys**

Carts and trolleys should be restricted to the cleanroom area only and should not be cleaned in any critical areas or in the airlock. Carts and trolleys should be vacuumed or wiped down, or both, using wipers and starting from the top and working down using acceptable cleaning solutions.

#### **F.4.8 Hazardous processing surfaces**

Procedures should be developed to neutralise existing hazards before beginning normal cleanroom cleaning procedures. Use the appropriate cleaning technique for the surface involved and described in F.4.1 to F. 4.11.

#### **F.4.9 Cross-over benches, garment and supply cabinets, lockers, and other compartmented surfaces**

Compartments should be periodically emptied so that the interiors can be cleaned. Vacuum cleaning followed by wiping will effectively remove contamination from exposed surfaces.

#### **F.4.10 Rubbish bins and containers**

Rubbish bins and containers can be lined with plastic bags to facilitate removal of refuse and protect container surfaces. Rubbish should be removed before it collects in excess. Plastic bags or liners should never be removed from bins in the vicinity of critical areas. All bins should be removed to general, non-critical areas before any rubbish is removed. This can be done as required or at the end of each shift. They should be emptied, cleaned and re-lined, if required, before being returned to service.

#### **F.4.11 Cleanroom mats and sticky flooring**

Cleanroom mats and sticky flooring should be cleaned or maintained on a regular basis during the normal workday. Cleanroom mats and sticky flooring should be serviced according to the manufacturer's instructions as frequently as needed. Mats with renewable surfaces should be cleaned frequently. After wet mopping, a rubber squeegee is used to pull contamination and water to the edge to be mopped dry. A wet vacuum with a squeegee head can also be used for this purpose.

Mats with removable, sticky surfaces are cleaned by slowly peeling each of the four corners and rolling the film towards the middle of the mat until the layer is removed.

### **F.5 Surface treatment**

Specific cleanroom applications require that certain surface treatments or finishes are applied to cleanroom surfaces, to provide characteristics that normally would not exist. These treatments may protect the products being produced in the cleanroom, but should be carefully considered. The use of surface treatments and finishes, after cleaning, should be avoided if at all possible. These treatments deteriorate with time and will compromise the cleanliness of the cleanroom. In addition, these treatments can pose the risk of process or product contamination if not used or maintained properly. Surfaces that receive these treatments should be inspected or tested on a periodic basis to ensure they do not compromise the cleanroom. Steps can then be taken to remedy the situation.

#### **F.5.1 Anti-static treatment**

Anti-static materials can be applied to surfaces to minimise static charge build up. Treating surfaces with anti-static agents should be done carefully. Improper use will result in non-uniform, anti-static characteristics and residues that can become a source of contamination. The coating should be thick enough to be effective but thin enough to

avoid flaking and generation of contamination. Anti-static surface characteristics can often be achieved, simply, by changing the humidity of the air supplied to the cleanroom.

### **F.5.2 Disinfection**

Thorough cleaning programs help to control microbiological contamination. However, certain industries and regulatory agencies require disinfection procedures in addition to normal cleaning procedures. Additional disinfection procedures help ensure products are not exposed to or contaminated by microbes. The effectiveness of the disinfectants and the methods used for disinfection should be determined in each cleanroom. In general, disinfectant efficacy is a function of the type of disinfectant, its concentration and its contact time on the surface being disinfected. Some disinfectants can damage cleanroom surfaces (e.g. chlorine-based compounds on stainless steel) if they are not properly removed and may be toxic if they are deposited on products. Therefore, it may be appropriate to remove such residues by rinsing the surfaces with sterile water. Disinfectants can have harmful effects on personnel if used improperly.

### **F.6 Cleaning personnel**

A specific training program should be provided for any personnel performing the cleaning operation. Specific personnel should be designated for each part of the cleaning program. It is quite common to assign cleanroom cleaning to specialised cleaning personnel. Operators, with proper training, are often assigned to clean the work surfaces they use.

### **F.7 Cleaning program**

The classification of different kinds of cleanroom surfaces and the rate at which they become contaminated should be understood when setting up a cleaning program. Schedules should be specified to ensure cleaning is performed frequently enough to maintain the required cleanliness of the cleanroom. Testing and evaluation of the surface contamination will assist in drawing up schedules. The process and product within the cleanroom should determine which tasks need to be accomplished on a daily, weekly, or other periodic basis.

#### **F.7.1 Preparing a cleaning program**

The following steps should be followed in preparing a cleaning program.

- a) Classify all surfaces into critical, general or other surfaces.
- b) Determine the best cleaning and surface treatment method for achieving the desired cleanliness level.
- c) Determine the cleaning frequency required to maintain the desired cleanliness levels for each surface type.
- d) Determine which cleaning operations can be accomplished during normal operating hours.
- e) Prepare the cleaning schedules.
- f) Decide which part of the cleaning schedule operators will execute and which part cleaning staff will execute.
- g) Choose the correct materials, machines, cleaning solutions and surface treatments for the specified methods.
- h) Train all personnel for the expected level of involvement in the cleaning program.
- i) Provide adequate storage facilities for the required cleaning materials.
- j) Decide how to monitor the cleaning results and react to discrepancies.
- k) Organize all documents and schedules so that they can be reviewed and managed effectively.

## **F.7.2 Schedule considerations**

Most cleaning operations should be performed on a regularly scheduled and frequent basis. Other cleaning operations are performed on a scheduled basis but infrequently. Some cleaning operations must be done in reaction to events that create contamination and are not subject to normal scheduling. The frequencies in F.7.2.1 and F.7.2.2 can be used as guidelines but should be adjusted to the needs of the cleanroom as based on a risk assessment and cleaning evaluation.

### **F.7.2.1 Daily cleaning**

If specified, daily cleaning is done in general areas at least once in a 24-hour period. Many tasks may be allowed during working hours, such as trash removal, vacuuming, mopping floors, and wiping surfaces in changing areas, passthroughs and common halls. Each room within the cleanroom may need a special written cleaning program depending on criticality of cleanliness to product or process concerns.

Air locks and changing areas should be cleaned at least daily. These areas can harbour high contamination levels, due to the high level of personnel activity. Therefore, cleaning is required more frequently than in manufacturing cleanrooms to control the cleanliness level and reduce the opportunity for contamination transfer. Daily cleaning will enhance the level of cleanliness within the general cleanroom areas. Thorough vacuum cleaning and mopping procedures described in F.3.2 and F.3.3 should be implemented. Cleanroom mats and sticky flooring should be serviced as described in F.4.11 but with greater frequency to prevent the migration of contamination into the cleanroom.

### **F.7.2.2 Periodic cleaning**

Surfaces not cleaned on a daily basis should be cleaned periodically. Special precautions may need to be taken to ensure product integrity during cleaning procedures.

Many surfaces should be cleaned on a weekly basis (i.e. at least once during a seven-day period). Product may need to be covered or removed from areas where weekly service is performed.

Surfaces that present less of a risk can be scheduled for less frequent cleaning. This type of less frequent cleaning should be performed on a monthly or longer time interval. Schedules should reflect these less frequent intervals.

Arrangements should also be made to thoroughly clean the entire cleanroom facility, from top-to-bottom, on a scheduled basis. Thorough cleaning should include storage areas, service areas, pipes and fittings. Thorough cleaning is often best accomplished during extended facility shutdowns or during weekends, holidays or other planned facility shutdowns. Continuously operating cleanrooms only shut down sporadically and may only have certain times when thorough cleaning can be accomplished. Intensive cleaning efforts should be taken at these times to accomplish the task.

## **F.7.3 Cleaning during and after construction or maintenance**

Effective cleaning during cleanroom construction is essential to control and eliminate contamination sources. Effective cleaning is as important in operational cleanrooms as it is in new construction. See annex D for maintenance-related procedures. The sample 10-step cleaning schedule in F.9 can be used to aid in planning, executing, and documenting efforts. Annex D provides guidance for maintenance activities.

## **F.7.4 Cleaning during emergency situations**

Procedures should be instituted to ensure that work in progress, the process and the cleanroom environment are not compromised in the case of a gross contamination event. Special tools and materials should be readily available to neutralise or control any hazardous situations that may arise. Events that may trigger special cleaning include:

- a) environmental incident (e.g. utility failure, spill, major equipment failure, broken product, biological hazard, etc.);

- b) failure of routine cleaning procedures resulting in contamination rising to unacceptable levels;
- c) monitoring that reveals the occurrence of unacceptable contamination of the facility.

Work should be suspended in the area deemed at risk until acceptable levels of cleanliness are attained.

## F.8 Monitoring cleaning effectiveness and testing

**F.8.1** Cleanroom equipment, apparatus or surfaces may require cleanliness testing after cleaning. Users are responsible for selecting appropriate cleanliness verification methods. An acceptable degree of cleanliness should be determined for each element or characteristic that will affect the products or processes in the cleanroom. The user should specify limits for tests performed. It is recommended that, when possible, limits be determined from actual measurements, using the test methods. Routine surface contamination checks should be defined and carried out to ensure that the specified levels are being maintained.

Visual inspection techniques can be used to determine surface cleanliness. Visual-clean surfaces demonstrate an absence of soiling that can be seen without magnification. Visual inspection may be accomplished with or without the aid of high-intensity white light or ultraviolet light sources. Wiper-clean surfaces can be demonstrated by passing a clean wiper over a clean surface. This inspection aid detects visual contamination that may adhere to the wiper surface indicating further cleaning is needed. Coloured wipers are available from some suppliers and may be helpful in detecting some forms of contamination. Other methods that may be considered include:

- a) tape lift method<sup>[35]</sup>;
- b) surface particle detector method<sup>[31]</sup>.

NOTE Additional methods for measuring surface cleanliness in critical areas are discussed in other sources<sup>[10][31][33][36]</sup>.

**F.8.2** A variety of methods and sampling schemes exist for detecting microbiological contamination in the cleanroom. These are described in other sources<sup>[38]</sup>. The following methods are most common:

- a) Contact plates (for flat surfaces);
- b) Surface swabbing (for uneven surfaces).

## F.9 Construction-related cleaning program

Depending on the user requirements, the following 10-stage program can be used effectively to schedule, assign, and document cleaning procedures that are needed during different phases of construction operations (see also ISO 14644-4, annex E).

**Table A.1 — Stages of construction-related cleaning program**

| Stage  | Purpose   | Responsibility   | Method                        | Standard      |
|--|---|--|-------------------------------|---------------|
| <b>Stage 1 —</b><br>Clean during demolition/<br>framing. | Prevent unnecessary dust concentration in places that will be difficult to reach during later construction. | Contractor. If the contractor has no relevant experience in cleanroom cleaning, it is advisable to hire a professional cleaning contractor specialising in cleanroom cleaning. | Vacuum clean upon completion. | Visual-clean. |

| Stage   | Purpose  | Responsibility  | Method  | Standard   |
|---|--|---|---|--|
| <b>Stage 2 —</b><br>Clean during utility installation.                      | Removing local contaminants caused by installing electricity, gas, water, etc.   | Installation engineer.  | Vacuum clean; wipe-down piping and fixtures with moistened wipers upon completion. The use of vacuum cleaning and/or other cleaning materials is necessary. | Visual-clean.  |
| <b>Stage 3 —</b><br>Early construction cleaning.                            | All visible contamination should be cleaned from ceilings, walls, floors, (filter mountings), etc. after completion of construction and installation activities. | Cleaning contractor.  | Vacuum clean; wipe-down piping and fixtures with moistened wipers. If necessary a protective sealing could be put on the floor.                             | Visual-clean.  |
| <b>Stage 4 —</b><br>Preparation for air conditioning ductwork installation. | Clean any dust from ductwork sections before installing using a vacuum cleaner and wipers. Meanwhile, a positive pressure should be introduced to the cleanroom. | Installation engineer and cleaning contractor.  | Vacuum clean; wipe down with moistened wipers.  | Wiper-clean.   |
| <b>Stage 5 —</b><br>Clean before mounting all air filters into the system.  | Remove deposited or settled dust, or both, from ceilings, walls, and floors.   | Cleaning contractor.  | Wipe down with moistened wipers.  | Wiper-clean.   |
| <b>Stage 6 —</b><br>Mount the (HEPA/ULPA) filters into the air systems      | Removing possible contamination caused by the mounting operation.  | Cleanroom HVAC filter engineer/ technician.   | Clean all surface edges on all sides.   | Wiper-clean.   |
| <b>Stage 7 —</b><br>Adjust the air conditioning equipment.                  | Remove suspended dust from the airflow and create over-pressure installation, including the filters.   | Cleanroom HVAC filter engineer/ technician.   | Air conditioning air flushing operation.  | Wiper-clean.   |
| <b>Stage 8 —</b><br>Upgrade the room into prescribed classification.        | Remove all deposited and clinging dust from every surface (in order: ceilings, walls, equipment, floors).  | A professional cleanroom cleaning by personnel specially instructed on regulations, routing and behavior. | Wipe down with moistened wipers.  | Wiper-clean.   |
| <b>Stage 9 —</b><br>Validation.   | To verify the cleanroom to the prescribed design specifications. Customer acceptance.  | Installation engineer and certification engineer.   | Monitor airborne and surface particles, air velocities, temperature, and humidity.  | Wiper-clean. Results should conform to agreed design criteria. |

| Stage  | Purpose  | Responsibility                               | Method                       | Standard   |
|--|--|--|------------------------------|--|
| <b>Stage 10</b> —<br>Daily and<br>periodic<br>cleaning   | Maintaining the<br>cleanroom in long-<br>term compliance with<br>designed<br>classification.<br>Microbiological<br>cleaning and testing<br>begins in<br>biocleanrooms. | Cleanroom<br>manager/cleaning<br>contractor. | Listed in annex F.1–<br>F.8. | A tailor-made cleaning<br>program for the<br>cleanroom, accounting<br>for the specific<br>demands of the<br>production process<br>and the customer.<br>Routine testing of<br>critical operation<br>parameters. |
| <p>NOTE 1 During Stages 4–10, all high efficiency and ultra-high purity components, such as filters, ducts, etc. should arrive on site protected by a plastic or foil covers on both ends. Covers should only to be removed when ready for use.</p> <p>NOTE 2 During Stages 6–10, all activities should be done wearing prescribed cleanroom clothing.</p> |  |  |                              |  |

## Bibliography

- [1] Kletz, T.A.: *Hazop and Hazan: Identifying and assessing process industry hazards*. London: Taylor & Francis Group, 1999.
- [2] Pierson, M.D. and Corlett, D.A. Jr.: *HACCP principles and applications*. New York: Van Nostrand Rheinhold, 1992.
- [3] CEI/IEC 812:1985, *Analysis techniques for system reliability — Procedure for failure mode and effective analysis (FMEA)*. Geneva, Switzerland: Commission Electrotechnique Internationale / International Electrotechnical Commission.
- [4] Palady P.: *FMEA, failure modes and effect analysis*. West Palm Beach, Florida: PT Publications, Inc., 1995.
- [5] CEI/IEC 61025:1990, *Fault tree analysis (FTA)*. Geneva, Switzerland: Commission Electrotechnique Internationale / International Electrotechnical Commission.
- [6] IEST-RP-CC027.1:1999, *Personnel practices and procedures in cleanrooms and controlled environments*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [7] AS 2013.1:1989, *Cleanroom garments: product requirements*. North Sidney: Standards Association of Australia.
- [8] IEST-RP-CC003.2:1993, *Garments required in cleanrooms and controlled environments*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [9] VCCN-RL-6.2:1996, *Cleanroom garments: Recommended practices for choice, logistics and use of cleanroom garments*. Amersfoort: Dutch Society of Contamination Control (Dutch language only).
- [10] VDI 2083 part 4:1996, *Cleanroom technology — Surface cleanliness*. Berlin: Beuth Verlag GmbH.
- [11] ISO 9237:1995, *Textiles — Determination of permeability of fabrics to air*.
- [12] ASTM-D737-96:1996, *Test method for air permeability of textile fabrics*. West Conshohocken, Pennsylvania: American Society for Testing and Materials.
- [13] JIS B 9923:1997, *Methods for sizing and counting particle contaminants in and on clean room garments*. Tokyo: Japanese Industrial Standards.
- [14] ASTM F51-68:1989, *Standard methods for sizing and counting particulate contamination in non-cleanroom garments*. West Conshohocken, Pennsylvania: American Society for Testing and Materials.
- [15] EN 1149-1:1994, *Protective clothing — Electrostatic properties — Part 1 Surface resistivity (test methods and requirements)*.
- [16] IEST-RP-CC022.1:1992, *Electrostatic charge in cleanrooms and other controlled environments*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [17] AS 2013.2:1989, *Cleanroom garments: Processing and use*. North Sidney: Standards Association of Australia.
- [18] ISO 11092:1993, *Textiles — Physiological effects — Measurement of thermal and water-vapour resistance under steady-state conditions (sweating guarded-hotplate test)*.
- [19] BS 7209:1990, *Water vapour permeable apparel fabrics*. London: British Standards Institution.

- [20] ISO 7730:1995, *Moderate thermal environments — Determination of the PMV and PPD Indices and specification of the conditions for thermal comfort*.
- [21] VCCN-RL-5:1996, *Thermal comfort: Recommended practices for thermal comfort requirements for people working in cleanrooms*. Amersfoort: Dutch Society of Contamination Control. (Dutch language only).
- [22] IEST-RP-CC005.2:1996, *Cleanroom gloves and finger cots*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [23] S2C2 Procedure Number 1:1998, *Cleanroom disciplines and entry procedures*. Glasgow: Scottish Society for Contamination Control.
- [24] VDI 2083 part 6:1996, *Cleanroom technology — Personnel at the clean work place*. Berlin: Beuth Verlag GmbH.
- [25] JACA Number 14C:1992, *Guidance for operation of clean rooms*. Tokyo: Japan Air Cleaning Association (Japanese language only).
- [26] VCCN-RL-6.3:1996, *Rules for behaviour in the cleanroom: Recommended practices for personnel behavior in cleanrooms*. Amersfoort: Dutch Society of Contamination Control (Dutch language only).
- [27] IEST-RP-CC026.1:1995, *Cleanroom operations*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [28] JIS B 9926:1991, *Test methods for dust generation from moving mechanisms*. Tokyo: Japanese Industrial Standards.
- [29] IEST-RP-CC004.2:1992, *Evaluating wiping materials used in cleanrooms and other controlled environments*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [30] IEST-RP-CC020.2:1996, *Substrates and forms for documentation in cleanrooms*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [31] IEST-RP-CC018.2:1995, *Cleanroom housekeeping — Operating and monitoring procedures*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.
- [32] JACA Number 27:1992, *Guidance for cleaning of clean room facilities*. Tokyo: Japan Air Cleaning Association (Japanese language only).
- [33] VCCN-RL-4:1996, *Surface cleanliness: Recommended practices for microbiological and particle surface cleanliness, and cleaning in cleanrooms*. Amersfoort: Dutch Society of Contamination Control (Dutch language only).
- [34] JACA Number 32:1996, *Guideline for cleaning of biological clean room facilities*. Tokyo: Japan Air Cleaning Association (Japanese language only).
- [35] ASTM E 1216-87:1987, *Practice for sampling for surface particulate contamination by tape lift*. West Conshohocken, Pennsylvania: American Society for Testing and Materials.
- [36] JACA Number 22:1988, *A guideline of measuring methods for surface particle contamination*. Tokyo: Japan Air Cleaning Association (Japanese language only).
- [37] JACA Number 30:1993, *The report of the surface contamination control technology survey committee*. Tokyo: Japan Air Cleaning Association (Japanese language only).
- [38] IEST-RP-CC023.1:1993, *Microorganisms in cleanrooms*. Mount Prospect, Illinois: Institute of Environmental Sciences and Technology.

**ICS 13.040.30**

Price based on 18 pages

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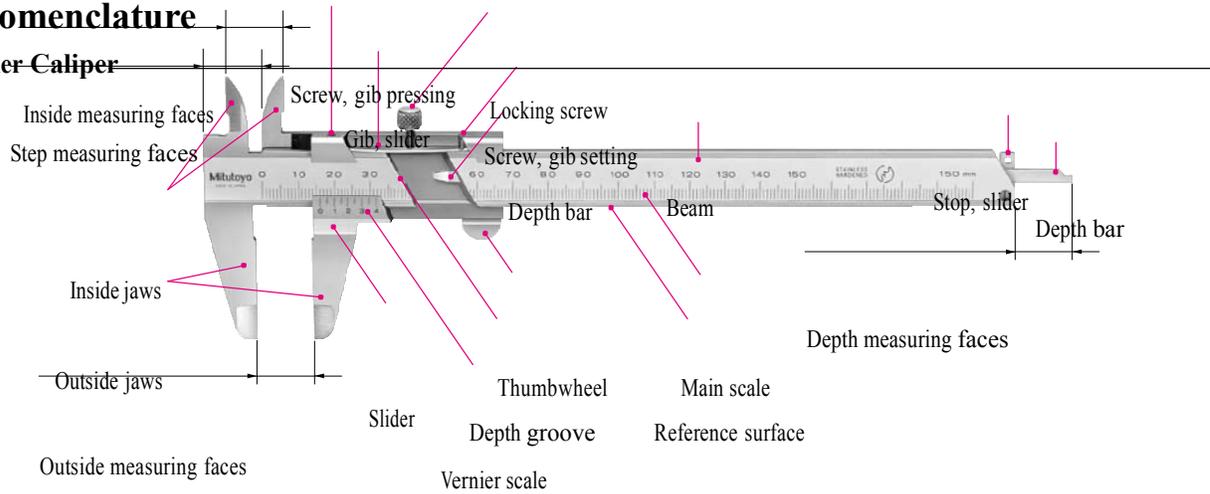
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## E2. Dimensional Measurements (Guide from Mitutoyo, manufacturer of measurement equipment)

### Calipers

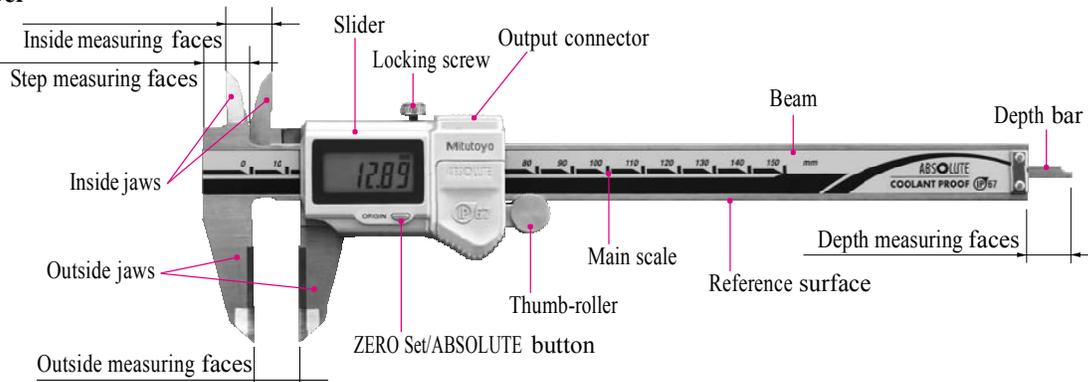
#### ■ Nomenclature

##### Vernier-Caliper

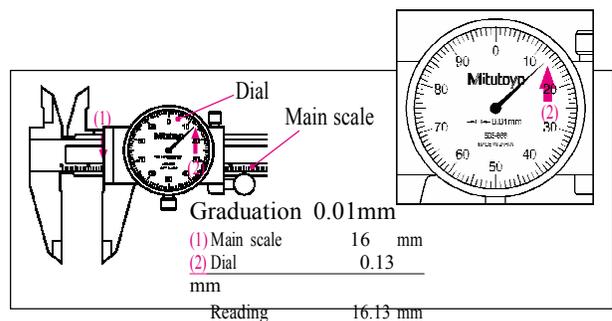
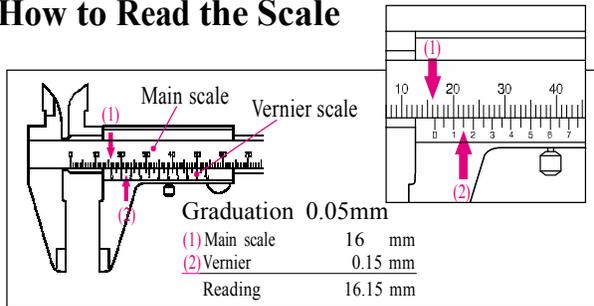


##### Absolute Caliper

##### Digital

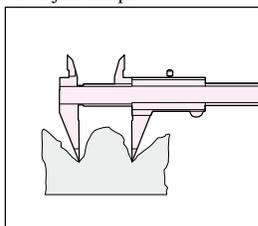


#### ■ How to Read the Scale



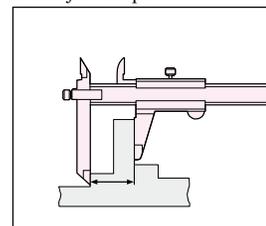
#### ■ Special Purpose Caliper Applications

##### Point jaw caliper



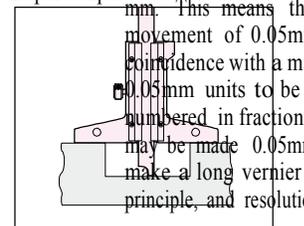
For uneven surface measurement

##### Offset jaw caliper



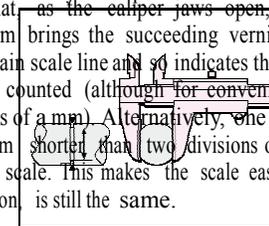
For stepped feature measurement

##### Depth caliper



For depth measurement

##### Blade jaw caliper

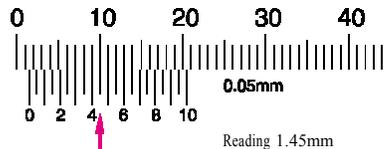


For diameter of narrow groove measurement

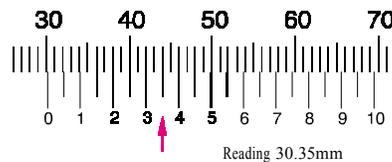
#### ■ Types of Vernier Scale

The Vernier scale is attached to the caliper's slider and each division on this scale is made 0.05mm shorter than one main scale division of 1 mm. This means that as the caliper jaws open, each successive movement of 0.05mm brings the succeeding vernier scale line into coincidence with a main scale line and so indicates the number of 0.05mm units to be counted (although for convenience the scale is numbered in fractions of a mm). Alternatively, the vernier division may be made 0.05mm shorter than two divisions of the main scale to make a long vernier scale. This makes the scale easier to read but the principle, and resolution, is still the same.

- Standard Vernier scale (resolution 0.05mm)



- Long Vernier scale (resolution 0.05mm)

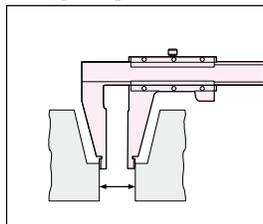


## ■ Sources of Error

Main sources of error include scale misreading (parallax effect), excessive measuring force causing jaw tilt, thermal expansion caused by a temperature difference between the caliper and workpiece, and small-hole diameter error caused by inside jaw offset. There are other minor error sources such as graduation accuracy, reference edge straightness, main scale flatness and squareness of the jaws. These sources are allowed for within the specified accuracy of a new caliper and only cause significant error in case of wear or damage.

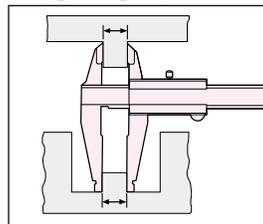
The JIS standard emphasizes that care must be used to ensure that measurement is performed with an appropriate and constant measuring force, since a caliper has no constant-force device, and that the user must be aware of the increased possibility of error due to measuring a workpiece using the tips of the jaws (Abbe's Principle).

CM-type caliper



For outside measurement  
For measurement of inside bore

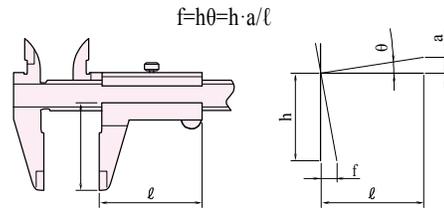
CN-type caliper (with knife-edge)



For outside measurement  
For stepped feature measurement

## ■ Moving Jaw Tilt Error

If the moving jaw becomes tilted out of parallel with the fixed jaw, either through excessive force being used on the slider or lack of straightness in the reference edge of the beam, a measurement error will occur as shown in the figure. This error may be substantial due to the fact that a caliper does not conform to Abbe's Principle.



Example: Assume that the error slope of the jaws due to tilt of the slider is 0.01mm in 50mm and the outside measuring jaws are 40mm deep, then the error (at the jaw tip) is calculated as  $(40/50) \times 0.01\text{mm} = 0.008\text{mm}$ .  
If the guide face is worn then an error may be present even using the correct measuring force.

## ■ About Long Calipers

Steel rules are commonly used to roughly measure large workpieces but if a little more accuracy is needed then a long caliper is suitable for the job. A long caliper is very convenient for its user friendliness but does require some care in use. In the first place it is important to realize there is no relationship between resolution and accuracy. Resolution is constant whereas the accuracy obtainable varies dramatically according to how the caliper is used.

The measuring method with this instrument is a concern since distortion of the main beam causes a large amount of the measurement error, so accuracy will vary greatly depending on the method used for supporting the caliper at the time. Also, be careful not to use too much measuring force when using the outside measuring faces as they are furthest away from the main beam so errors will be at a maximum here. This precaution is also necessary when using the tips of the outside measuring faces of a long-jaw caliper.

## ■ Inside Measurement with a CM-type Caliper

Because the inside measuring faces of a CM-type caliper are at the tips of the jaws the measuring face parallelism is heavily affected by measuring force, and this becomes a large factor in the measurement accuracy attainable.

In contrast to an M-type caliper, a CM-type caliper cannot measure a very small hole diameter because it is limited to the size of the stepped jaws, although normally this is no inconvenience as it would be unusual to have to measure a very small hole with this type of caliper. Of course, the radius of curvature on the inside measuring faces is always small enough to allow correct hole diameter measurements right down to the lowest limit (jaw closure).

Mitutoyo CM-type calipers are provided with an extra scale on the slider for inside measurements so they can be read directly without the need for calculation, just as for an outside measurement. This useful feature eliminates the possibility of error that occurs when having to add the inside-jaw-thickness correction on a single-scale caliper



## Standard Test Methods for Pore Size Characteristics of Membrane Filters by Bubble Point and Mean Flow Pore Test<sup>1</sup>

This standard is issued under the fixed designation F316; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (  $\epsilon$  ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 These test methods cover the determination of two of the pore size properties of membrane filters with maximum pore sizes from 0.1 to 15.0  $\mu\text{m}$ .

1.2 Test Method A presents a test method for measuring the maximum limiting pore diameter of nonfibrous membranes. The limiting diameter is the diameter of a circle having the same area as the smallest section of a given pore (Fig. 1).

1.3 Test Method B measures the relative abundance of a specified pore size in a membrane, defined in terms of the limiting diameter.

1.4 The analyst should be aware that adequate collaborative data for bias statements as required by Practice D2777 is not provided. See the precision and bias section for details.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

E128 Test Method for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use

### 3. Terminology

3.1 *Definitions*—For definitions of other terms used in these test methods, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

Current edition approved Jan. 10, 2003. Published April 2003. Originally published as F316–70. Last previous edition F316–86. DOI: 10.1520/F0316-03.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

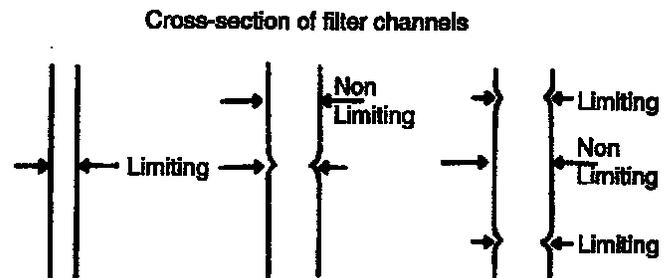


FIG. 1 Examples of Limiting Diameters

3.2.1 *pore size*—capillary equivalent pore diameter.

3.2.2 *limiting pore diameter*—diameter of a circle having the same area as the smallest section of a given pore.

### TEST METHOD A—MAXIMUM PORE SIZE

#### 4. Summary of Test Method

4.1 The bubble point test for maximum pore size is performed by prewetting the filter, increasing the pressure of gas upstream of the filter at a predetermined rate and watching for gas bubbles downstream to indicate the passage of gas through the maximum diameter filter pores.

4.2 The pressure required to blow the first continuous bubbles detectable by their rise through a layer of liquid covering the filter is called the  $\text{\textcircled{b}}$  bubble point $\text{\textcircled{b}}$ , and is used to calculate maximum pore size.

#### 5. Significance and Use

5.1 This test method may be used to:

5.1.1 Determine the maximum pore size of a filter,

5.1.2 Compare the maximum pore sizes of several filters, and

5.1.3 Determine the effect of various processes such as filtration, coating, or autoclaving on the maximum pore size of a membrane.

5.2 Membrane filters have discrete pores from one side to the other of the membrane, similar to capillary, tubes. The bubble point test is based on the principle that a wetting liquid is held in these capillary pores by capillary attraction and surface tension, and the minimum pressure required to force liquid from these pores is a function of pore diameter. The



pressure at which a steady stream of bubbles appears in this test is the bubble point pressure. The bubble point test is significant not only for indicating maximum pore size, but may also indicate a damaged membrane, ineffective seals, or a system leak.

5.3 The results of this test method should not be used as the sole factor to describe the limiting size for retention of particulate contaminants from fluids. The effective pore size calculated from this test method is based on the premise of capillary pores having circular cross sections, and does not refer to actual particle size retention. See Test Method E128 for additional information.

### 6. Apparatus

6.1 *Filter Holder*, as shown in Fig. 2, consisting of a base A, a locking ring B, O-ring seal C, support disk D, and gas inlet E. The support disk shall be 2-ply construction, consisting of a 100 by 100 mesh or finer screen and a perforated metal plate for rigidity. The diameter of the test filter may be either 25 or 47 mm, as appropriate to the holder being used for the test.

6.2 *Manifold*, as shown in Fig. 3, a micrometric flow control valve capable of providing a linear rise in pressure and a gas ballast of at least 16 000-cm<sup>3</sup> capacity.

NOTE 1—For less accurate determinations, the simplified apparatus shown in Fig. 4 may be used.

6.3 *Pressure Gages* (and mercury manometer if required), covering the range of pressures needed for the pore sizes under investigation (see Table 1).

6.4 *Metal Punch*, used to cut a suitable size filter from the test sheet to fit the test filter holder.

### 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society

where such specifications are available.<sup>3</sup> Other grades may be used provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

7.2 *Water*, conforming to Specification D1193, Type IV or higher purity.

7.3 *Denatured Alcohol*.

7.4 *Petroleum Distillate*, with surface tension of 30 dynes/cm at 25°C.

7.5 *Mineral Oil*, such as USP liquid petrolatum heavy, with surface tension of 34.7 dynes/cm at 25°C.

7.6 *1,1,2-trichloro-1,2,2-trifluoroethane (Freon TFt)*, available from commercial chemical supply houses.

7.7 *Clean Gas Pressure Source*, with regulation (filtered air or nitrogen).

NOTE 2—Table 1 lists the nominal surface tension of these liquids at 25°C. Table 2 lists the simplified maximum pore size formulas based on these values, where the liquid completely wets the membrane.

### 8. Procedure

8.1 Wet the test membrane completely by floating it on a pool of the liquid. Use a vacuum chamber to assist in wetting the filter, if needed.

8.2 Place the wet membrane in the filter holder.

8.3 Close the filter holder and apply slight gas pressure to eliminate possible liquid back flow.

8.4 Cover the perforated metal plate with 2 to 3 mm of test liquid.

8.5 Increase the gas pressure slowly. Record the lowest pressure at which a steady stream of bubbles rises from the central area of the liquid reservoir.

NOTE 3—Faulty sealing may cause erroneous bubbling from the sealing edge of the liquid reservoir. Be sure to record the bubble point pressure with bubbles from the central area of the reservoir (see Fig. 5).

### 9. Calculation

9.1 If the test liquid is known to wet the membrane completely, calculate the maximum pore size from the following equation:

$$d = 5 Cg/p \tag{1}$$

where:

$d$  = limiting diameter,  $\mu\text{m}$ ,

$g$  = surface tension,  $\text{mN/m}$ , (dynes/cm),

$p$  = pressure, Pa or cm Hg, and

$C$  = constant, 2860 when  $p$  is in Pa, 2.15 when  $p$  is in cm Hg, and 0.415 when  $p$  is in psi units.

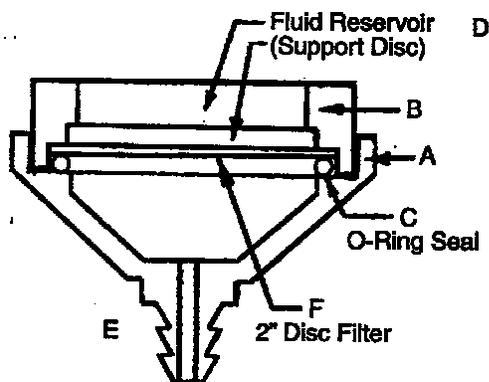
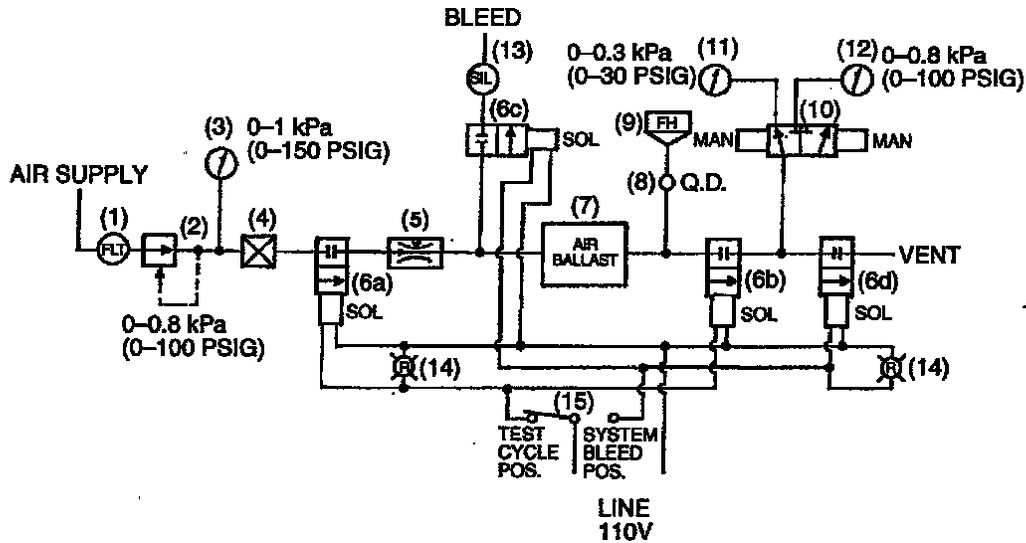


FIG. 2 Filter Holder

<sup>3</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



| Key | Quantity | Component                         |
|-----|----------|-----------------------------------|
| 1   | 1        | Filter                            |
| 2   | 1        | Pressure regulator                |
| 3   | 1        | Pressure gage                     |
| 4   | 1        | Valve shutoff, manual             |
| 5   | 1        | Valve, flow control, manual       |
| 6   | 4        | Valve, solenoid, nc               |
| 7   | 1        | Air ballast                       |
| 8   | 1        | Quick disconnect fitting          |
| 9   | 2        | Open filter holder, 47 mm         |
| 10  | 1        | Valve, 3-way, manual              |
| 11  | 1        | Test gage, 0-0.3 kPa (0-30 psig)  |
| 12  | 1        | Test gage, 0-0.8 kPa (0-100 psig) |
| 13  | 1        | Exhaust silencer                  |
| 14  | 2        | Pilot light, red, elec.           |
| 15  | 1        | Switch, spdt, elec.               |

FIG. 3 Manifold for Bubble Point Testing

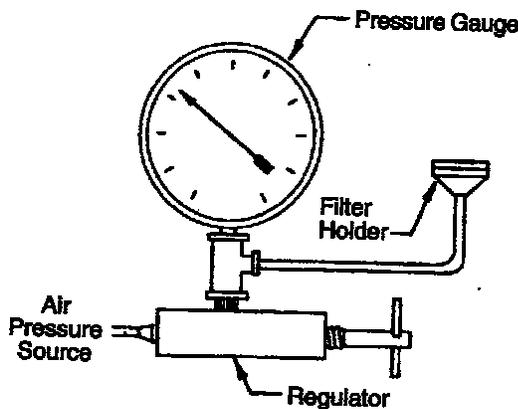


FIG. 4 Test Setup (Simplified)

NOTE 4—The fluid must completely wet the membrane filter with the contact angle being zero. If the contact angle is greater than zero, the calculated effective pore size will be larger than the actual effective pore size rating.

## 10. Reporting Results

10.1 Record the minimum pressure for gas passage as indicated by continuous bubbles. Record the maximum pore

size calculated, along with identification of the membrane tested and the liquid used.

## TEST METHOD B—DETERMINATION OF PORE SIZE DISTRIBUTION

### 11. Summary of Test Method

11.1 A fluid-wet filter will pass air when the applied air pressure exceeds the capillary attraction of the fluid in the pores. Smaller pores will exhibit similar behavior at higher pressures. The relationship between pore size and pressure has been established, as indicated in Table 2.

11.2 By comparing the gas flow rates of both a wet and dry filter at the same pressures, the percentage of the flow passing through the filter pores larger than or equal to the specified size may be calculated from the pressure-size relationship. By increasing pressure in small steps, it is possible to determine the flow contribution of very small pore size increments by difference.

11.3 To determine the mean flow pore size, a pressure is sought at which the wet filter flow is one half of the dry filter flow.

### 12. Significance and Use

12.1 This test method may be used to:

**TABLE 1 Pressure Ranges Required**

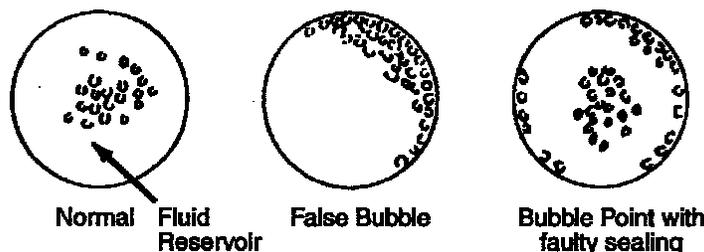
| Fluid Used           | Pore Size Range to be Investigated (Alternative psi) |                                 |                                  |                                   | Surface Tension, dynes/cm at 25°C |
|----------------------|--|---------------------------------|----------------------------------|-----------------------------------|-----------------------------------|
|                      | \$1 μm   | \$0.5 μm                        | \$0.1 μm                         | \$0.05 μm                         |                                   |
| Water                | 0 to 155 cm Hg<br>(0 to 30 psi)                      | 0 to 310 cm Hg<br>(0 to 60 psi) | 0 to 775 cm Hg<br>(0 to 150 psi) | 0 to 3100 cm Hg<br>(0 to 600 psi) | 72.0                              |
| Petroleum distillate | 0 to 65 cm Hg<br>(0 to 15 psi)                       | 0 to 130 cm Hg<br>(0 to 25 psi) | 0 to 325 cm Hg<br>(0 to 65 psi)  | 0 to 1300 cm Hg<br>(0 to 250 psi) | 30.0                              |
| Denatured alcohol    | 0 to 50 cm Hg<br>(0 to 10 psi)                       | 0 to 100 cm Hg<br>(0 to 20 psi) | 0 to 250 cm Hg<br>(0 to 50 psi)  | 0 to 1000 cm Hg<br>(0 to 200 psi) | 22.3                              |
| Mineral oil          | 0 to 75 cm Hg<br>(0 to 15 psi)                       | 0 to 150 cm Hg<br>(0 to 30 psi) | 0 to 305 cm Hg<br>(0 to 75 psi)  | 0 to 1500 cm Hg<br>(0 to 300 psi) | 34.7                              |
| Freon TFI            | 0 to 40 cm Hg<br>(0 to 8 psi)                        | 0 to 80 cm Hg<br>(0 to 16 psi)  | 0 to 200 cm Hg<br>(0 to 40 psi)  | 0 to 800 cm Hg<br>(0 to 100 psi)  | 17.3                              |

**TABLE 2 Calculation of Maximum Pore Size from Bubble Point Pressure,<sup>A</sup>  $d = C1/P$**

NOTE—Refer to [Appendix X1](#) for derivation of maximum pore size formula.

| Fluid Used           | Pressure Units of Bubble Point |                   |                                  |
|----------------------|--------------------------------|-------------------|----------------------------------|
|                      | cm Hg                          | psi               | Pa                               |
| Water                | 155/Bubble Point               | 30.0/Bubble Point | 2.06 $\times 10^5$ /Bubble Point |
| Petroleum distillate | 64.6/Bubble Point              | 12.5/Bubble Point | 8.58 $\times 10^4$ /Bubble Point |
| Denatured alcohol    | 47.75/Bubble Point             | 9.25/Bubble Point | 6.38 $\times 10^4$ /Bubble Point |
| Mineral oil          | 74.5/Bubble Point              | 14.4/Bubble Point | 9.92 $\times 10^4$ /Bubble Point |
| Freon TFI            | 37.2/Bubble Point              | 7.2/Bubble Point  | 4.95 $\times 10^4$ /Bubble Point |

<sup>A</sup> Example—A certain filter was observed to have a bubble point of 41.0 psi with petroleum distillate:  
 Size (μm) = 12.5/41.0 psi  
 Therefore, maximum pore size = 0.305 μm.



**FIG. 5 Examples of Erroneous Bubble Points**

- 12.1.1 Determine the pore size distribution of a membrane filter,
- 12.1.2 Determine the mean flow pore size of a membrane filter,
- 12.1.3 Determine the effect of processes such as sterilization, upon the pore size distribution,
- 12.1.4 Measure the maximum pore size of a membrane filter, and
- 12.1.5 Determine the gas flow rate of a filter, and thereby its gas flow capability.

**13. Apparatus**

- 13.1 *Clean Gas Pressure Source*, with regulation (filtered air or nitrogen).
- 13.2 *Pressure Gage* (or set of gages), covering the necessary pressure range for the pore sizes under study. (See [Table 1](#).)
- 13.3 *Closed Filter Holder*, (see [Fig. 6](#)).

NOTE 5—Two 1-in. (25-mm) filter holders, instead of one holder, eliminate the task of cleaning the filter holder after a fluid-wet air flow

determination. A two-position gas valve to switch the gas pressure from dry to wet holders may be used (see [Fig. 7](#)).

- 13.4 *Set of Rotameters*, covering the range from 0 to 100 L/min.
- 13.5 *Test Tube and Capillary Tube*, to serve for detection of the bubble point.
- 13.6 *In-Line Fluid Trap*, to protect the rotameters from the fluid.
- 13.7 *Appropriate Fittings, Hose, Connectors, Piping*, to assemble apparatus as shown in [Figs. 6 and 7](#).
- 13.8 *x-y Recorder*, to assist in graphing results of mean flow pore test.

**14. Reagents**

- 14.1 *Mineral Oil*, such as USP liquid petrolatum heavy.

**15. Procedure for One Holder ( [Fig. 6](#) )**

- 15.1 Place a dry filter sample disk in the filter holder.
- 15.2 Close the holder and apply gas pressure in increments.

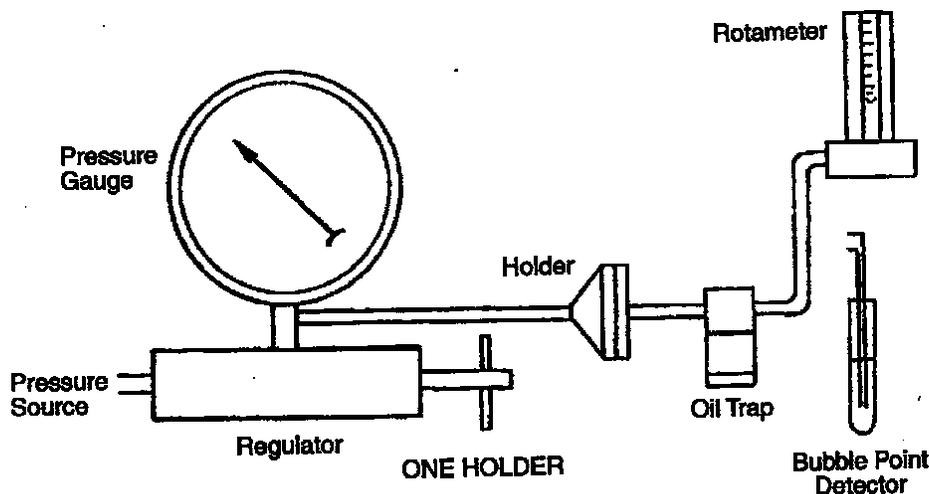


FIG. 6 Setup for One Holder

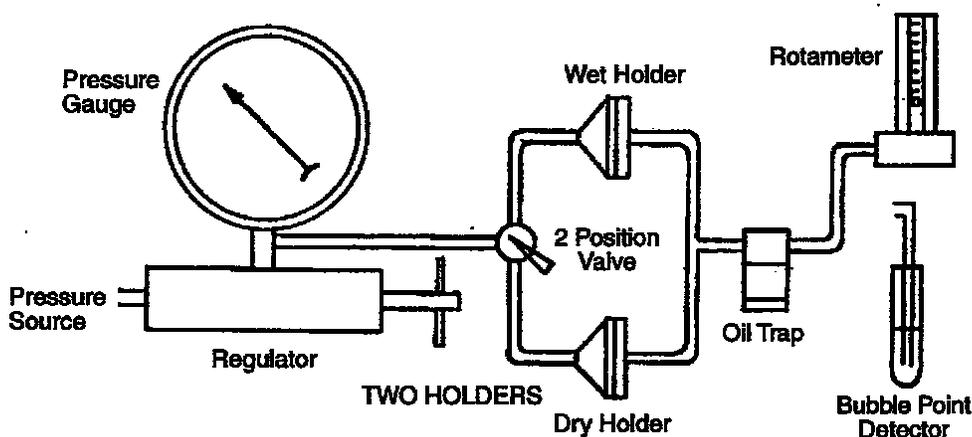


FIG. 7 Setup for Two Holders

15.3 Plot gas flow versus gas pressure over the intended range of use. (Estimate from pore size-pressure formula, see Table 1.)

15.4 Reduce gas pressure and remove the filter from the holder.

15.5 Completely wet the filter in mineral oil, replace the wet filter in the holder, and apply a slight pressure, checking to see that a tight seal exists.

15.6 Increase pressure gradually and record the pressure of the first gas flow detected by the capillary tube tip immersed in the test tube half-filled with mineral oil.

15.7 Change to rotameters and plot the fluid-wet filter gas flow versus pressure on the same coordinates as for the plot made in accordance with 15.3.

15.8 Reduce the pressure, remove the filter, and clean the holder for the next test.

**16. Procedure for Two Holders ( Fig. 7 )**

16.1 Place a dry filter in the holder to be used exclusively for dry filters.

16.2 Wet a filter of the same type and lot as the filter used in 16.1, in mineral oil and place it in a holder to be used exclusively for wet filters.

16.3 Apply gas pressure to the dry filter and plot gas flow versus gas pressure.

16.4 Change the two position valve to apply gas pressure to the wet filter holder and record the first gas flow as detected by the capillary tube as the bubble point. Switch to rotameters and plot fluid-wet gas flow versus gas pressure.

**17. Calculation of Mean Flow Pore Size**

17.1 Record the minimum pressure for gas passage (bubble point pressure). Calculate the maximum pore size from the formula in 9.1 and correlation data in Table 2.

17.2 Using the graph from 16.3, draw the line (or curve) corresponding to one half the dry gas-flow rate of the tested filter. Find the intersection of this (one-half dry-flow) line and the wet flow line (or curve), as shown in Fig. 8. Determine the pressure coordinate of the intersection and substitute into the pressure-pore size formula. (See Table 2 for mineral oil.)

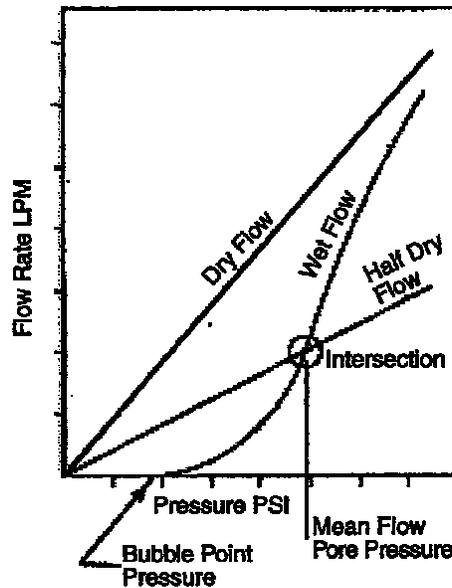


FIG. 8 Example of Mean Flow Pore Determination

18. Calculation of Pore Size Frequency

18.1 Select the limits of the pore size range being evaluated. Substitute the limits individually into the pore-size versus pressure formula and obtain their respective pressures. From the test graph, determine the wet and dry flows at the pressure limits (pore size limits of the range) as shown in Fig. 9.

18.2 Substitute the values found in the following equation:

$$Q = FS \frac{\text{wet flow}_h}{\text{dry flow}_h} \geq S \frac{\text{wet flow}_l}{\text{dry flow}_l} DG \geq 100 \quad (2)$$

where:

- Q = filter flow, %
- l = lower pressure limit, and
- h = higher pressure limit.

18.3 Report Q as the percentage of the filter flow passing through pores within the range specified.

NOTE 6—Example—Determine the percentage of the filter flow passing

through a pore size range from 0.8 to 0.2 μm of the filter described in Fig. 9.

$$Q = FS \frac{7 \text{ L/min}}{21 \text{ L/min}} \geq S \frac{2 \text{ L/min}}{15 \text{ L/min}} \geq 100$$

$$= 0.333 \geq 0.133 \geq 100$$

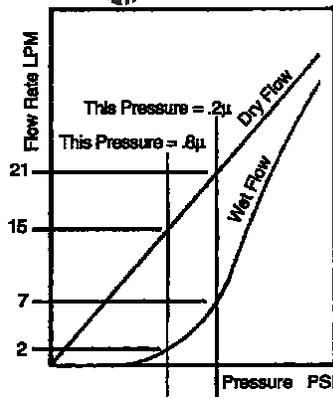
$$= 20\% \quad (3)$$

Twenty percent of the gas passing through the filter described in Fig. 9 moves through pores between 0.8 and 0.2 μm.

19. Precision and Bias

19.1 Results should not differ from the mean by more than the following amounts:

| Pore Size Range   | Repeatability, %<br>Same Operator<br>and Apparatus | Reproducibility, %<br>Different Operator<br>and Apparatus |
|-------------------|--|---|
| Greater than 1 μm | 2  | 4   |
| 1 to 0.5 μm       | 1  | 2.5   |
| Less than 0.5 μm  | 0.5  | 1   |



**FIG. 9 Example of Pore Size Frequency Determination**

FIG. 9 Example of Pore Size Frequency Determination

19.2 Since the change in pore size per unit change in applied pressure is greatest at large pore sizes and diminishes hyperbolically, repeatability and reproducibility increase accordingly with applied pressure.

## APPENDIX

### (Nonmandatory Information)

#### X1. PORE SIZE FORMULA DERIVATION

X1.1 The pore size formula is derived as follows:

Surface tension formula:

$$g \sin \theta = \frac{2 \cos \theta B}{d} \tag{X1.1}$$

Rearranging for diameter (2r):

$$d = \frac{4 \cos \theta B}{g \sin \theta} \tag{X1.2}$$

At bubble point pressure  $P = hgp$ :

$$d = \frac{4 \cos \theta B}{p} \tag{X1.3}$$

When a fluid wets a filter  $\cos \theta = 1$ :

$$d = \frac{4B}{p} \tag{X1.4}$$

where:

$h$  = height of rise,  
 $p$  = density of fluid,

$g$  = gravitational constant,

$r$  = radius of tube,

$\theta$  = contact angle,

$B$  = capillary constant, and

$\gamma$  = surface tension.

Substituting pressure conversion factors and the capillary constant:

For centimetres of Hg:

$$d = \frac{2.145 \text{ g-dynes/cm}^2}{P \text{ -cm Hg}} \tag{X1.5}$$

For psi:

$$d = \frac{0.415 \text{ g-dynes/cm}^2}{P \text{ -psi}} \tag{X1.6}$$

For Pa:

$$d = \frac{2860 \text{ g-dynes/cm}^2}{P \text{ -Pa}} \tag{X1.7}$$

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## **E4. Filter Weighing Procedures**

### **E4.1 NIST Handbook145 SOP2**

#### **SOP 2**

#### **Recommended Standard Operations Procedure for**

#### **Applying Air Buoyancy Corrections**

##### **1. Introduction**

###### **1.1. Purpose**

If uncorrected, the effect of air buoyancy on the objects being compared is frequently the largest source of error in mass measurement. This SOP provides the equations to be used to correct for the buoyant effect of air. The significance of the air buoyancy correction depends upon the accuracy required for the measurement, the magnitude of the air buoyancy correction relative to other sources of error in the overall measurement process, and the precision of the mass measurement. An air buoyancy correction should be made in all high accuracy mass determinations. The gravimetric volume procedure uses a high accuracy mass determination with the corresponding buoyancy corrections. The Appendix to this SOP provides a brief theoretical discussion of this subject.

###### **1.2. Prerequisites**

1.2.1. Verify that (true) mass values or corrections are available for the standards used in the measurement process. The calibration values for the mass standards must be traceable to NIST.

1.2.2. Verify that the thermometer, barometer, and hygrometer used have been calibrated and are in good operating condition as verified by periodic tests or cross-checks with other standards.

##### **2. Methodology**

###### **2.1. Scope, Precision, Accuracy**

This procedure is applicable to all weighings using a comparison of mass standards. The precision will depend upon the accuracy of the thermometer, barometer, and hygrometer used to determine the air density. When the calculations for the air density and air buoyancy corrections are made, a sufficient number of decimal places must be carried so the error due to the rounding of numbers is negligible relative to the error in the measurement process. Typically, carrying six to eight decimal places is sufficient, but depends upon the precision of the measurement.

## 2.2. Apparatus/Equipment Required<sup>1</sup>

2.2.1. Barometer accurate to  $\pm 66.5$  Pa (0.5 mm Hg) to determine air pressure.

2.2.2. Thermometer accurate to  $\pm 0.10$  °C to determine air temperature.

2.2.3. Hygrometer accurate to  $\pm 10$  % to determine relative humidity.

## 2.3. Estimating the Magnitude of the Air Buoyancy Correction

2.3.1. Estimate the magnitude of the air buoyancy correct, *MABC*, using the following formula:

$$MABC = (\rho_a - \rho_n)(V_x - V_s)$$

2.3.2. The equation may also be represented as follows:

$$MABC = m_o (\rho_a - \rho_n) \left( \frac{1}{\rho_x} - \frac{1}{\rho_s} \right)$$

**Table 1. Variables for *MABC* equation**

| Variable | Description of Variable  |
|----------|--|
| $\rho_a$ | air density at the time of the measurement in mg/cm <sup>3</sup> |
| $\rho_n$ | density of "normal" air; i.e., 1.2 mg/cm <sup>3</sup>            |
| $m_o$    | nominal mass (in grams)  |
| $V_x$    | volume of the unknown weight, X in cm <sup>3</sup>               |
| $V_s$    | volume of the reference standard, S in cm <sup>3</sup>           |
| $\rho_s$ | density of reference standard, S in g/cm <sup>3</sup>            |
| $\rho_x$ | density of unknown weight, X in g/cm <sup>3</sup>                |

The relative magnitude of the correction can be compared to the expanded measurement uncertainty to determine the importance of the air buoyancy correction and the uncertainty components for a particular measurement.

<sup>1</sup> The barometer, thermometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air buoyancy correction. The accuracies specified are recommended for high precision calibration. Less accurate equipment can be used with only a small degradation in the overall accuracy of the measurement.

## 2.4. Procedure

- 2.4.1. Record the temperature, pressure, and relative humidity at the start and at the end of the measurement process as near the location of the measurement as necessary and practical. If these parameters change significantly during the measurement process, it may be necessary to wait for more stable operating conditions or to use average values to compute the air density. Use of the average environmental values may influence the uncertainty of the measurement result and must be evaluated for significance.
- 2.4.2. Determine the air density using the equation given in Section 8 of the Appendix to this SOP.

## 3. Calculations

- 3.1. Calculate the mass,  $M_x$ , of the unknown weight,  $X$ , using the following equation, where  $d$  represents the “difference” obtained with buoyancy corrections applied to the sensitivity weight.

$$M_x = \frac{M_s \left( 1 - \frac{\rho_a}{\rho_s} \right) + d}{\left( 1 - \frac{\rho_a}{\rho_x} \right)}$$

If tare weights were carried with  $X$  and/or  $S$ , use the following equation:

$$M_x = \frac{M_s \left( 1 - \frac{\rho_a}{\rho_s} \right) + d + M_{t_s} \left( 1 - \frac{\rho_a}{\rho_{t_s}} \right) - M_{t_x} \left( 1 - \frac{\rho_a}{\rho_{t_x}} \right)}{\left( 1 - \frac{\rho_a}{\rho_x} \right)}$$

**Table 2. Variables not previously defined**

| Variable     | Description of Variable  |
|--------------|--|
| $d$          | measured difference between X and the reference standard, S, using one of the weighing designs given in other SOPs |
| $M_s$        | [true] mass of the reference standard  |
| $M_{t_s}$    | [true] mass of the tare weights carried with S   |
| $M_{t_x}$    | [true] mass of the tare weights carried with X   |
| $\rho_s$     | density of the reference standard, S   |
| $\rho_x$     | density of the unknown standard, X   |
| $\rho_{t_s}$ | density of the tare weights carried with S   |
| $\rho_{t_x}$ | density of the tare weights carried with X   |

3.2. If reporting the conventional mass<sup>2</sup>,  $CM_x$ , compute it using the following.

$$CM_x = \frac{M_x \left( 1 - \frac{0.0012}{\rho_x} \right)}{\left( 1 - \frac{0.0012}{8.0} \right)}$$

3.3. If reporting the apparent mass,  $AM_x$ , versus brass, compute it using the following.

$$AM_{x \text{ vs brass}} = \frac{M_x \left( 1 - \frac{0.0012}{\rho_x} \right)}{\left( 1 - \frac{0.0012}{8.3909} \right)}$$

3.4. The conventional and apparent mass values are related by the following:

$$CM_x = \frac{AM_{x \text{ vs brass}} \left( 1 - \frac{0.0012}{8.3909} \right)}{\left( 1 - \frac{0.0012}{8.0} \right)}$$

<sup>2</sup> Conventional Mass: "The conventional value of the result of weighing a body in air is equal to the mass of a standard, of conventionally chosen density, at a conventionally chosen temperature, which balances this body at this reference temperature in air of conventionally chosen density." The conventions are: reference density 8.0 g/cm<sup>3</sup>; reference temperature 20 °C; *normal* air density 0.0012 g/cm<sup>3</sup>. Conventional mass was formerly called "Apparent Mass versus 8.0 g/cm<sup>3</sup>" in the United States. See *OIML IR 33 (1973, 1979), under revision*.

4. Assignment of Uncertainty

The uncertainty in determining the air buoyancy correction is usually negligible relative to the precision of the measurement process itself. Consequently, the uncertainty for the measurement is based upon the uncertainty for the measurement process used. The uncertainty in the air density equation as given in numerous periodicals is 0.0012 mg/cm<sup>3</sup> (or about 0.1 % of normal air density.)

**Table 3. Tolerances for measurements related to air density estimation**

| Variable              | Uncertainty of air density values in % of air density |                        |                           |
|-----------------------|---|------------------------|---------------------------|
|                       | ± 0.1 % of air density                                | ± 1.0 % of air density | Recommended (Section 2.2) |
| Air pressure (Pa)     | ± 101   | ± 1010                 | ± 66.5                    |
| Air pressure (mm Hg)  | ± 0.76  | ± 7.6                  | ± 0.5                     |
| Air temperature ( °C) | ± 0.29  | ± 2.9                  | ± 0.1                     |
| Relative Humidity (%) | ± 11.3  | .....                  | ± 10                      |

## Appendix A

### Based on “The Basic Theory of Air Buoyancy Corrections”

by Richard S. Davis<sup>3</sup>

#### 1. Introduction

In performing measurements of mass, the balance or scale used acts as a force transducer. The force produced by an object to be weighed in air has two components: one proportional to the mass of the object, and the other proportional to its volume. The latter component, or buoyant force, may under some circumstances be large enough to require correction. The following shows under what circumstances buoyancy corrections are required as well as how they are made.

#### 2. Scope

The method for applying buoyancy corrections presented below applies to mass measurements made in air. The density of air is computed from auxiliary measurements of temperature, pressure and relative humidity after which the buoyancy corrections are calculated directly from the Principle of Archimedes. The following weighing situations are considered.

##### 2.1. Two-Pan Balance

##### 2.2. Single-Pan Balance

###### 2.2.1. With Built-In Weights

###### 2.2.2. With Electronic Control

#### 3. Summary of Method

In general, buoyancy corrections are applied to mass measurements by calculating the difference in volume between the unknown weight and the standard, multiplying this volume difference by the density of air at the balance or scale, and adding the product to the mass of the standard. The density of air is computed from an equation of state using measured values for the temperature, pressure and relative humidity of the air.

#### 4. Significance and Use

---

<sup>3</sup>Richard S. Davis, formerly of the National Institute of Standards and Technology, Mass Group.

Buoyancy corrections generally must be applied when determining the mass of an unknown object to high accuracy. The corrections may become important even at modest accuracies if the unknown object whose mass is to be determined has a density that differs widely from that of the standards (weighing of water, for example). Many mass standards are calibrated in terms of a so-called "apparent mass" [conventional mass] scale (See Chapter 7.3, Handbook 145). Use of this scale does not indiscriminately eliminate the need for buoyancy corrections as is sometimes assumed.

## 5. Terminology

### 5.1. Weighing by Substitution

Substitution weighing is the procedure by which one determines the assembly of standard weights that will produce nearly the same reading on a one-pan balance as does the unknown object. The balance thus serves as a comparator. A two-pan balance may be used in this mode if one of the pans contains a counterpoise and the standards and unknown are substituted on the second pan. (See SOP No. 3.)

### 5.2. Mass

The term "mass" is always used in the strict Newtonian sense as a property intrinsic to matter. Mass is the proportionality constant between a force on a material object and its resulting acceleration. This property is sometimes referred to as "true mass", "vacuum mass", or "mass in vacuum" to distinguish it from conventional [apparent] mass.

### 5.3. Conventional [Apparent] Mass<sup>4</sup>

The mass of material of a specified density that would exactly balance the unknown object if the weighing were carried out at a temperature of 20 °C in air of density 0.0012 g/cm<sup>3</sup>. The mass,  $M_N$ , of an object,  $N$ , is related to its apparent mass  $M_{N,A}$  by the equation:

$$M_{N,A} = \frac{M_N \left( 1 - \frac{0.0012}{\rho_N} \right)}{\left( 1 - \frac{0.0012}{\rho_B} \right)}$$

---

<sup>4</sup>Pontius, P. E., Mass and Mass Values, NBS Monograph 122, 1974, pp 12-23, 26-33 described the concept of apparent mass. The term conventional mass is described by OIML R 33, the Conventional Value of Mass in Air.

**Table A-1. Variables for conventional (apparent) mass equation**

| Variable | Description   |
|----------|---|
| $\rho_N$ | density of the object <i>N</i> at 20 °C in g/cm <sup>3</sup>                    |
| $\rho_B$ | density of the conventional (apparent) mass scale at 20 °C in g/cm <sup>3</sup> |

There are at present two apparent mass scales in wide use. The older is based on  $\rho_B = 8.4000 \text{ g/cm}^3$  at 0 °C with a coefficient of volumetric expansion of 0.000054/ °C and the more recent (Conventional Mass) specifies  $\rho_B = 8.0000 \text{ g/cm}^3$  at 20 °C. The quantity  $M_{N,A}$  is a function of the particular conventional or apparent mass scale, which has been used in its calculation. OIML IR 33 only recognizes Conventional Mass.

5.4. Sensitivity

The response of a balance under load to an additional small weight:

$$sensitivity = \frac{M_{sw} - \rho_a V_{sw}}{\Delta R} = \frac{M_{sw} \left( 1 - \frac{\rho_a}{\rho_{sw}} \right)}{\Delta R}$$

**Table A-2. Variables for sensitivity equation**

| Variable           | Description   |
|--------------------|---|
| <i>sensitivity</i> | balance sensitivity (mass per division)   |
| $M_{sw}$           | mass of the small, additional weight  |
| $\rho_a$           | density of the air  |
| $\rho_{sw}$        | density of the small, additional weight   |
| $V_{sw}$           | volume of the small, additional weight  |
| $\Delta R$         | change in balance reading due to the addition of the small weight, balance deflection |

6. Apparatus

In order to ascertain the density of air at the balance, the following measuring instruments are necessary: thermometer, barometer, and hygrometer. Ideally, these instruments should be placed in or next to the balance case (as near the measurement location as is practical). It may only be practical for the thermometer or temperature sensor to actually be placed inside the balance chamber. A calculator or computer will be extremely useful for this procedure.

## 7. Procedure

Weigh the unknown object as directed by the balance manufacturer or in accordance with accepted procedure. Record the temperature, pressure and relative humidity of the air in the balance at the time of weighing. Do not correct the barometric pressure to sea level.

## 8. Calculation

### 8.1 Air density, Option A (Option B is preferred)

The density of air, in g/cm<sup>3</sup>, can be *approximated for lesser accuracy* from the following formula:<sup>5</sup>

$$\rho_a = \left\{ \frac{0.46460 [P - (0.0037960 U e_s)]}{(273.15 + t)} \right\} \times 10^{-3} \quad (1)$$

**Table A-3. Variables for air density equation**

| Variable | Description   |
|----------|---|
| $\rho_a$ | density of air, g/cm <sup>3</sup>                       |
| $P$      | barometric pressure, mm Hg                              |
| $U$      | % relative humidity, entered as a whole number          |
| $t$      | temperature, °C   |
| $e_s$    | $1.3146 \times 10^9 \times e^{[-5315.56/(t + 273.15)]}$ |

Note:  $e_s$  can be written as follows in a spreadsheet and in some calculators:  
[1.3146E9\\*@EXP\(-5315.56/\(t+273.15\)\)](#)

Small errors (of order 0.01 %) in this equation occur for locations well above sea level or under conditions in which the concentration of carbon dioxide differs greatly from the global average. See the references for a more general formulation of the equation.

<sup>5</sup>Jones, F.E., "The Air Density Equation and the Transfer of the Mass Unit," Journal of Research, National Institute of Standards and Technology, Vol. 83, 1978, p. 419.

8.2. Air density, Option B – Preferred

The density of air should be calculated with the following formula.<sup>6</sup>

$$\rho = \frac{pM_a}{ZRT} (1 - 0.3780 x_v)$$

$$x_v = (h / 100) f \frac{p_{sv}}{p}$$

$$Z = 1 - \frac{p}{T} [a_0 + a_1t + a_2t^2 + (b_0 + b_1t)x_v + (c_0 + c_1t)x_v^2] + \frac{p^2}{T^2} (d + ex_v^2)$$

**Table A-4. Variables for CIPM air density equation**

| Variable | Description   |
|----------|---|
| $M_a$    | molar mass of the air within laboratory 28.963 5 x 10 <sup>-3</sup> kg/mol        |
| $p$      | ambient barometric pressure in Pascal   |
| $T$      | ambient temperature in Kelvin   |
| $R$      | universal gas constant: 8.314 510 J mol <sup>-1</sup> K <sup>-1</sup>             |
| $h$      | relative humidity in %  |
| $f$      | 1.000 62 + (3.14 x 10 <sup>-8</sup> ) p + (5.6 x 10 <sup>-7</sup> )t <sup>2</sup> |
| $t$      | ambient temperature in degrees Celsius  |
| $p_{sv}$ | 1 Pascal x exp (AT <sup>2</sup> + BT + C + D/T)                                   |
| $A$      | 1.237 884 7 x 10 <sup>-5</sup> K <sup>-2</sup>                                    |
| $B$      | -1.912 131 6 x 10 <sup>-2</sup> K <sup>-1</sup>                                   |
| $C$      | 33.937 110 47   |
| $D$      | -6.343 164 5 x 10 <sup>3</sup> K  |
| $a_0$    | 1.581 23 x 10 <sup>-6</sup> K Pa <sup>-1</sup>                                    |
| $a_1$    | -2.933 1 x 10 <sup>-8</sup> Pa <sup>-1</sup>                                      |
| $a_2$    | 1.104 3 x 10 <sup>-10</sup> K <sup>-1</sup> Pa <sup>-1</sup>                      |
| $b_0$    | 5.707 x 10 <sup>-6</sup> K Pa <sup>-1</sup>                                       |
| $b_1$    | -2.051 x 10 <sup>-8</sup> Pa <sup>-1</sup>  |
| $c_0$    | 1.989 8 x 10 <sup>-4</sup> K Pa <sup>-1</sup>                                     |
| $c_1$    | -2.376 x 10 <sup>-6</sup> Pa <sup>-1</sup>  |
| $d$      | 1.83 x 10 <sup>-11</sup> K <sup>2</sup> Pa <sup>-2</sup>                          |
| $e$      | -0.765 x 10 <sup>-8</sup> K <sup>2</sup> Pa <sup>-2</sup>                         |

Calculate the density of air at the balance during the weighing. Then determine the mass of the unknown,  $M_x$ , as follows:

<sup>6</sup> CIPM References: Giacomo, P. Metrologia 18: 33-40 (1982), Davis, R.S., Metrologia 29: 67-70 (1992).

8.3 If a two-pan balance is used:

$$M_x = M_s + \rho_a (V_x - V_s) + \text{sensitivity } \Delta M_{opt} \quad (3a)$$

or

$$M_x = \frac{M_s \left( 1 - \frac{\rho_a}{\rho_s} \right) + \text{sensitivity } \Delta M_{opt}}{\left( 1 - \frac{\rho_a}{\rho_x} \right)} \quad (3b)$$

or

$$M_x = \frac{M_s - \rho_a V_s + \text{sensitivity } \Delta M_{opt}}{\left( 1 - \frac{\rho_a}{\rho_x} \right)} \quad (3c)$$

**Table A-5. Variables not previously defined**

| Variable         | Description  |
|------------------|--|
| $M_x$            | mass of the unknown object                         |
| $M_s$            | mass of the standard weights                       |
| $\rho_s$         | density of the standard weights, $\frac{M_s}{V_s}$ |
| $\Delta M_{opt}$ | off-balance indication read on the optical scale   |
| $V_x$            | volume of the unknown object                       |
| $V_s$            | volume of the standard weights                     |
| $\rho_x$         | density of the unknown object, $\frac{M_x}{V_x}$   |

Volumes and densities are, in general, a function of temperature. The thermal coefficients of volumetric expansion of the unknown object and the standard may be significant in very accurate work.<sup>7</sup> The coefficient of volumetric expansion is usually estimated as three times the linear coefficient of expansion of the weight material.

<sup>7</sup>In general,  $V(t) = V_{20} [1 + \beta(t - 20 \text{ }^\circ\text{C})]$  where  $t$  is the temperature of the weight,  $V_{20}$  is the volume at 20 °C, and  $\beta$  is the coefficient of volumetric expansion.

The error in  $M_x$  incurred by ignoring the buoyancy correction is  $\rho_a (V_x - V_s)$ . To estimate quickly whether such an error is of consequence in a particular measurement, (assume  $\rho_a = 1.2 \times 10^{-3} \text{g/cm}^3$ ).

If the mass and volumes of the standards have been adjusted to a conventional mass scale, then

$$M_x \approx \frac{CM_s \left( 1 - \frac{\rho_a}{\rho_B} \right) + \text{sensitivity } \Delta M_{opt}}{\left( 1 - \frac{\rho_a}{\rho_x} \right)} \quad (4)$$

**Table A-6. Variables not previously defined**

| Variable  | Description                       |
|---|-----------------------------------|
| $CM_s$  | conventional mass of the standard |
| The symbol $\approx$ signifies an approximation |                                   |

- 8.4 If a single-pan balance with built-in weights is used, it is probable that the built-in weights have been adjusted on an apparent mass or conventional mass scale<sup>8</sup>. Determine which apparent mass scale has been used and calculate the mass of the unknown from the equation

$$M_x \approx \frac{M_D \left( 1 - \frac{\rho_a}{\rho_B} \right) + \text{sensitivity } M_{opt}}{\left( 1 - \frac{\rho_a}{\rho_x} \right)} \quad (5)$$

**Table A-7. Variables not previously defined**

| Variable  | Description                                      |
|-----------|--|
| $M_D$     | mass indicated by dial or digital readings       |
| $M_{opt}$ | mass indicated on the optical scale when present |

If the balance has been used only as a comparator, that is, to compare the mass of the unknown object with that of some external standard, then:

<sup>8</sup>Schoonover, R. M. and Jones, F. E., "Air Buoyancy in High-Accuracy Weighing on Analytical Balances," *Anal. Chem.*, 53, 1981, p. 900.

$$M_x = M_s + \rho_a (V_x - V_s) + \text{sensitivity } \Delta M'_{opt}$$

**Table A-8. Variable not previously defined**

| Variable          | Description  |
|-------------------|--|
| $\Delta M'_{opt}$ | difference in optical scale reading between observations of the standard and the unknown |

For some balances, operation requires that the user restore the balance to null by means of a manually controlled dial. The portion of the mass reading controlled by this dial should be treated, for purposes of buoyancy corrections, as an optical scale.

- 8.5 If a single-pan balance with full-range electronic control is used, the following should be noted. As part of its calibration, the electronic gain has been adjusted by means of a weight of known mass. For example, if the range of electronic control is 100 g, the electronics have been adjusted so that a 100-g standard weight produces an indication of precisely 100 g. This procedure effectively builds an apparent mass calibration into the balance. The reference density of the apparent mass scale is the density of the standard mass used for the calibration and the reference air density is the air density at the time of calibration.

**The mass of an unknown object weighed on the balance is then**

$$M_x = \frac{M_R \left( 1 - \frac{\rho_a}{\rho_c} \right)}{\left( 1 - \frac{\rho_a}{\rho_x} \right)} \quad (6)$$

**Table A-9. Variables not previously defined**

| Variable | Description   |
|----------|---|
| $M_R$    | readout displayed on the balance  |
| $\rho_a$ | density of air at the time of balance calibration   |
| $\rho_c$ | density of the standard used to calibrate the balance (or $\rho_B$ if the apparent [conventional] mass of the standard was used instead of the true mass) |

If the balance includes both an electronic control system and built-in weights, the buoyancy considerations for the built-in weights are as described in section 8.2 and the considerations for the electronically determined mass are those given directly above.

8.6 Top-loading balances may be considered a form of single-pan balance and the appropriate procedure for buoyancy correction followed.

9. Precision

The contribution of the random error of the evaluation of air density to the precision of mass measurement may be estimated as follows:

For mechanical balances, or electronic balances used in weighing by substitution, the contribution is:

$$\delta\rho_a (V_x - V_s)$$

**Table A-10. Variables for above equation**

| Variable       | Description  |
|----------------|--|
| $\delta\rho_a$ | random error of evaluation of $\rho_a$   |
| $V_s$          | volume of standards, if weighing by substitution<br>$V_s = M_D / D_B$ , if using the built-in weights on a single pan balance. |
| $V_x$          | volume of object weighed   |

The quantity,  $\delta\rho_a$  will have contributions from the measurements of temperature, pressure and relative humidity which are required for the calculation of  $\rho_a$ . Equation (1) may be used to estimate the effects of imprecision in measurements of  $P$ ,  $t$ , and  $U$ . It is unrealistic to expect  $\delta\rho_a / \rho_a$  ever to be less than 0.05 % even using the best techniques available.

10. Accuracy

Inattention to problems of buoyancy most often results in systematic errors. For a substitution weighing, for example, the buoyancy correction is of the order of  $\rho_a(V_x - V_s)$ . While this quantity may be significant to a measurement, day-to-day variation in  $\rho_a$  (usually no more than 3 %) may not be noticeable and hence need not be measured. For the most accurate work, not only must  $\rho_a$  be accurately determined, but the volumes of the unknown and standard may have to be measured to better than 0.05 % - the minimum systematic uncertainty attainable in the calculation of  $\rho_a$ .

If the standards have been calibrated in terms of conventional mass, complete neglect of buoyancy corrections will produce an error in the measured result  $M_x$  of order:

$$CM_s \left[ \left( 1.2 \times 10^{-3} - \rho_a \right) \left( \frac{1}{\rho_s} - \frac{1}{\rho_B} \right) + \left( \frac{\rho_B - \rho_x}{\rho_B} \right) \left( \frac{\rho_a}{\rho_x} \right) \right] \quad (7)$$

This error is often unacceptably large.

Use of equation (4), on the other hand, introduces only an error of approximately

$$CM_s (1.2 \times 10^{-3} - \rho_a) \left( \frac{1}{\rho_s} - \frac{1}{\rho_B} \right) \quad (8)$$

It is a requirement for manufacture that the actual density of standard weights be near enough to the assumed density of the apparent mass scale to which they are adjusted that the magnitude of (8) will always be small under normal conditions in laboratories near sea level.

The fact that there are two apparent mass scales widely used - one based on density  $8.0 \text{ g/cm}^3$  and an older one based on  $8.4 \text{ g/cm}^3$  - means that some caution is required on the part of the user. Conventional mass is generally preferred and reported for all calibrations where mass standards will be used to calibrate weighing instruments. For the most accurate work, the apparent mass scale should be abandoned in favor of substitution weighing with standards of known mass and volume.

The user must decide the accuracy required of the particular mass measurement and choose a buoyancy correction technique commensurate with that accuracy.

The same considerations, which apply to the accuracy of buoyancy corrections in weighing by substitution, are easily extended to the other types of weighing indicated above.

There are many factors, which affect the accuracy of a mass measurement. The above has dealt only with those arising from problems of buoyancy.

## Appendix B

### Examples

#### Example 1:

The weight set of Table B-1 is used with an equal-arm balance to find the mass of a piece of single-crystal silicon. The following weights were used to balance the silicon: 10 g, 3 g. The balance pointer showed the silicon side to be light by 3.5 divisions. The 10 mg weight of Table 1 was used to find the sensitivity of the balance. When the weight was added, the pointer moved 10.3 divisions. At the time of the weighing, the following measurements were taken:

$$P = 748.1 \text{ mm Hg}$$

$$t = 22.3 \text{ }^\circ\text{C}$$

$$U = 37 \% \text{ relative humidity}$$

What is the mass of the silicon?

#### Answer:

From Eq. 1, or Table B-2, calculate

$$e_s = 1.3146 \times 10^9 \times e^{\left(\frac{-5315.56}{22.3 + 273.15}\right)} = 20.194 \text{ mm Hg}$$

$$\rho_a = \left\{ \frac{0.46460 [748.1 - (0.0037960)(37)(20.2)]}{(273.15 + 22.3)} \right\} 10^{-3}$$

$$\rho_a = 1.17194 \times 10^{-3} \text{ g/cm}^3$$

The density of silicon at 20 °C is 2.329 1 g/cm<sup>3</sup> and its coefficient of linear expansion is 0.000 002 6 /°C.

Make use of Eq. 3c and Table B-1.

$$M_s = 10.0001264 + 3.0000459 = 13.000172 \text{ g}$$

$$V_s = (1.267 + 0.380) = 1.647 \text{ cm}^3$$

Calculate the sensitivity:

$$\text{sensitivity} = \frac{0.010\,003\text{ g} - (0.003\,70)(1.172 \times 10^{-3})}{10.3}$$

$$\text{sensitivity} = \frac{(0.009\,999)}{10.3} = 0.000\,971\text{ g/division} = 0.971\text{ mg/division}$$

$$M_x = \frac{[13.000\,172 - (1.647)(1.172 \times 10^{-3})] + [(0.971 \times 10^{-3})(-3.5)]}{\left(1 - \frac{1.172 \times 10^{-3}}{2.3291}\right)}$$

$$M_x = 13.001\,385\text{ g}$$

Note that the thermal expansion is insignificant in this example.

**Example 2:**

Let us again consider the weighing performed in Example 1. This time, all we know about our weight set is that it has been adjusted to the 8.4 apparent mass scale at the best available commercial tolerance.

Using Eq. (4),

$$M_x = \left\{ \frac{\left[ 13.00 \left( 1 - \frac{1.172 \times 10^{-3}}{8.390\,9} \right) \right] + \left[ 0.010 \left( 1 - \frac{1.172 \times 10^{-3}}{8.390\,9} \right) \right] \left( \frac{-3.5}{10.3} \right)}{\left( 1 - \frac{1.172 \times 10^{-3}}{2.3291} \right)} \right\}$$

$$M_x = 13.001\,329\text{ g}$$

For routine weighing, it is sometimes satisfactory to assume that the temperature is 20 °C and the density of air is  $1.2 \times 10^{-3}\text{ g/cm}^3$ . Had this been done, the computed value for the silicon would be

$$M_x = \left\{ \frac{\left[ 13.00 \left( 1 - \frac{1.2 \times 10^{-3}}{8.390\,9} \right) \right] + \left[ 0.010 \left( 1 - \frac{1.2 \times 10^{-3}}{8.390\,9} \right) \right] \left( \frac{-3.5}{10.3} \right)}{\left( 1 - \frac{1.2 \times 10^{-3}}{2.3291} \right)} \right\}$$

$$M_x = 13.001\,442\text{ g}$$

which is within 100  $\mu\text{g}$  of the answer found in Example 1.

### Example 3:

Another piece of silicon is measured on a single-pan microbalance. The balance weights were adjusted by the manufacturer to the conventional mass scale. The sensitivity of the balance has been determined to be exactly 1.000. This particular laboratory is well above sea level. At the time of the weighing, the following measurements were recorded:

$$P = 612.3 \text{ mm Hg}$$

$$t = 23.4 \text{ }^\circ\text{C}$$

$$U = 23 \text{ \% relative humidity}$$

The balance reading was 15.00 g on the built-in weights and 0.000 358 g on the optical screen. What is the mass of the silicon?

### Answer:

First, calculate  $e_s$  and  $\rho_a$ :

$$e_s = 21.59 \text{ mm Hg}$$

$$\rho_a = 0.956 \times 10^{-3} \text{ g/cm}^3$$

Then, use Eq. 5:

$$M_x = \frac{(15.00 + 0.000\,358) \left( 1 - \frac{0.956 \times 10^{-3}}{8.0} \right)}{\left( 1 - \frac{0.956 \times 10^{-3}}{2.3291} \right)}$$

$$M_x = 15.004\,724 \text{ g}$$

### Example 4:

The built-in weights in Example 3 are actually stainless steel of density  $7.78 \text{ g/cm}^3$  at  $20 \text{ }^\circ\text{C}$ . What is the approximate error caused by using the apparent mass scale?

**Answer:**

Using (7), the error is approximately

$$15 \text{ g} \left( 0.2 \times 10^{-3} \text{ g/cm}^3 \right) \left( \frac{1}{7.78 \text{ g/cm}^3} - \frac{1}{8.0 \text{ g/cm}^3} \right) = 11 \mu\text{g}$$

This discrepancy, though larger than the precision of the best analytical balances, is actually well within the tolerance of Class 1 weights.

**Table B-1.**  
**calibration report data**

**Example of**

| Mass<br>(g)  | Uncertainty<br>(g) | Vol at 20 °C<br>(cm <sup>3</sup> ) | Coefficient of<br>Expansion |
|--------------|--------------------|------------------------------------|-----------------------------|
| 100.00094070 | 0.00002535         | 12.67439                           | 0.000045                    |
| 50.00046277  | 0.00001550         | 6.33719                            | 0.000045                    |
| 30.00029259  | 0.00001361         | 3.80232                            | 0.000045                    |
| 20.00015779  | 0.00001113         | 2.53487                            | 0.000045                    |
| 10.00012644  | 0.00001330         | 1.26744                            | 0.000045                    |
| 5.00004198   | 0.00000688         | 0.63372                            | 0.000045                    |
| 3.00004588   | 0.00000459         | 0.38023                            | 0.000045                    |
| 2.00000627   | 0.00000333         | 0.25349                            | 0.000045                    |
| 1.00001438   | 0.00000300         | 0.12674                            | 0.000045                    |
| 0.49995376   | 0.00000160         | 0.03012                            | 0.000020                    |
| 0.29996145   | 0.00000115         | 0.01807                            | 0.000020                    |
| 0.19994984   | 0.00000087         | 0.01205                            | 0.000020                    |
| 0.09996378   | 0.00000091         | 0.00602                            | 0.000020                    |
| 0.04998659   | 0.00000072         | 0.00301                            | 0.000020                    |
| 0.02999100   | 0.00000077         | 0.00181                            | 0.000020                    |
| 0.02000570   | 0.00000066         | 0.00741                            | 0.000069                    |
| 0.01000277   | 0.00000086         | 0.00370                            | 0.000069                    |
| 0.00499706   | 0.00000070         | 0.00185                            | 0.000069                    |
| 0.00300299   | 0.00000076         | 0.00111                            | 0.000069                    |
| 0.00200197   | 0.00000066         | 0.00074                            | 0.000069                    |
| 0.00100083   | 0.00000086         | 0.00037                            | 0.000069                    |

**Table B-2.  $e_s$  approximation in terms of temperature**

| Temperature (°C) | $e_s$ (mm Hg) |
|------------------|---------------|
| 18               | 15.48         |
| 18.5             | 15.97         |
| 19               | 16.48         |
| 19.5             | 17            |
| 20               | 17.54         |
| 20.5             | 18.09         |
| 21               | 18.65         |
| 21.5             | 19.23         |
| 22               | 19.83         |
| 22.5             | 20.44         |
| 23               | 21.07         |
| 23.5             | 21.72         |
| 24               | 22.38         |
| 24.5             | 23.07         |
| 25               | 23.77         |
| 25.5             | 24.49         |
| 26               | 25.23         |
| 26.5             | 25.99         |
| 27               | 26.77         |
| 27.5             | 27.57         |
| 28               | 28.39         |
| 28.5             | 29.23         |
| 29               | 30.09         |
| 29.5             | 30.98         |

**E.4.2 NIST Handbook 145 SOP 4****SOP No. 4****Recommended Standard Operations Procedure  
for  
Weighing by Double Substitution Using a Single-Pan Mechanical  
Balance, a Full Electronic Balance, or a Balance with Digital  
Indications and Built-In Weights**

## 1.0 Introduction

## 1.1. Purpose

The double substitution procedure is one in which a standard and an unknown weight are intercompared twice to determine the average difference between the two weights. Errors in any built-in weights or in the balance indications are eliminated by using the balance only as a comparator and by calibrating the balance indications over the range of use for the measurement with a sensitivity weight. Accordingly, the procedure is especially useful for high accuracy calibrations. The procedure does not incorporate measurement control steps to ensure the validity of the standards and the measurement process; additional precautions must be taken.

## 1.2. Prerequisites

- 1.2.1. Verify that valid calibration certificates are available for the standards used in the test.
- 1.2.2. Verify that the standards to be used have sufficiently small standard uncertainties for the intended level of calibration. Primary standards should not be used at this level.
- 1.2.3. Verify that the balance is in good operating condition with sufficiently small process standard deviation as verified by a valid control chart or preliminary experiments to ascertain the performance quality when a new balance is put into service.
- 1.2.4. Verify that the operator is experienced in precision weighing techniques and has had specific training in SOP 2, SOP 4, SOP 29, GMP 4, and GMP 10.
- 1.2.5. Verify that the laboratory facilities comply with the following minimum conditions to meet the expected uncertainty possible with this procedure.

**Table 1. Environmental conditions**

| Echelon | Temperature   | Relative Humidity (percent) |
|---------|---|-----------------------------|
| II      | <b>20 °C to 23 °C, a set point <math>\pm</math> 2 °C, maximum change 1.0 °C/h</b> | 40 to 60 $\pm$ 10 / 4 h     |
| III     | <b>18 °C to 27 °C, maximum change 2.0 °C/h</b>                                    | 40 to 60 $\pm$ 20 / 4 h     |

## 2. Methodology

### 2.1. Scope, Precision, Accuracy

This method is applicable to all weighings utilizing a mass comparator, a single-pan mechanical balance, a full electronic balance, or a balance that combines digital indications with the use of built-in weights (combination balance). The precision will depend upon the sensitivity of the balance and the care exercised to make the required weighings. The accuracy achievable with this procedure depends on the accuracy of the calibration of the working standards and the precision of the intercomparison.

### 2.2. Summary

The balance is adjusted if necessary, to obtain balance indications for all measurements that will be within the range of the optical scale or digital indications of the balance without changing the dial settings for the built-in weights, if present. The standard and the test weight are each weighed. A small, calibrated weight, called a sensitivity weight, is added to the test weight and these are weighed. The standard and the same sensitivity weight are then weighed. The latter two weighings provide both second weighings of the standard and the test weight as well as a determination of the sensitivity of the balance under the load conditions at the time of the intercomparison. All weighings are made at regularly spaced time intervals to average out any effects due to instrument drift.

The double substitution procedure is the same for all of the balances mentioned above, but the adjustment of the balance to prepare for the intercomparison and the selection of the sensitivity weight differs slightly depending upon the balance used. When steps specific to a particular balance are required, they are given in subsections of the procedure identified by a, b, and c along with the balance type.

### 2.3. Apparatus/Equipment Required

2.3.1. Precision balance with sufficient capacity and sensitivity for the calibrations planned.

2.3.2. Calibrated working standard and sensitivity weights with recent calibration values that are traceable to NIST.

- 2.3.3. Calibrated small standard weights with recent calibration certificates and values traceable to NIST to be used as tare weights.
- 2.3.4. Uncalibrated weights to be used to adjust the balance to the desired reading range.
- 2.3.5. Forceps to handle the weights, or gloves to be worn if the weights are moved by hand.
- 2.3.6. Stop watch or other timing device to observe the time of each measurement.
- 2.3.7. Calibrated barometer accurate to  $\pm 66.5$  Pa (0.5 mm Hg) with recent calibration values traceable to NIST to determine air pressure.
- 2.3.8. Calibrated thermometer accurate to  $\pm 0.10$  °C with recent calibration values traceable to NIST to determine air temperature.
- 2.3.9. Calibrated hygrometer accurate to  $\pm 10$  percent with recent calibration values traceable to NIST to determine relative humidity.<sup>9</sup>

#### 2.4. Symbols

**Table 2. Symbols used in this procedure**

| Symbol   | Description   |
|----------|---|
| $S$      | standard reference weight   |
| $X$      | weight to be calibrated   |
| $t$      | small calibrated tare weight, A subscript $s$ or $x$ is used to indicate the larger weight with which it is associated                        |
| $sw$     | small calibrated weight used to evaluate the sensitivity of the balance   |
| $M$      | the mass (true mass) of a specific weight. Subscripts $s$ , $x$ , $t$ , $sw$ are used to identify the weight (equals Nominal plus Correction) |
| $N$      | the nominal value of a specific weight. Subscripts $s$ , $x$ , are used to identify the weight.   |
| $C$      | the correction for a specific weight. Subscripts $s$ , $x$ , are used to identify the weight.   |
| $CM$     | the conventional mass of a specific weight. Subscripts $s$ , $x$ , $t$ , $sw$ are used to identify the weight.                                |
| $\rho_a$ | density of air at time of calibration   |

<sup>9</sup>The barometer, thermometer, and hygrometer are used to determine the air density at the time of the measurement. The air density is used to make an air buoyancy correction. The accuracies specified are recommended for high precision calibration. Less accurate equipment can be used with only a small degradation in the overall accuracy of the measurement.

| Symbol   | Description   |
|----------|---|
| $\rho_n$ | density of normal air (1.2 kg/m <sup>3</sup> )                                      |
| $\rho$   | density of masses; subscripts $s, x, t_s, t_x, sw$ are used to identify the weight. |

## 2.5. Procedure

### 2.5.1. Preliminary Procedure

- 2.5.1.1. Place the test weight and standards in the balance chamber or near the balance overnight to permit the weights and the balance to attain thermal equilibrium.
- 2.5.1.2. Conduct preliminary measurements to obtain an approximate value for the difference between the standard and the unknown, to determine where the readings occur on the balance, to determine if tare weights are required, to determine the sensitivity weight that must be used, and to determine the time interval required for the balance indication to stabilize.

Tare weights are rarely needed for high precision mass standards. If tare weights are required, carry tare weights,  $t_s$  and  $t_x$ , with the standard and the unknown,  $S$  and  $X$ , respectively. The tare weights must be calibrated standards with valid uncertainties that are evaluated in the process of determining calibration uncertainties. The standard and its tare weight,  $S + t_s$ , should be "nearly the same mass" as the unknown with its tare weight,  $X + t_x$ . "Nearly the same mass" depends upon the balance used (See GMP 14, Table 1). Select  $t_s$  and  $t_x$  such that the difference in mass between  $S + t_s$  and  $X + t_x$  is:

- a. Single-pan mechanical balance - less than  $\frac{1}{10}$  the range of the optical scale.
- b. Full electronic balance - less than 0.05 percent of the balance capacity.
- c. Combination balance - less than  $\frac{1}{10}$  the range of the digital indications.
- d. Mass comparator – less than  $\frac{1}{10}$  digital range

A sensitivity weight must be used on equal-arm balances, and is normally used on single-pan mechanical and electronic balances, to ensure that the measured differences determined through the

use of the optical scale or electronic range have valid accuracy and traceability (See GMP 14, Table 2). (e.g., The optical scale is *calibrated* each time the procedure is performed through the use of a sensitivity weight). The uncertainty of the sensitivity weight does not generally need to be included in calculations of uncertainty since the uncertainty value is distributed across its range of use.

If a sensitivity weight will be used, select one that is:

- a. Single-pan balance - between  $\frac{1}{4}$  and  $\frac{1}{2}$  the range of the optical scale, and at least 4 times the mass difference between  $X$  and  $S$ .
- b. Full electronic balance - at least 4 times the mass difference between  $X$  and  $S$ , but not exceeding 1 percent of the balance capacity.
- c. Combination balance - between  $\frac{1}{4}$  and  $\frac{1}{2}$  the range of the digital indications, and at least 4 times the mass difference between  $X$  and  $S$ .
- d. Mass comparator – at least 4 times the mass difference between  $X$  and  $S$ , but not exceeding  $\frac{1}{2}$  of the digital range.

A sensitivity weight is not required if the electronic mass comparator that is used has been tested (with supporting data available) to determine that the balance has sufficient accuracy, resolution, repeatability, and stability so that no advantage is gained through the use of a sensitivity weight. For example, any possible errors must be less than what contributes to the uncertainty. When a mass comparator is used without a sensitivity weight, the sensitivity must be periodically verified and documented.

- 2.5.1.3. Determine which optional sequence will be used, A or B. Optional sequence A uses the standard on the balance for the first and fourth observations and the unknown on the balance for the second and third observations; this is often called the “SXXS” sequence. Optional sequence B starts with the unknown on the balance first and last with the standard on the balance for the second and third observations; this is often called the “XSSX” sequence. The primary advantage of sequence B is less handling of the mass standards. The advantage of sequence A is in the case where the unknown is a summation of weights that require careful arrangement on the balance pan only once.
- 2.5.1.4. Adjust the single pan balance or the combination balance so the first two readings of the double substitution fall in the first quarter of the optical scale or digital indications. The zero adjustment and tare adjustment may be used. Small weights may be placed on the balance pan to reach the desired reading range. These weights remain on the pan throughout the double substitution. Once the balance has been adjusted to the desired

position, neither the balance dials, the zero and tare adjustments, nor the small weights placed on the balance pan, are to be changed during the measurement.

- 2.5.1.5. If the balance is equipped with a pan arrestment mechanism, arrest the pan between each observation.

2.5.2. Measurement Procedure, Optional Sequence A (SXXS)

**Table 3. Optional Sequence A**

| Measurement No. | Weights on Pan | Observation |
|-----------------|----------------|-------------|
| 1               | $S + t_s$      | $O_1$       |
| 2               | $X + t_x$      | $O_2$       |
| 3               | $X + t_x + sw$ | $O_3$       |
| 4               | $S + t_s + sw$ | $O_4$       |

All observations should be recorded on suitable data sheets, such as those in the appendix. Record the laboratory ambient temperature, barometric pressure, and relative humidity.

- 2.5.2.1. Observation 1. Place the standard weight(s),  $S$ , along with  $t_s$  on the balance pan. If equipped with a pan arrestment mechanism, release the balance pan. When the pan is released, start the stop-watch and record observation  $O_1$  once the balance indication has stabilized.**
- 2.5.2.2. Observation 2. Remove weight(s)  $S$  and  $t_s$  and replace with test weight  $X$  and its tare weight,  $t_x$ . Release the pan, time the interval, and record observation  $O_2$ .**
- 2.5.2.3. Observation 3. Add the sensitivity weight,  $sw$ , to the weights of observation 2. Release the pan, time the interval, and record observation  $O_3$ .**
- 2.5.2.4. Observation 4. Remove weights  $X$  and  $t_x$  and replace with  $S$  and  $t_s$ . The sensitivity weight,  $sw$ , remains on the balance pan. Release the pan, time the interval, and record observation  $O_4$ .**
- 2.5.2.5. Compare the two differences ( $O_2 - O_1$ ) and ( $O_3 - O_4$ ); they should not differ from one another by more than 2 standard deviations of the balance for this process and load. If this difference is exceeded, reject the data and redo the**

measurements. Investigate possible causes of excess variability if measurements do not agree within these limits.

**2.5.2.6. If repeated double substitutions are performed, the values between successive trials should not differ from one another by more than  $\pm 2$  standard deviations of the balance for this process and load. If this difference is exceeded, reject the data and take a new series of measurements that do so agree.**

### 2.5.3. Measurement Procedure, Optional Sequence B (XSSX)

**Table 4. Optional Sequence B**

| Measurement No. | Weights on Pan | Observation |
|-----------------|----------------|-------------|
| 1               | $X + t_x$      | $O_1$       |
| 2               | $S + t_s$      | $O_2$       |
| 3               | $S + t_s + sw$ | $O_3$       |
| 4               | $X + t_x + sw$ | $O_4$       |

Measurements for Option B are made as described in Option A except that  $X$ ,  $S$ ,  $t_x$ , and  $t_s$  are interchanged.

## 3. Calculations

3.1. No air buoyancy correction. Calculate the conventional mass correction,  $C_x$ , for the test weight as follows, according to the optional sequence used. In each case, the conventional mass corrections for the standard weight(s),  $C_s$ , the conventional mass of the tare weights,  $CM_{t_s}$  and  $CM_{t_x}$ , and the conventional mass of the sensitivity weight,  $CM_{sw}$ , are included. The symbols  $N_s$  and  $N_x$  refer to the nominal values of  $S$  and  $X$ , respectively. If no tare weights, and equal nominal values are used, those terms may all be deleted from the equations.

### 3.1.1. Optional Sequence A (SXXS)

$$C_x = C_s + CM_{t_s} - CM_{t_x} + \left[ \frac{(O_2 - O_1) + (O_3 - O_4)}{2} \right] \left[ \frac{CM_{sw}}{O_3 - O_2} \right] + N_s - N_x$$

## 3.1.2. Optional Sequence B (XSSX)

$$C_x = C_s + CM_{t_s} - CM_{t_x} + \left[ \frac{(O_1 - O_2) + (O_4 - O_3)}{2} \right] \left[ \frac{CM_{sw}}{O_3 - O_2} \right] + N_s - N_x$$

## 3.2. Air Buoyancy Correction

3.2.1. Calculate the air density,  $\rho_a$ , as described in the Appendix to SOP No. 2.3.2.2. Calculate the mass  $M_x$  of the test weight, and its mass correction  $C_x$  using the mass of the standard weight(s), the tare weights and the sensitivity weights according to the optional sequence used.

## 3.2.2.1. Optional Sequence A (SXXS)

$$M_x = \frac{M_s \left( 1 - \frac{\rho_a}{\rho_s} \right) + M_{t_s} \left( 1 - \frac{\rho_a}{\rho_{t_s}} \right) - M_{t_x} \left( 1 - \frac{\rho_a}{\rho_{t_x}} \right) + \left[ \frac{(O_2 - Q) + (O_3 - Q)}{2} \right] \left[ \frac{M_{sw} \left( 1 - \frac{\rho_a}{\rho_{sw}} \right)}{O_3 - Q} \right]}{\left( 1 - \frac{\rho_a}{\rho_x} \right)}$$

## 3.2.2.2. Optional Sequence B (XSSX)

$$M_x = \frac{M_s \left( 1 - \frac{\rho_a}{\rho_s} \right) + M_{t_s} \left( 1 - \frac{\rho_a}{\rho_{t_s}} \right) - M_{t_x} \left( 1 - \frac{\rho_a}{\rho_{t_x}} \right) + \left[ \frac{(O_1 - O_2) + (O_4 - O_3)}{2} \right] \left[ \frac{M_{sw} \left( 1 - \frac{\rho_a}{\rho_{sw}} \right)}{O_3 - O_2} \right]}{\left( 1 - \frac{\rho_a}{\rho_x} \right)}$$

3.2.3. Calculate the mass correction  $C_x$ , as follows:

$$C_x = M_x - N_x$$

where  $N_x$  is the nominal value for  $X$ .

- 3.2.4. Calculate the conventional mass<sup>10</sup> of  $X$ ,  $CM_x$ . It is recommended that the conventional mass be reported.

**3.2.4.1. Conventional mass**

$$CM_x = \frac{M_x \left( 1 - \frac{\rho_n}{\rho_x} \right)}{\left( 1 - \frac{\rho_n}{8.0} \right)}$$

- 3.2.5. If requested, the apparent mass versus the reference density of brass may be calculated. This value should only be used when calibrating mechanical balances that have been adjusted to this reference density.

**3.2.5.1. Apparent mass versus brass**

$$AM_{x \text{ vs brass}} = \frac{M_x \left( 1 - \frac{\rho_n}{\rho_x} \right)}{\left( 1 - \frac{\rho_n}{8.3909} \right)}$$

4. Measurement Assurance

- 4.1. Duplicate the process with a suitable check standard (See GLP 1, SOP 9, SOP 30, and Sec. 7.4)
- 4.2. Plot the check standard value and verify that it is within established limits; a t-test may be incorporated to check observed value against accepted value.
- 4.3. The mean of the check standard is used to evaluate bias and drift over time.
- 4.4. Check standard observations are used to calculate the standard deviation of the measurement process,  $s_p$ .

5. Assignment of Uncertainty

The limits of expanded uncertainty,  $U$ , include estimates of the standard uncertainty of the mass standards used,  $u_s$ , estimates of the standard deviation of the measurement process,  $s_p$ , and estimates of the effect of other components associated with this procedure,  $u_o$ . These estimates should be combined using the root-sum-squared method

<sup>10</sup> Conventional Mass: "The conventional value of the result of weighing a body in air is equal to the mass of a standard, of conventionally chosen density, at a conventionally chosen temperature, which balances this body at this reference temperature in air of conventionally chosen density." The conventions are: reference density 8.0 g/cm<sup>3</sup>; reference temperature 20 °C; *normal* air density 0.0012 g/cm<sup>3</sup>. Conventional mass was formerly called "Apparent Mass versus 8.0 g/cm<sup>3</sup>" in the United States. See OIML IR 33 (1973, 1979), *under revision*.

(RSS), and the expanded uncertainty,  $U$ , reported with a coverage factor of two ( $k=2$ ), to give us an approximate 95 percent level of confidence. See SOP 29 for the complete standard operating procedure for calculating the uncertainty.

- 5.1. The expanded uncertainty for the standard,  $U$ , is obtained from the calibration report. The combined standard uncertainty,  $u_c$ , is used and not the expanded uncertainty,  $U$ , therefore the reported uncertainty for the standard will usually need to be divided by the coverage factor  $k$ .
- 5.2. The value for  $s_p$  is obtained from the control chart data for check standards using double substitution measurements. (See SOP No. 9.)
- 5.3. Other standard uncertainties usually included at this calibration level include uncertainties associated with calculation of air density and standard uncertainties associated with the density of the standards used.
- 5.4. The expanded uncertainty,  $U$ , must be  $\leq 1/3$  of the tolerance applicable as per ASTM E617-97 and OIML R111 to classify mass standards.

## 6. Report

Report results as described in SOP No. 1, Preparation of Calibration/Test Reports.

Appendix  
Double Substitution Data Sheet  
(Optional Sequence A)  
SXXS

**Laboratory data and conditions:**

|  |  |                   |
|--|--|-------------------|
| Operator   |  |                   |
| Date   |  | Temperature       |
| Balance  |  | Pressure          |
| Nominal Load   |  | Relative Humidity |
| Standard deviation of the process, from control chart, $s_p$ |  |                   |

**Mass standard(s) data:**

| ID       | Nominal | Mass Correction* | Expanded Unc:<br>From cal. report | Unc:<br>k factor | Density<br>g/cm <sup>3</sup> |
|----------|---------|------------------|-----------------------------------|------------------|------------------------------|
| <b>S</b> |         |                  |                                   |                  |                              |
| X        |         |                  |                                   |                  |                              |
| sw       |         |                  |                                   |                  |                              |
| $t_s$    |         |                  |                                   |                  |                              |
| $t_x$    |         |                  |                                   |                  |                              |
|          |         |                  |                                   |                  |                              |
|          |         |                  |                                   |                  |                              |

\*Mass Correction = *True Mass* if using buoyancy correction. Mass Correction = *Conventional Mass* if NOT using buoyancy correction. Density is used only with buoyancy corrections.

**Observations:**

| Observation No. | Weights        | Balance Observations, Units |
|-----------------|----------------|-----------------------------|
| Time:           |                |                             |
| 1 ( $O_1$ )     | $S + t_s$      | ⋮                           |
| 2 ( $O_2$ )     | $X + t_x$      | ⋮                           |
| 3 ( $O_3$ )     | $X + t_x + sw$ | ⋮                           |
| 4 ( $O_4$ )     | $S + t_s + sw$ | ⋮                           |
| Time:           |                |                             |

**Measurement Assurance (Duplication of the Process):**

| Observation No. | Weights             | Balance Observations, Units |
|-----------------|---------------------|-----------------------------|
| Time:           |                     |                             |
| 1 ( $O_1$ )     | $S + t_s$           | ⋮                           |
| 2 ( $O_2$ )     | $S_c + t_{Sc}$      | ⋮                           |
| 3 ( $O_3$ )     | $S_c + t_{Sc} + sw$ | ⋮                           |
| 4 ( $O_4$ )     | $S + t_s + sw$      | ⋮                           |
| Time:           |                     |                             |

Note: dotted line represents decimal point

## Appendix

### Double Substitution Data Sheet (Optional Sequence B) XSSX

#### Laboratory data and conditions:

|  |  |                   |
|--|--|-------------------|
| Operator   |  |                   |
| Date   |  | Temperature       |
| Balance  |  | Pressure          |
| Load   |  | Relative Humidity |
| Process standard deviation from control chart, $s_p$ |  |                   |

#### Mass standard(s) data:

| ID       | Nominal | Mass Correction* | Expanded Unc:<br>From Cal. report | Unc:<br>k factor | Density<br>g/cm <sup>3</sup> |
|----------|---------|------------------|-----------------------------------|------------------|------------------------------|
| <b>X</b> |         |                  |                                   |                  |                              |
| S        |         |                  |                                   |                  |                              |
| sw       |         |                  |                                   |                  |                              |
| $t_x$    |         |                  |                                   |                  |                              |
| $t_s$    |         |                  |                                   |                  |                              |
|          |         |                  |                                   |                  |                              |
|          |         |                  |                                   |                  |                              |

\*Mass Correction = *True Mass* if using buoyancy correction. Mass Correction = *Conventional Mass* if NOT using buoyancy correction. Density is used only with buoyancy corrections.

#### Observations:

| Observation No. | Weights        | Balance Observations, Units |
|-----------------|----------------|-----------------------------|
| Time:           |                |                             |
| 1 ( $O_1$ )     | $X + t_x$      |                             |
| 2 ( $O_2$ )     | $S + t_s$      |                             |
| 3 ( $O_3$ )     | $S + t_s + sw$ |                             |
| 4 ( $O_4$ )     | $X + t_x + sw$ |                             |
| Time:           |                |                             |

#### Measurement Assurance (Duplication of the Process):

| Observation No. | Weights             | Balance Observations, Units |
|-----------------|---------------------|-----------------------------|
| Time:           |                     |                             |
| 1 ( $O_1$ )     | $S_c + t_{sc}$      |                             |
| 2 ( $O_2$ )     | $S + t_s$           |                             |
| 3 ( $O_3$ )     | $S + t_s + sw$      |                             |
| 4 ( $O_4$ )     | $S_c + t_{sc} + sw$ |                             |

|       |  |  |
|-------|--|--|
| Time: |  |  |
|-------|--|--|

Note: dotted line represents decimal point.

Example: With Buoyancy Corrections

Double Substitution Data Sheet

(Optional Sequence A)

SXXX

### Laboratory data and conditions:

|  |         |                   |             |
|--|---------|-------------------|-------------|
| Operator   |         |                   | HO          |
| Date   | 8/24/86 | Temperature       | 22.3 °C     |
| Balance  | M5SA    | Pressure          | 753.5 mm Hg |
| Nominal Load   | 10 g    | Relative Humidity | 45 %        |
| Standard deviation of the process, from control chart, $s_p$ |         |                   | 0.002 9 mg  |

### Mass standard(s) data:

| ID       | Nominal | Mass Correction* | Expanded Unc:<br>From Cal. Rpt.<br>(mg) | Unc:<br>k factor | Density<br>g/cm <sup>3</sup> |
|----------|---------|------------------|---|------------------|------------------------------|
| <b>S</b> | 10 g    | -0.679 mg        | 0.014 mg                                | 3                | 8.00                         |
| X        | 10 g    | TBD              | TBD                                     | 2                | 7.84                         |
| sw       | 5 mg    | -0.0227 mg       | 0.000 28                                | 2                | 8.5                          |
| $t_s$    | ---     | ---              | ---                                     | ---              | ---                          |
| $t_x$    | ---     | ---              | ---                                     | ---              | ---                          |
| $S_c$    | 10 g    | 0.321 mg         | 0.025 mg                                | 2                | 8.0                          |

\*Mass Correction = *True Mass* if using buoyancy correction. Mass Correction = *Conventional Mass* if NOT using buoyancy correction. Density is used only with buoyancy corrections.

### Observations:

| Observation No. | Weights        | Balance Observations, Units mg |
|-----------------|----------------|--------------------------------|
| Time: 8:35 AM   |                |                                |
| 1 ( $O_1$ )     | $S + t_s$      | 1 : 268                        |
| 2 ( $O_2$ )     | $X + t_x$      | 1 : 821                        |
| 3 ( $O_3$ )     | $X + t_x + sw$ | 6 : 798                        |
| 4 ( $O_4$ )     | $S + t_s + sw$ | 6 : 245                        |
| Time: 8:47 AM   |                |                                |

### Measurement Assurance (Duplication of the Process):

| Observation No. | Weights        | Balance Observations, Units |
|-----------------|----------------|-----------------------------|
| Time: 9:00 AM   |                |                             |
| 1 ( $O_1$ )     | $S + t_s$      | 1 : 270                     |
| 2 ( $O_2$ )     | $S_c + t_{Sc}$ | 2 : 271                     |

|                     |                                       |         |
|---------------------|---------------------------------------|---------|
| 3 (O <sub>3</sub> ) | S <sub>c</sub> + t <sub>sc</sub> + sw | 7 : 248 |
| 4 (O <sub>4</sub> ) | S + t <sub>s</sub> + sw               | 6 : 248 |
| Time: 9:10 AM       |                                       |         |

Note: dotted line represents decimal point

Calculate the air density (SOP 2):

$$\rho_a = 1.1795 \text{ mg/cm}^3 = 0.0011795 \text{ g/cm}^3$$

Use equation 3.2.2.1 for optional sequence A (SXXS) with buoyancy corrections<sup>11</sup>:

$$M_x = \frac{M_s \left(1 - \frac{\rho_a}{\rho_s}\right) + M_{t_s} \left(1 - \frac{\rho_a}{\rho_{t_s}}\right) - M_{t_x} \left(1 - \frac{\rho_a}{\rho_{t_x}}\right) + \left[ \frac{(O_2 - Q) + (O_3 - Q)}{2} \right] \left[ \frac{M_{sw} \left(1 - \frac{\rho_a}{\rho_{sw}}\right)}{O_3 - Q} \right]}{\left(1 - \frac{\rho_a}{\rho_x}\right)}$$

$$M_x = \frac{9.999321 \left(1 - \frac{0.0011795}{8.0}\right) + 0 - 0 + \left[ \frac{(1.821 - 1.268) + (6.798 - 6.245)}{2} \right] \left[ \frac{0.0049773 \left(1 - \frac{0.0011795}{8.5}\right)}{6.798 - 1.821} \right]}{\left(1 - \frac{0.0011795}{7.84}\right)}$$

$$M_x = \frac{(9.9978461 + 0.000552957)}{0.99984949} = 9.9999041 \text{ g}$$

Calculate the mass (*true mass*) correction:

$$C_x = M_x - N_x$$

$$C_x = 9.9999041 \text{ g} - 10 \text{ g} = -0.0000959 \text{ g} = -0.0959 \text{ mg}$$

Calculate the conventional mass value:

<sup>11</sup> Keep in mind that these equations may be truncated for the purpose of this example and minor differences may be seen in the ending decimal places due to the use of calculators or spreadsheets.

$$CM_x = \frac{M_x \left( 1 - \frac{\rho_n}{\rho_x} \right)}{\left( 1 - \frac{\rho_n}{8.0} \right)}$$

$$CM_x = \frac{9.999\,9041 \left( 1 - \frac{0.0012}{7.84} \right)}{0.999\,850}$$

$$CM_x = \frac{9.999\,9041 (0.999\,846\,9)}{0.999\,850} = 9.999\,873\,51\text{ g}$$

$$C_x = CM_x - N_x$$

$$C_x = 9.999\,873\,51\text{ g} - 10\text{ g}$$

$$C_x = -0.000\,126\,49\text{ g} = -0.126\,49\text{ mg}$$

Calculate the uncertainty for the calibration:

$$U = u_c * 2$$

$$u_c = \sqrt{u_s^2 + s_p^2 + u_o^2}$$

The uncertainty for the standard,  $U$ , must be divided by the k factor to determine the  $u_s$ .

$$u_c = \sqrt{(0.004\,667)^2 + (0.002\,9)^2 + (0.000\,000\,032)^2}$$

$$u_c = 0.005\,494\,623\,6\text{ mg}$$

$$\mathbf{U = 0.005\,494\,623\,6 * 2 = 0.010\,989\,247\,3\text{ mg}}$$

### Uncertainty Statement

The uncertainty reported is the root sum square of the standard uncertainty of the standard, the standard deviation of the process, and an uncorrected systematic error for lack of buoyancy corrections, multiplied by a coverage factor of 2 (k=2) for an approximate 95 percent confidence interval. Factors not considered in the evaluation: magnetism (weights are considered to meet magnetism specifications unless measurement aberrations are noted), balance eccentricity and linearity (these factors are considered as a part of the measurement process when obtaining the standard deviation of the process).

### Compliance Evaluation

We have to evaluate the correction with its expanded uncertainty to determine if the weight is in tolerance or not. The magnitude of the expanded uncertainty has to be less than 1/3 of the tolerance to be able to perform that evaluation, according to ASTM E617-97 and OIML R111.

Load = 10 g

| ASTM E617 |                | OIML R111      |                |
|-----------|----------------|----------------|----------------|
| Class     | Tolerance (mg) | Class          | Tolerance (mg) |
| 0         | 0.025          | E <sub>1</sub> | 0.020          |
| 1         | 0.050          | E <sub>2</sub> | 0.060          |
| 2         | 0.054          | F <sub>1</sub> | 0.20           |

If we look at three times the uncertainty:  $0.011 \text{ mg} \times 3 = 0.033 \text{ mg}$ , we realize that the uncertainty complies with the 1/3 rule for ASTM classes 1, 2, and OIML classes E<sub>2</sub>, F<sub>1</sub>.

Next, we look at the correction with the uncertainty:  $-0.126 \text{ mg} \pm 0.011 \text{ mg}$ .

We can see that the absolute value of the correction is within:  $0.115 \leq C_x \leq 0.137$ , therefore, it only complies with OIML class F<sub>1</sub>.

### Reporting

The conventional mass correction and uncertainty would be reported as follows:

$$C_X = -0.126 \text{ mg} \pm 0.011 \text{ mg}$$

**Example: Without Buoyancy Corrections**

Double Substitution Data Sheet  
(Optional Sequence B)  
XSSX

**Laboratory data and conditions:**

|  |                  |                   |             |
|--|------------------|-------------------|-------------|
| Operator   |                  | HO                |             |
| Date   | 8/24/86          | Temperature       | 22.3 °C     |
| Balance  | CB 100           | Pressure          | 753.5 mm Hg |
| Load   | 30 g & 1 troy oz | Relative Humidity | 45 %        |
| Process standard deviation from control chart, $s_p$ |                  |                   | 0.018 mg    |

**Mass standard(s) data:**

| ID       | Nominal | Mass Correction* | Expanded Unc:<br>From cal. report | Unc:<br>k factor | Density<br>g/cm <sup>3</sup> |
|----------|---------|------------------|-----------------------------------|------------------|------------------------------|
| <b>S</b> | 30 g    | 0.407 mg         | 0.022 mg                          | 3                | 8                            |
| X        | 1 t oz  | TBD              | TBD                               | 2                | 7.84                         |
| sw       | 50 mg   | -0.084 00 mg     | 0.000 65 mg                       | 2                | 8.5                          |
| $t_s$    | 1.1 g   | 0.359 6 mg       | 0.006 3 mg                        | 3                | 8.04                         |
| $t_x$    | None    | ---              | ---                               | ---              | ---                          |
| $S_c$    | 30 g    | 0.907 mg         | 0.030 mg                          | 2                | 8                            |

\*Mass Correction = *True Mass* if using buoyancy correction. Mass Correction = *Conventional Mass* if NOT using buoyancy correction. Density is used only with buoyancy corrections.

**Observations:**

| Observation No. | Weights        | Balance Observations, Units <u>mg</u> |
|-----------------|----------------|---------------------------------------|
| Time: 9:00 AM   |                |                                       |
| 1 ( $O_1$ )     | $X + t_x$      | 20 : 93                               |
| 2 ( $O_2$ )     | $S + t_s$      | 17 : 21                               |
| 3 ( $O_3$ )     | $S + t_s + sw$ | 67 : 08                               |
| 4 ( $O_4$ )     | $X + t_x + sw$ | 70 : 81                               |
| Time: 9:05 AM   |                |                                       |

**Measurement Assurance (Duplication of the Process):**

| Observation No. | Weights             | Balance Observations, Units |
|-----------------|---------------------|-----------------------------|
| Time: 9:15 AM   |                     |                             |
| 1 ( $O_1$ )     | $S + t_s$           | 20 : 95                     |
| 2 ( $O_2$ )     | $S_c + t_{Sc}$      | 21 : 45                     |
| 3 ( $O_3$ )     | $S_c + t_{Sc} + sw$ | 71 : 32                     |
| 4 ( $O_4$ )     | $S + t_s + sw$      | 70 : 83                     |
| Time: 9:20 AM   |                     |                             |

Note: dotted line represents decimal point

Use equation 3.1.2 for optional sequence B (XSSX) with NO buoyancy corrections<sup>12</sup>:

$$C_x = C_s + CM_{t_s} - CM_{t_x} + \left[ \frac{(O_1 - O_2) + (O_4 - O_3)}{2} \right] \left[ \frac{CM_{sw}}{O_3 - O_2} \right] + N_s - N_x$$

Note: be careful to combine like units only!

$$C_x = 0.407 \text{ mg} + 1.1003596 \text{ g} - 0 + \left[ \frac{(20.93 - 17.21) + (70.81 - 67.08)}{2} \right] \left[ \frac{49.916 \text{ mg}}{67.08 - 17.21} \right] + 30 \text{ g} - 1 \text{ t oz}$$

$$C_x = 0.407 \text{ mg} + 1.1003596 \text{ g} - 0 + 3.7284359 \text{ mg} + 30 \text{ g} - 31.1034768 \text{ g}$$

$$C_x = 1104.495036 \text{ mg} - 1103.4768 \text{ g}$$

$$C_x = 1.018236 \text{ mg}$$

Calculate the uncertainty for the calibration:

$$U = u_c * 2$$

$$u_c = \sqrt{u_s^2 + s_p^2 + u_o^2}$$

The uncertainty for the standard,  $U$ , must be divided by the  $k$  factor for the standard and the tare weight to determine each  $u_s$ . The additional uncertainty for not performing the air buoyancy correction can be determined using the magnitude of the air buoyancy correction from SOP 2.

$$u_c = \sqrt{(0.00733)^2 + (0.0021)^2 + (0.018)^2 + (0.0016)^2}$$

$$u_c = 0.01961715 \text{ mg}$$

$$U = 0.01961715 * 2 = 0.039234 \text{ mg}$$

The conventional mass correction and uncertainty would be reported as follows:

$$C_x = 1.018 \text{ mg} \pm 0.039 \text{ mg}$$

OR

$$C_x = 0.0000327 \text{ t oz} \pm 0.0000013 \text{ t oz}$$

<sup>12</sup> Keep in mind that these equations may be truncated for the purpose of this example and minor differences may be seen in the ending decimal places due to the use of calculators or spreadsheets.

## E.4.3 NIST Handbook 145 SOP 29

### SOP No. 29

#### Standard Operating Procedure for the Assignment of Uncertainty

#### 1. Introduction

##### 1.1. Purpose

Laboratories performing calibrations that meet ISO/IEC Guide 25, ISO/IEC 17025, or ANSI/NCSL Z 540-1-1994 must report uncertainties in conformance with the 1993 *ISO Guide to the Expression of Uncertainty in Measurement* (hereafter called the *GUM*). This SOP provides instruction for the laboratory to meet this requirement.

##### 1.2. Prerequisites

1.2.1. Calibration certificates with valid uncertainties must be available for all standards.

1.2.2. Statistical data regarding the calibration measurement process must be available; preferably from measurement control programs within the laboratory.

1.2.3. Knowledge of the technical basis for the measurement is critical for completeness in uncertainty evaluation. This can be obtained through brainstorming, experimentation, interlaboratory comparisons, cause and effect diagrams and the like. Flow charts at the end of this SOP show a number of common factors affecting measurements in the areas of mass, length, and volume.

#### 2. Methodology

##### 2.1. Scope, Precision, Accuracy

Each measurement made in a laboratory has a corresponding uncertainty assigned to the calibration value. The uncertainty is directly related to the measurement parameter (scope), range of the measurement, the equipment or measurement process being used (affecting precision), and the standards available with associated uncertainties.

## 2.2. Summary

This uncertainty analysis process follows the following steps:

- 1) the measurement process is clearly specified;
- 2) uncertainty sources are identified and characterized;
- 3) uncertainty sources are quantified;
- 4) uncertainty sources are converted to standard uncertainties;
- 5) the combined uncertainty is calculated;
- 6) the expanded uncertainty is calculated;
- 7) the expanded uncertainty is evaluated against appropriate tolerance or calibration limits; and
- 8) measurement results are reported.

Special methods for handling bias/errors are addressed as well.

## 3. The Process of Measurement Uncertainty Estimation

### 3.1. Step 1. Specify the process.

Clearly specify the measurement process in question, including the item being measured and the input quantities upon which it depends. This will usually require a quantitative expression related to the process. Where possible, you may reference an SOP or other method description along with the specific standards and measurement assurance process that is used to adequately complete this step.

### 3.2. Step 2. Identify and characterize uncertainty sources.

Identify all possible sources of uncertainty in a comprehensive list, characterizing them based on the evaluation method that will be used to quantify them (Type A, statistical methods or Type B, scientific judgement) and to categorize them based on their relatedness with something such as a flow chart (shown as Appendices), a cause and effect diagram, or an uncertainty budget table. Using the expression identified in 3.1 provides a good starting point. All of the parameters in this expression may have an uncertainty associated with them. When there are discrete steps in the measurement process, additional uncertainties may be associated with each.

What follows are the most common uncertainties associated with metrological measurements. Keep in mind that this list is exhaustive. Each item listed below is identified as a standard uncertainty  $u$ , when determined using Type B methods of evaluation and a standard uncertainty  $s$ , when determined with Type A methods of evaluation (statistical methods). Each standard uncertainty is further defined by an arbitrary subscript related to the source for ease in remembering that source.

#### 3.2.1. Standard uncertainty from the measurement *process*, $s_p$ , (Type A evaluation).

##### 3.2.1.1. Standard deviation from a measurement assurance chart or control chart.

The value for  $s_p$  is obtained from the control chart limits and the current knowledge that the measurements are in a state of statistical control. This will have to be ascertained by measuring at least one check standard during the course of the current measurements.

### 3.2.1.2. Standard deviation from a series of measurements.

Measure a stable test object at least seven times, no two measurements of which may be made on the same day. Calculate the standard deviation in the conventional manner. The latter is the value of  $s_p$ , keeping in mind that it does not fully represent the measurement process under all typically encountered conditions.

Note: Repetitive measurements made on the same day estimate only the short-term standard deviation of the process.

### 3.2.2. Standard uncertainty for the *standards*, $u_s$ (Type B evaluation).

#### 3.2.2.1. When using standards calibrated by another laboratory.

The information for the standards comes from the calibration report, generally reported as an expanded uncertainty with its coverage factor ( $k$ ). The expanded uncertainty is simply divided by  $k$  to obtain the combined uncertainty for the standard,  $u_c$ , which represents the  $u_s$  when used in your laboratory.

#### 3.2.2.2. When using a standard calibrated in your laboratory (Type B evaluation).

If the standard was calibrated in your own laboratory, calculate the combined standard uncertainty,  $u_c$ , at  $k=1$  and use that as the standard uncertainty for the standard,  $u_s$ .

#### 3.2.2.3. When using more than one standard (Type B evaluation).

When more than one standard is used in a calibration, the standard uncertainty for each,  $u_{s1}$ ,  $u_{s2}$ ,  $u_{s3}$ , etc., is included in the RSS equation if the standards have had independent calibrations. When calibrations are performed at the same time, the standards may be dependent, so the standard uncertainties may be added ( $u_{s1} + u_{s2}$ ) to determine a value to represent  $u_s$ . (This will be the case with two 1 kg standards that were calibrated at the same time using a weighing design and subsequently used together as standards (restraints) in a weighing design.)

#### 3.2.3. Standard uncertainty due to *other* factors, $u_o$ (Type B evaluation.)

These are factors related to the measurement equation, but distinct from the standard uncertainties associated with the process and the standards. These items are often much smaller in a well-controlled process than the standard uncertainties associated with the process and the standards. Examples are given in the flow charts.

The laboratory must carefully consider any other components of uncertainty that might contribute to the uncertainty of the measurement. In mass measurements, these might include effects from the magnitude of air buoyancy corrections as discussed in SOP 2, when these corrections are not made, or the uncertainty from the calculation of air density when buoyancy corrections are made (also discussed in SOP 2).

Additionally, the laboratory should include any other components that are not negligible, such as the uncertainty associated with the density of the standards, the density of the unknown test items, effects due to lack of thermal equilibrium during the test, or known and quantified effects due to magnetized mass standards. Each component that is considered should be included as an additional standard uncertainty  $u_{01}$ ,  $u_{02}$ ,  $u_{03}$ , etc., and included in the RSS equation when data shows these factors to be significant. Note that components of uncertainty are not limited to those mentioned. Documentation of each component evaluated should be maintained to complete the documentation required by ISO/IEC 17025.

#### 3.2.4. Standard uncertainty due to factors *unrelated* to the measurement process, $u_i$ .

These are factors that may be related to characteristics of the items being testing or of the standard and are usually minimized in well-known and controlled measurement processes.

#### 3.2.5. Special uncertainties from other sources (Type B evaluations). Includes bias or unidentified errors.

It is a general requirement of the GUM that corrections be applied for all recognized and significant systematic effects. Where a correction is applied based on a bias, an estimate of the associated uncertainty must then be included in the uncertainty analysis. Due to the various approaches present in the metrology system, several examples and possible approaches are presented in the section on calculating the combined or expanded uncertainties. At this stage, a determination must be made with regard to 1) identifying cause and 2) level of significance.

### 3.2.5.1. Identifying cause.

If the cause can be identified, it is usually corrected or applied to the measurement equation. In some cases, it is not possible to unarguably define the cause without exhaustive studies that provide little benefit. In those cases, the significance level must be evaluated.

### 3.2.5.2. Significance level.

When there is little to be gained from exhaustive studies on the measurement process to identify bias or potential errors, a test of significance may be conducted to determine alternative approaches for incorporating the bias into the uncertainty calculations.

In deciding whether a known bias can reasonably be included in the uncertainty, the following approach may be followed:

3.2.5.2.1. Estimate the combined uncertainty without considering the relevant bias.

3.2.5.2.2. Evaluate whether or not the bias is less than two times the combined uncertainty (i.e.,  $bias < 2\sqrt{u_s^2 + s_p^2 + u_o^2}$ ).

3.2.5.2.3. If the bias is less than twice the combined uncertainty, it may be included in the uncertainty using one of several approaches that must clearly be communicated in the report.

3.2.5.2.4. If the bias is larger than twice the combined uncertainty, the error must be investigated further prior to providing calibration data.

If the deviations show that a standard is out of control, it should not be used for calibration until corrective action has been taken and the value for the standard is verified as being within criteria limits.

$$criteria\ limit : \left| \bar{x}_{lab} - x_{ref} \right| < 2u_c$$

If these differences are smaller than the criteria limits, investigation and corrective action may be unrealistic. If the deviations are less than the surveillance limits shown above, and corrective action is not taken, the deviations may be included in the uncertainty statement following one of several options given in the following section. In ALL cases, the method used to incorporate bias must be clearly reported.

- 3.2.5.3. Adding the bias to the expanded uncertainty (e.g., used in PMAP software). In this case, the bias is simply added to the expanded

$$U + \text{bias} = (u_c * k) + \text{bias}$$

uncertainty and is reported as such.

- 3.2.5.4. Example when uncertainties for the laboratory data and the reference data are considered equivalent (e.g., laboratory data is compared to another laboratory's data).

In this case, a rectangular distribution is considered where the value might possibly be anywhere within the range shown for each laboratory data point. This method is referenced in section 4.6 of NIST Technical Note 1297. This approach may also be used in the case where a standard is predictably drifting with use over time. In this case, a mid-range value is chosen and  $u_d$  (uncertainty for *differences*) is calculated as follows:

$$u_d = \frac{\text{bias}}{2} \frac{1}{\sqrt{3}} \text{ or more simply : } 0.29 \text{ d, where d is the bias}$$

- 3.2.5.5. Example when uncertainties for the laboratory data are considered secondary to a reference value (e.g., the difference between the laboratory data and NIST data). This method was originally published in 1994.

In this case, a reference value is given precedence over the laboratory data and a mid-range value is not chosen. The extreme value is more probable. In this case, the following equation may be used:

$$u_d = \frac{\text{bias}}{\sqrt{3}} \text{ or more simply : } 0.577 \text{ d, where d is the bias}$$

### 3.3. Step 3. Quantify uncertainty estimates

All uncertainty estimates identified in the previous step must be quantified in units that represent the measured values. Type A methods of evaluation usually provide quantified estimates in the units of interest.

Type B methods of evaluation may be conducted with spreadsheets using the basic expression identified in the SOP or identified when the process was specified. Scenario testing can be done to determine the impact and quantify specific variables on the final measured quantity. The knowledge gained in this step often proves useful in identifying potential areas of improvement.

### 3.4. Step 4. Convert all factors to standard uncertainties

In those cases where the uncertainty factors were determined statistically (Type A methods), the standard deviation is used to represent the standard uncertainty. In

other cases, estimates must be made to ensure that the quantified uncertainties represent “one-standard-deviation” values or a k=1 coverage level. The appropriate distribution factor must be used when converting values.

3.5. Step 5. Calculate the combined uncertainty

The combined standard uncertainty,  $u_c$ , includes the standard uncertainty reported for the standards used,  $u_s$ , the standard uncertainty of the measurement process,  $s_p$ , the standard uncertainty from other sources,  $u_o$ , which includes all other factors the laboratory considers significant, the standard uncertainty due to factors related to the measured item but unrelated to the measurement process,  $u_u$ , and finally, the standard uncertainty due to bias or differences,  $u_d$ , when  $u_d$  is included. The standard uncertainties are combined using the root-sum-of-the-squares (RSS) method as follows:

$$u_c = \sqrt{s_p^2 + u_s^2 + u_o^2 + u_u^2 + u_d^2}$$

**Table 1. Symbol descriptions**

| <b>Symbo<br/>l</b> | <b>Description</b>   |
|--------------------|--|
| U                  | Expanded uncertainty   |
| $u_c$              | combined standard uncertainty  |
| $s_p$              | standard uncertainty (standard deviation) of the “process”               |
| $u_s$              | standard uncertainty of the “standard”                                   |
| $u_o$              | standard uncertainty of “other factors”                                  |
| $u_u$              | standard uncertainty of factors “unrelated” to the measurement process   |
| $u_d$              | standard uncertainty of “differences” (may be treated in different ways) |
| k                  | coverage factor  |

### 3.6. Step 6. Calculate the expanded uncertainty

The combined standard uncertainty is then multiplied by a coverage factor,  $k$ , equal to 2 or 3, as chosen by the laboratory, to provide a level of confidence of approximately 95 % or 99 %, respectively. This procedure is based on the Central Limit Theorem and in most cases allows the use of 2 or 3 to provide an approximate 95 % or 99 % level of confidence. The equation used to determine the expanded uncertainty is as follows:

$$U = u_c * k$$

where  $k = 2$  or  $k = 3$ .

### 3.7. Step 7. Evaluate the expanded uncertainty

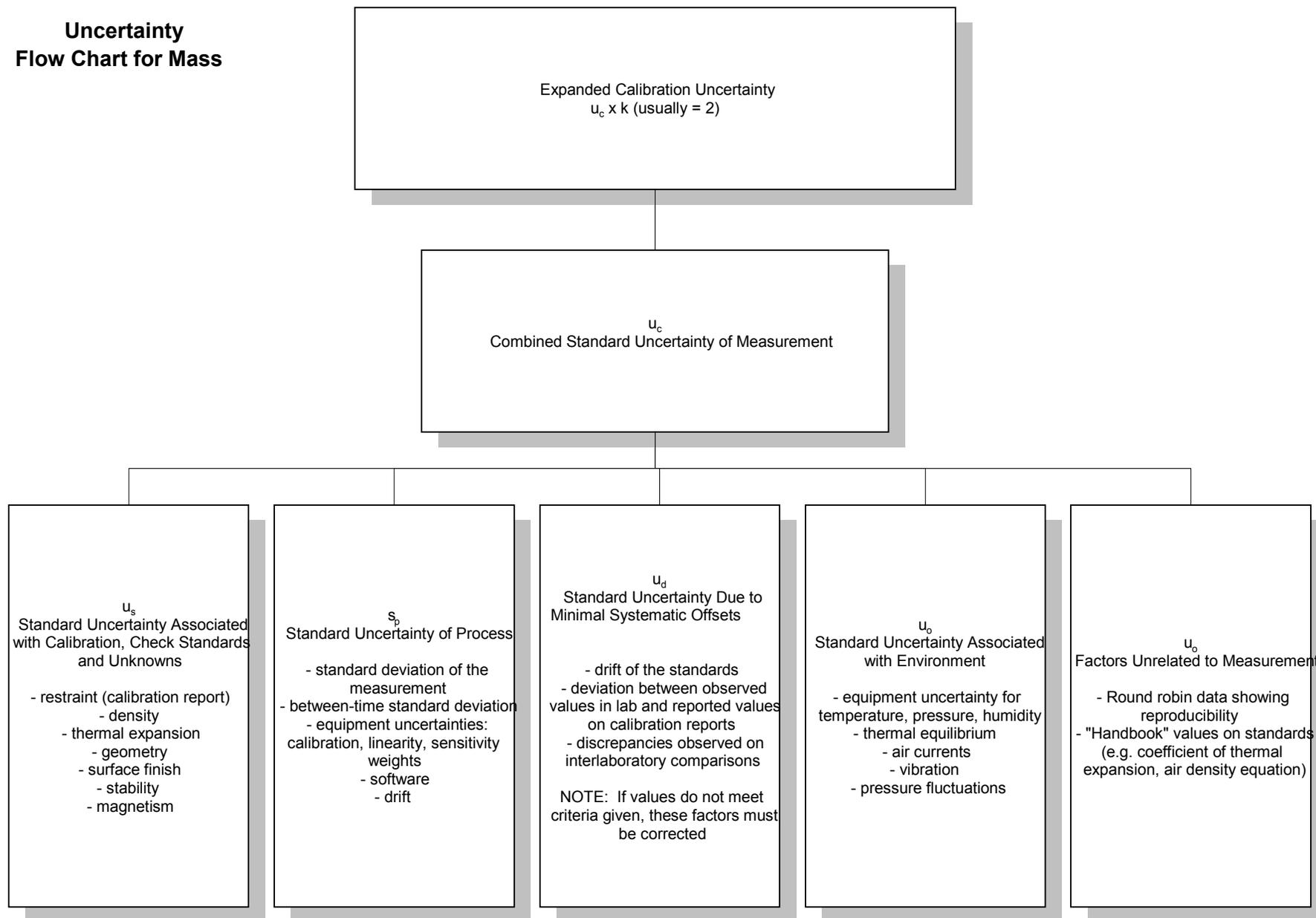
The expanded uncertainty may be evaluated against established criteria such as tolerance limits. For mass standards, the specifications clearly state that the expanded uncertainty must be less than 1/3 of the tolerance.

### 3.8. Step 8. Report the uncertainty

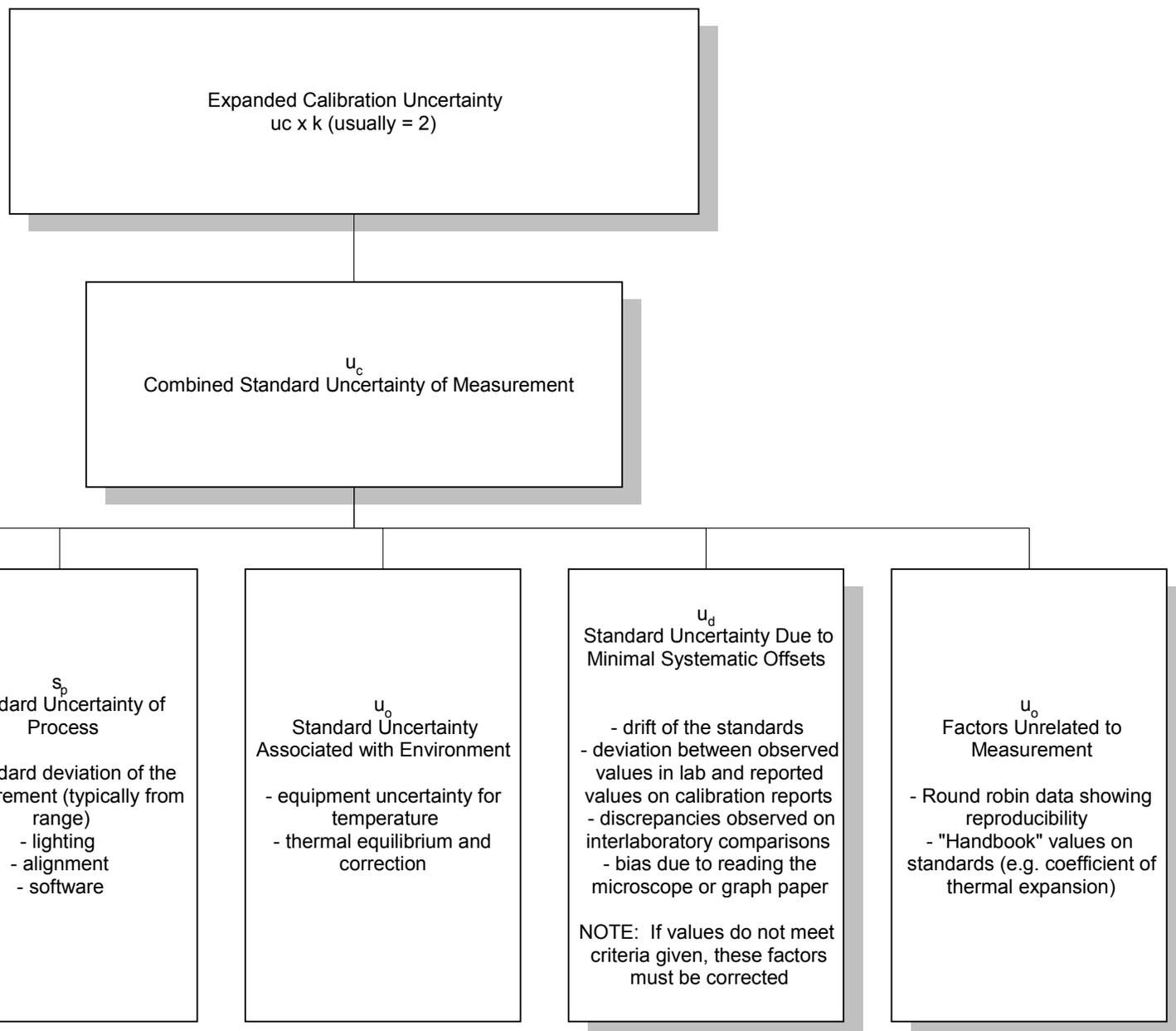
Once the uncertainty has been calculated, a statement such as the following is reported:

The combined standard uncertainty includes the standard uncertainty reported for the standard, for the measurement process, and for any observed deviations from reference (e.g., NIST) values, which are less than surveillance limits. The combined standard uncertainty is multiplied by  $k$ , a coverage factor of (2, 3) to give the expanded uncertainty (which defines an interval with an approximate (95, 99) % level of confidence).

**Uncertainty  
Flow Chart for Mass**



**Uncertainty  
Flow Chart for Length**



**E.4.4 NIST Handbook 145 SOP 30****SOP 30****Recommended Standard Operating Procedure  
for a  
Process Measurement Assurance Program****1. Introduction****1.1. Purpose**

The Process Measurement Assurance Program (PMAP) is used for the control and surveillance of measurement performance using check standards in measurement and calibration procedures. Incorporation of these measurement control steps ensures the validity of the measurement process and the standards used. The variables used in calculation and assignment of uncertainty can be determined and controlled by the use of this Standard Operating Procedure (SOP).

**1.2. Prerequisites**

- 1.2.1. A Standard Operating Procedure that describes and provides rigor and consistency in the calibration process.
- 1.2.2. Check standards that represent the items to be calibrated. These check standards should be stable and their values should be established with accuracy, since they will be used to control the uncertainty in the calibration process. Check standard values are to be determined by NIST or inside the laboratory with a procedure one level higher than the calibration process to be controlled.
- 1.2.3. The operator must be experienced in the calibration process and the standard operating procedure for the calibration to which this method is applied. The operator should also have had specific training on SOP 29 (uncertainty identification and calculations).
- 1.2.4. A calculating system for statistical control that performs standard deviations, control limits, "F" and "t" tests, root-sum-of-the-squares (RSS), and control charting. It is recommended that a computer program be used for efficiency, consistency, and uniformity.

## 2. Methodology

### 2.1. Scope, Precision, Accuracy

This method can be used on any measurement or calibration process where a check standard can be substituted for, or measured as, the item being calibrated. The method duplicates the standard operating procedure with the check standard serving as a surrogate for the item being calibrated. The results of these check standard measurements are recorded, charted, and analyzed to establish the measurement capability and to set process control limits, called reference limits. The reference limits are used to establish process uncertainties and to control future measurement performance.

### 2.2. Summary

Process Measurement Assurance Program (PMAP) is used for determining and controlling the measurement process uncertainty as the calibration is performed. The uncertainty includes effects of the measurement instrument, the operator, the procedure, the standards, and the environment. Each application is modeled to meet the following objective for determining and controlling uncertainties 1) in the measurement process; 2) in the calibration standards; or 3) in both the process and the standards.

The check standard is selected to evaluate the standard deviation of the process ( $s_p$ ), other process uncertainties ( $u_o$ ) such as material density or air density accuracy, and possible bias ( $u_d$ ) of the process (see SOP 29). Reference measurements for the check standard are performed after calibrating the calibration standards and after the servicing of the measurement instrument.

Control measurements of the check standard are graphed for visual examination of process performance and are evaluated against control reference limits.

Statistical tools, 'F' and 't' tests, determine if there are process changes in variability and bias (systematic error). These tests are used when process performance is questionable; when current data is evaluated to establish new reference control limits; and when evaluating uncertainty statements.

2.3. Apparatus/Equipment Required

2.3.2. A calibration process that meets the requirements of the standard operating procedure (SOP).

2.3.3. A check standard that has been selected to evaluate the established objectives of: the 1) measurement process uncertainty; 2) the calibration standards; or 3) both the process and the standards.

2.3.4. A data input, statistical calculating, and control charting system to provide analysis of measurement control data. (i.e., special software package or spreadsheet)

2.4. Table 1. Symbols description

| Symbol   | Description                           |
|----------|---------------------------------------|
| $S_c$    | Control measurement of check standard |
| $S_{cs}$ | Accepted value of check standard      |
| $U$      | Expanded Uncertainty (of the process) |
| $u_c$    | Combined standard uncertainty         |
| $u_s$    | Standard uncertainty of the standard  |
| $u_o$    | Standard uncertainty of other factors |
| $u_d$    | Standard uncertainty of differences   |
| $s_p$    | Standard deviation of the process     |
| $k$      | Coverage factor                       |

2.5. Procedure

2.5.1.

Prelimin

ary Procedure

2.5.1.1. Model the Calibration Process

Set objectives for the PMAP application. These objectives will establish the value of the check standard, the check standard measurement procedure, and influence the frequency of control measurements of the check standard. Objectives may be: 1) Determine the standard deviation of the process ( $s_p$ ); 2) Determine the Expanded Uncertainty ( $U$ ); 3) Measure the value of the calibration standard uncertainty ( $u_s$ ). The model may allow any one objective or a combination of the objectives to be established with a single PMAP application and PMAP control chart.

Diagram the process to clarify the measurement steps and determine the approach that will achieve the established objectives. When determining and controlling the expanded uncertainty ( $U$ ), evaluate the range of use of the process to ensure that the check standard values will determine the variability and the maximum bias that result from the process and the calibration standard. Determining and controlling expanded uncertainty requires duplicating the calibration process and determining where in the process to insert the control measurement using the check standard. When the objective of PMAP is to control the uncertainty of the calibration standard ( $u_s$ ), the PMAP measurement may vary from the calibration process to allow inserting the check standard measurement close to the measurement of the calibration standard (see appendix, Model SOP 8). Diagram the calibration process to establish how many check standards and PMAP applications are required to meet objectives. Also, diagram the process to determine where in the process to insert the control measurements of the check standard.

#### **2.5.1.2. Select and Calibrate Check Standard**

For calibration process uncertainty determination and control, select a check standard that approximates the item to be calibrated. The selected check standard should be selected to evaluate maximum random variation ( $s_p$ ) and bias of the process. The check standard selected should also be used to evaluate other objectives ( $u_o$ ) of the specific calibration process. For multiple ranges of use, a check standard and PMAP application will be required for various portions of the range. For example, Double Substitution (SOP 4) will require, at a minimum, a check standard for each decade (1000 g, 100 g, 10 g, 1 g, 100 mg, and 10 mg) on each balance used. The selected check standard should be calibrated to establish its accepted value ( $S_c$ ) with an uncertainty level sufficient to control the calibration process uncertainty. The calibration of the check standard must be completed using a standard which is independent of the calibration standard that the PMAP process is designed to control.

For control of the calibration standard, use a check standard that is not part of routine measurement and that will evaluate the changes in the calibration standard's accepted value, not the maximum random variation of the process. The check standard used to control the calibration standard should be used less frequently (less than  $\frac{1}{4}$  as often) than the calibration standard. For example, in (SOP 28), using Design A.1.2 (a 4-1 weighing design) at 1000 g, requires the selection of a 1000 g check standard that is measured less frequently than the item under test in order to evaluate the two 1000 g calibration standards. The selected check standard should be calibrated using a

calibration standard other than the calibration standard(s) it will be controlling.

## **2.5.2. Establish Control Measurements for Reference Limits**

**2.5.2.1. Control measurements to establish initial reference limits may be made at any time to verify current measurement process performance. But to achieve control of calibration standards accuracy and measurement instrument capability, the control measurements should be made just after calibration of the calibration standards and servicing of the measurement instrument. Any significant change in the calibration status can then be detected by the performance change in the reference limits data.**

**2.5.2.2. Make the control measurement by duplicating the calibration process with the check standard substituted for the calibrated item. Make an observation of the check standard ( $S_c$ ) and determine its measured value by completing calculations as described in the calibration SOP.**

**2.5.2.3. Record the measured value ( $S_c$ ) and plot it on the control chart and evaluate it with reference to the accepted value of the check standard. Record date, time, and information tags with data.**

**2.5.2.4. Evaluate the bias (difference) between the mean of the measured  $S_c$  values and the accepted value for  $S_c$  from its calibration report.**

Bias (deviation) of check standard = Observed mean of  $S_c$  - Accepted  $S_c$

**2.5.2.5 Repeat the control measurement at various intervals to sample environmental change and other factors than can affect measurement performance. Although a control chart and some statistical control can be established with as few as seven to 12 measurements, a minimum of twenty-five is recommended for estimates and control of uncertainties**

## **2.5.3 Create and Prepare Control Charts**

**2.5.3.1 Construct a graph with the deviation of the check standard measurements on the y-axis and chronological date and**

time (or observation number) on the x-axis. The accepted value of the check standard ( $S_c$ ) is identified near the center of the chart. The y-axis of the control chart should extend plus and minus three standard deviations from the mean ( $\bar{x}$ ) of the control measurements. All control measurements are charted as deviation from the accepted value of the check standard ( $S_c$ )

**2.5.3.2 Establish control chart parameters by calculating the mean and the estimate of the standard deviation ( $s_p$ ) of the check standard reference measurements ( $S_c$ ).**

Control chart parameters are as follows :

Reference Line = Accepted  $S_c$

Mean Line =  $\bar{x}$  of Observed  $S_c$  data

3s Action Limits =  $\bar{x} + 3(s_p)$

2s Warning Limits =  $\bar{x} + 2(s_p)$

Process Bias = Mean of Observed  $S_c$  - Accepted  $S_c$

## 2.5.4 Establish Reference Limits and Process Uncertainty

**2.5.4.1 Establish reference control limits (as described in 2.5.3.2) by calculating control limits and process bias using the control measurements obtained when calibration standards and measurement instruments are calibrated. These limits are to be stored and used as a reference for future control measurements. Future control measurements should be control-charted and tested against these limits for “in” or “out” of control status. This reference data will also be statistically used to periodically evaluate process and calibration standard performance for change from the calibrated reference status.**

**2.5.4.2 Establish the Expanded Uncertainty (U) by using the reference data, calibration standard uncertainty ( $u_s$ ), and any other uncertainty ( $u_o$ ) not covered by the reference data.**

$$u_c = \sqrt{(s_p)^2 + (u_s)^2 + (u_o)^2 + (u_d)^2}$$

$$U = k * u_c$$

According to the PMAP model,  $s_p$ ,  $u_o$ , and  $u_d$  are evaluated by comparison with the reference data. The uncertainty for the standard,  $u_s$ , and, in some situations, additional  $u_o$  are included in PMAP calculation of the process expanded uncertainty (U).

## 2.5.5 Make PMAP Control Measurement

**2.5.5.1 Control measurements of the check standard should be made periodically to ensure that the current measurement performance remains in control of the established reference limits. Control measurements should be tested for “in” or “out” of control status and charted on the control chart. The frequency of the control measurements is dependent on the objective of the PMAP application.**

When the objective is to determine and control the calibration process uncertainties, control measurements should be made during the calibration process to ensure the calibration results are accurate and within reference uncertainty statements. Control measurements should be made prior to returning calibrated items to the customer. A minimum of 25 control measurements are required within the

calibration period or interval assigned to the calibration standards and the calibration process.

When the objective is to control calibration standards, working standards, or primary standards, the frequency of control measurements should be less than ¼ the use of the standard being controlled. The reduction in measurements ensures that the check standard receives less use and wear than the standard being controlled. Control measurements are charted and the Process Bias (observed mean value – accepted) is evaluated to detect any significant change in the calibration standard being controlled.

## **2.5.6 Evaluate Process Performance at Specific Intervals**

**2.5.6.1 Examine each control measurement data point as it is charted. Evaluate each data point for its control status and investigate causes for out of control data. Analyze the measurement process uncertainty, including the process standard deviation ( $s_p$ ), process bias, and other uncertainties quarterly or every five to ten data points to ensure that significant changes in uncertainties do not occur.**

**2.5.6.2 Evaluate current performance using the control measurements described in 2.5.5 to establish the current standard deviation of the process ( $s_p$ ), process bias and other uncertainties. Perform each evaluation at specific calibration intervals established by the calibration of standards and service of the measurement instrument. This evaluation, referred to as “calibration” of the process, is performed at intervals that will ensure detection of changes in the calibration uncertainty statements.**

**2.5.6.3 Use statistical tools at specific calibration intervals to evaluate current data performance to reference data that was established at calibration of standards and at the service of the measurement instrument. This evaluation will assist in deciding when to recalibrate calibration standards and service the measurement instrument.**

Use the F test to evaluate if a significant change in ( $s_p$ ) performance has occurred.

$$F \text{ test} = \frac{(s_{p \text{ large}})^2}{(s_{p \text{ small}})^2}$$

The current and previously established reference standard deviations are compared and evaluated using F test table values based on degrees of freedom in the measurements.

Use the t-test to evaluate if a significant change in measurement process bias has occurred.

$$t \text{ test} = \frac{(Bias_{\text{new}}) - (Bias_{\text{old}})}{\sqrt{\frac{(s_{p \text{ new}})^2}{n_{\text{new}}} + \frac{(s_{p \text{ old}})^2}{n_{\text{old}}}}}$$

The current (new) and previous (old) reference bias is compared and evaluated using t test table values based on the degrees of freedom in the measurements.

**2.5.6.4. Take action based on the results of statistical evaluation. If F and t tests reveal no significant change in process performance, use the current data analysis to establish new process reference limits, control chart and uncertainty statements (as described in 2.5.3 and 2.5.4). If the process has a stable history, it is permissible to pool the current data with previous reference limits to establish new reference limits, control chart, and uncertainty statements. Continue control measurements until the next calibration interval.**

If F and t tests reveal significant change in the measurement process, investigate the specific cause. If the cause for the change cannot be identified and corrected, then collect new reference data to establish new reference limits and process uncertainties. If a specific cause is found and corrected, and subsequent control measurements indicate an “in-control” status, continue collecting control measurements and test against established reference limits until the next calibration interval.

**2.5.7. Continue the measurement assurance procedure as described in 2.5.2 through 2.5.6 to determine and control the measurement process capability.**

Significant changes in the measurement process capability can result from the following:

- measurement procedure change;
- measurement instrument change;
- calibration standards change; and/or
- location change.

These changes can require repeating the PMAP procedure (from 2.5 through 2.5.6).

## **E.4.5 NIST Handbook 145 GLP 1**

### **GLP 1**

#### **Good Laboratory Practice for the Quality Assurance of the Measurement Process**

Quality Assurance of the Measurement Process means establishing, documenting, implementing, and maintaining a quality system appropriate to the laboratory's scope of activities. Having such a system in place will allow the laboratory to know, within the limits of a measurement process, that a measurement is valid with respect to its traceability, accuracy, and precision.

The validity of tests and calibrations should be monitored with quality control procedures. Statistical techniques are used to record and monitor charted measurement results to permit the detection of trends. The metrologists and laboratory management should plan and review the results from quality assurance monitoring.

Other steps taken to ensure the quality of the measurement process may include, but are not limited to:

- the regular use of Standard Reference Materials (SRMs) and /or internal quality control using secondary reference materials;
- participation in interlaboratory comparisons (round robins);
- test replications with same and/or different methods;
- recalibration of retained items;
- correlation of different characteristics of an item; and
- proper calibration intervals.

Each measurement parameter in the laboratory's scope of activities should be reviewed and analyzed to determine the validity of the measurement process.

The standards and the measurement process for each parameter must be in a state of statistical control. Statistical control means that the variability of the measurement process is known and stable; when a process is in statistical control, we can assume that the reported measurement uncertainties are valid. The National Institute of Standards and Technology provides technical guidance and support to the State weights and measures laboratories to develop measurement control programs that provide measurement assurance. The objective of these programs is to evaluate the entire measurement process including:

- procedures;
- standards;
- equipment;

- personnel; and
- environment.

While other quality assurance programs could meet these objectives, the control programs developed for measurement assurance greatly increase the comprehensiveness of the program.

The State laboratories typically provide measurement services in the disciplines of mass, volume, and length. Some laboratories provide services in other measurement areas. Approximately 89 % of their workload is in mass standards calibration.<sup>13</sup> Mass calibration demands the greatest precision and is the first discipline for which a measurement assurance program was developed and implemented. Nevertheless, all measurement disciplines should have a measurement assurance system in place.

The most recent improvement in assuring the quality of each measurement parameter in the State Laboratories is the incorporation of a Process Measurement Assurance Program (PMAP).<sup>14</sup>

A measurement assurance program consists of duplicating the measurement process by including a check/control standard as surrogate for the test item. Measurements made throughout the year will show all the conditions that are likely to affect the measurement process and their combined effects. Controlled duplication of the process provides for the realistic evaluation of the variability of the measurement process as one of the components in the estimation of the measurement uncertainty.

Measurement results that are collected over several years may be statistically evaluated with current results being compared to results from previous years. Any observed problems or changes in the measurement results are investigated and if necessary, corrective action can be taken. Ongoing monitoring establishes a continuous, comprehensive, internal measurement assurance program in a State laboratory.

Data from internal measurement assurance programs is compared to the results of inter-laboratory comparisons (round robins) in which the laboratory participates as part of the Regional Measurement Assurance Program (RMAP) groups. (These groups consist of the metrologists of the State laboratories and their counterparts in private industry in a geographic region of the country. Six regional measurement assurance groups operate in the United States and its territories. )

The strength of the measurement assurance approach lies in its applicability to a wide variety of measurements with sufficient flexibility to permit each measurement control program to be tailored to the particular needs of a given measurement area. The sophistication of the control program depends on the criticality of the measurement.

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<sup>13</sup> State Laboratory Program Workload Survey, 1999, NIST/OWM & NCWM Metrology Subcommittee

<sup>14</sup> See NISTIR 6176 and SOP 30.



## E.4.6 NIST Handbook 145 GMP 10

### GMP 10

#### Good Measurement Practice for Understanding Factors Affecting Weighing Operations

Good laboratory techniques and practices, as well as good standards and balances, are required to obtain good mass measurements. Errors can arise from a variety of sources, such as balance drift, air currents, lack of thermal equilibrium, magnetic or electrostatic fields, and to manipulative skills using the balance. The magnitude and significance of these errors depends upon the laboratory environment, the balance, and the accuracy required for the measurement. Different methods of weighing are used to eliminate or reduce the effects of sources of error. Particular sources of error and ways to eliminate the errors are discussed with the weighing procedures. The accuracy required for a measurement and the criticality of the measurement often influence the choice of the weighing method used in the measurement. Regardless of the procedure used, several practices should be followed to promote good measurements.

#### Environment and Preparation

First, the balance should be installed in a laboratory having good temperature and humidity control. The requirements for temperature stability increase as more precision is needed in the measurement. For high precision measurement, the temperature should not vary by more than  $\pm 1$  °C in a 24 h period throughout the year and should vary by less than 0.5 °C during the course of any one measurement series (typically less than 1 h). General laboratory measurements can probably withstand temperature variations of  $\pm 2$  °C per day. Varying temperatures result in balance drift and in unequal temperatures for the air, balance, and objects to be weighed. The relative humidity should be maintained between 40 % and 60 %. If the relative humidity falls significantly below 40 %, electrostatic discharge may be generated both in and near the balance. This can result in erratic readings and make precision weighing impossible. If precision mass standards are cleaned, they should stabilize in the laboratory environment for seven to ten days.

Thermal and environmental equilibrium are critical for mass calibrations. This is why each mass SOP will provide instruction that the environment must be stable and the weights set in or near the balance for 24 hours prior to a calibration. The test objects, balances, reference standards, and air should all be in thermal equilibrium. When possible, the objects to be weighed and the reference standards should be placed either in the balance chamber or adjacent to the balance so all can come to the same thermal equilibrium. If there is a lack of thermal equilibrium, convection currents will be generated when an object is on the balance pan and an erroneous reading may result. These types of errors are likely to go unnoticed when check standards are handled the

same way, so care must be taken to reduce the likelihood of their occurrence. Tests have shown that these types of errors depend upon the temperature differences among the objects and the balance, and on the geometry of the objects being weighed. On 20 g standards of greatly differing geometry, studies have shown that errors as large as 0.2 mg can occur for a 4 °C temperature difference among the standards and the balance. Errors as large as 3 mg have also been observed at 1 kg loads when standards were stored in a cabinet, and unknown test items near the balance where a temperature gradient of 2 °C was present (when the uncertainty was less than 0.1 mg).

The balance must be installed in an area free from air currents. Balances should be installed away from heating/cooling vents. It is not adequate to merely close vents when weighing because disrupting the temperature control system may result in temperature changes near the balances.

### Handling Weights

The masses of standard weights or objects weighed can be affected significantly by the manner in which they are handled. Human contact can leave grease or oily films that affect the mass at the time of measurement and can even cause permanent damage due to corrosion.

Small weights should never be touched by hand, but handled using forceps, clean gloves, or swatches of cloth. In the latter case, the cloth must be lint free. Instructions for cleaning weights and for removing adhering foreign material are described in GMP 5.

Large weights of lower tolerance classes (NIST Class F) may be handled by bare hands. Large weights are a source of special problems. Fork lifts, portable cranes, hoists, or even overhead cranes may have to be used to move the weights within the laboratory and on or off the balances. Laboratory personnel must become expert in using such equipment, as necessary, to avoid damage to the laboratory facilities, to the balances used, and even to the weights themselves. Special hoist/crane hydraulics or multi-speed systems are available to gently set large weights in place on large comparators to avoid damage. The problem of temperature equilibrium for large weights is qualitatively the same as for small weights and needs consideration on an individual basis.

Large weights must be clean at the time of use, but cleaning may be a problem. Minimally, they should be inspected to ensure that foreign material is not present. Cleaning is addressed further in GMP 5.

### Operation

Analytical balances are high precision instruments and should be operated carefully. Excessive shock can damage a balance. Avoid shock loading the balance. Gently

place all weights in the center of the weighing pan/platform. The dials on mechanical balances should be turned slowly and gently. Careful balance operation will improve the repeatability of measurements.

Mechanical analytical balances are provided with partial and full release positions. The partial release position is used when determining if an unknown load will provide an on-scale reading. The balance beam has limited movement in this position. The partial release position provides some protection to the balance when the dialed-in weights are not close to the actual mass placed on the pan. It is good practice to arrest the pan each time a dial is being changed to protect the balance from shock loading. It is acceptable to change the dial representing the smallest built-in weights when in the partial release position because the small weight changes should not result in shock loading of the balance.

When releasing the pan to either the full or partial release position, the action should be done slowly and carefully. The objective is to minimize disturbances to the balance as much as possible.

Similarly, all loads should be placed on the balance pan carefully and centered on the pan.

When a mechanical balance is released, the beam goes through a series of oscillations. The frequency of these oscillations diminishes as time passes until they are almost imperceptible to the naked eye. At this point, optimal stabilization is achieved. This stabilization of the balance typically lasts for a very short period of time, after which the balance reading will usually drift. A similar situation occurs when a mass is placed on the pan of an electronic balance. Therefore readings should be taken at the precise moment of achieving balance stability. This interval between the releasing of a pan on a mechanical balance, or the placing of a mass on an electronic balance, and the reading of the observation, varies from balance to balance. Stabilization time differs for all balances, even among those of the same type and model. Knowledge of the instrument is critical to correctly establish this time interval. Although manufacturers will usually state this value, it is necessary for the metrologist to verify its reliability. Many electronic balances have a stability signal incorporated into the design, but this also must be verified. All measurements in a calibration should be performed at the same time interval, and within the shortest time possible.

All balances should be exercised before taking readings. A load equal to the load to be measured should be placed on the balance, a reading taken, and the pan arrested, if appropriate, or the weight removed from electronic balances. This operation should be repeated several times before readings are taken for measurement purposes. Once the balance has been "warmed-up", better repeatability will be achieved. Balances can be very accurate even when used without being exercised first, but improved results can be obtained by going through a "warm-up" procedure. If the larger variation present in initial weighings on a balance that has not been exercised is not significant to the measurement, the warm-up procedure may be minimized.

To determine the repeatability of measurements when a balance has not been exercised versus its performance after being exercised, and to determine how much warm-up time is required, begin measurements starting with the unexercised balance and record the readings. Repeat a series of measurements until you have obtained several measurements after the balance has been exercised. This test can be repeated over several days using the same objects and the results compared. The readings obtained when using an unexercised balance are likely to show a slightly larger variation than those obtained after the balance has been exercised. Balance drift is likely to be larger initially and then reach a steady state when the balance has been "warmed-up".

## Comparison Weighing - Mechanical Balance

For mass calibrations, the unknown object must be compared to a known mass standard. Comparison weighing eliminates the errors of the built-in weights, reduces disturbances during the measurement because dial settings are not changed during the measurement, and can cancel the effect of drift by selecting the appropriate weighing design. Comparing the unknown,  $X$ , to a standard,  $S$ , eliminates the built-in weights from the measurement: thus, the built-in weights act only as counterweights; they do not affect the difference measured between  $X$  and  $S$ . Consequently, the dial settings must not be changed during a comparison measurement; otherwise the built-in weights would be part of the measurement.

When comparison measurements are made on a single pan mechanical balance, all readings are taken from the optical scale. The unknown and the standard must have nearly the same mass so that the difference between them can be measured on the optical scale. If the masses of the unknown and the standard are significantly different, small mass standards are used as tare weights with either the unknown or the reference standard or both to obtain an observed mass difference that is significantly less than one-fourth the range of the optical scale.

As part of a comparison measurement, the mass value of a scale division is determined by performing a sensitivity determination. The small mass standard used as part of the weighing design to calibrate the optical scale is called the sensitivity weight and should have a mass between  $1/5$  and  $1/2$  the range of the optical scale, inclusive. Additionally, the mass of the sensitivity weight should have a mass that is at least twice the observed mass difference between the standard and the unknown. Since the maximum size of the sensitivity is limited to one-half the range of the optical scale, it may be necessary to carry tare weights with the standard and the unknown to ensure that the observed difference between them is less than one-half the mass of the sensitivity weight. The value of the sensitivity weight should include the correction plus its nominal value. For high precision calibrations, the inclusion of the air buoyancy correction for the sensitivity weight is critical. See GMP 14 for detailed guidance on the selection of sensitivity weights.

The first readings for the standard and the unknown in a comparison on a single pan balance should fall in the first quarter of the optical scale, but well ahead of zero, so the balance drift will not result in negative values for any readings. Although negative numbers may be used in calculations, they are avoided to simplify calculations and reduce calculation errors. Because the sensitivity weight may have a mass as large as one-half the range of the optical scale and the measured difference between the standard and the unknown may be as large as one-fourth the range of the optical scale, it is necessary to obtain the first two readings in the first quarter of the optical scale so all readings will remain on-scale for the measurement. In this way it is not necessary to change the dial settings to measure the difference between the standard and the unknown.

## Comparison Weighing - Electronic Balance

Measurements made on a full electronic balance are simplified because there are no built-in weights to consider. Although many electronic balances are equipped with a built-in calibration weight, the weight is not involved in the comparison weighing.

The principles for comparison weighing on a full electronic balance are the same as when using a single pan mechanical balance. The balance indications are used to measure the mass difference between the standard and the unknown, and a sensitivity weight is used to establish the mass value for a digital division on the balance. Since there are no built-in weights in the full electronic balance, the entire range of the digital indications can be considered for "optical scale" of the balance.

For comparison weighing the standard and the unknown should be "nearly the same mass." Since a full electronic balance has a much larger range for indicating mass values, the masses do not have to be as close together as when a mechanical balance is being used. When using an electronic balance, the difference in mass between the standard and unknown should be less than 0.05 % of the balance capacity. Tare weights that are known standards should be used if the masses are not sufficiently close together. The sensitivity weight used to determine the mass per digital division should have a mass that is at least 10 to 20 times the mass difference between the standard and the unknown but not exceeding 1 % of the balance capacity. For high precision weighing, air buoyancy corrections must be made for all objects used in the intercomparison.

## Magnetic and Electrostatic Fields

Care must be taken when weighing magnets or objects having electrostatic charges. A magnetic field will likely affect results of a mass measurement. A magnet is attracted to ferromagnetic materials in the balance and balance chamber. The magnetic field may also affect the magnetic field generated in an electronic balance that utilizes the principle of magnetic force restoration as its method of measurement.

Weights made of ferromagnetic material can become magnetized during manufacture or during use if they are placed in a strong magnetic field. Magnetized weights can result in measurement errors that may go undetected. If a measurement problem is suspected, the weights should be checked for magnetism and may have to be rejected if excessively magnetized.

If magnets or magnetized material must be weighed, the material should be placed in a magnetically shielded container to prevent the magnetic field from generating measurement errors. If balance design and conditions permit, an alternative is to position the magnetized material a relatively large distance from the balance pan using a non-ferromagnetic object on the pan to serve as a platform for the magnetic. Since the strength of the magnetic field drops off at a rate of the cube of the distance from the

magnetic, it may be possible to effectively isolate the magnet from other ferromagnetic material in the balance.

Electrostatic fields can also cause errors in measurements. If there is a static charge in a mechanical balance, the balance may give erratic readings and lack repeatability. If the object being weighed has a significant electrostatic charge, it may result in measurement errors and may leave a static charge on the balance. Electrostatic charges are of particular concern when plastic containers are placed on the balance.

Care should be taken to remove electrostatic charges from objects being weighed by grounding the objects, if necessary, before placing them on the balance. To prevent the build-up of static electricity in a balance, the relative humidity in the laboratory should be maintained between 40 % and 60 %. The water vapor in the air will serve to drain off electrostatic charges from the balance.

Balances utilizing the magnetic force restoration principle for weighing should be checked to verify that the magnetic field generated by the magnetic cell in the balance does not exist around the balance pan. If the shielding of the magnetic cell is inadequate, measurement errors may occur when weighing ferromagnetic objects or when the balance is placed on a surface comprised of ferromagnetic material.



## E.4.7 NIST Handbook 145 GMP 13

### GMP 13

#### Good Measurement Practice for Ensuring Traceability

##### 1. Introduction

###### 1.1 Purpose

Traceability is defined as “the property of a result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties”.<sup>15</sup> Traceability ensures that the measurements are accurate representations of the specific quantity subject to measurement, within the uncertainty of the measurement.

To ensure traceability, suitably calibrated standards that are appropriately maintained and cared for, proper standard operating procedures, continuous measurement control, surveillance, and suitable documentation must all be present.

Test numbers issued by NIST should not be used nor required as proof of the adequacy or traceability of a test or measurement. Having a NIST number does not provide evidence that the measurement value provided by another organization is traceable. (See section 1.3.3.)

###### 1.2 Prerequisites

Traceability can be characterized by the following essential elements:

1.2.1 *Unbroken chain of comparisons.* A documented system of comparisons going back to a standard acceptable to the parties, usually a national or international standard;

1.2.2 *Measurement uncertainty.* The measurement uncertainty for each step in the traceability chain must be calculated according to defined methods and must be stated so that an overall uncertainty for the whole chain may be calculated (see SOP 29);

1.2.3 *Documentation.* Each step in the chain must be performed according to documented and generally acknowledged procedures (see GMP 12) and the results must be documented (i.e., in a calibration report, see SOP 1);

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<sup>15</sup> ISO VIM, 2<sup>nd</sup> ed., 1993, definition 6.10  
GMP 13

- 1.2.4 *Competence.* The laboratories or bodies performing one or more steps in the chain must supply evidence of technical competence (e.g., by maintaining appropriate training records, participating in interlaboratory comparisons, and by demonstrating that they are accredited by a recognized accreditation body);
- 1.2.5 *Realization of SI Units.* The primary national, international or intrinsic standards must be primary standards for the realization of the SI units;
- 1.2.6 *Periodic recalibration.* Calibrations of standards (and equipment where appropriate) must be repeated at established (may be defined through measurement assurance) and appropriate intervals to preserve the traceability of the standard over time and use (see GLP 4, GMP 11); and
- 1.2.7 *Measurement assurance.* A proper measurement assurance program must be established to ensure the validity of the measurement process and the accuracy of standard used (see SOP's 9, 17, 20, 30).

### 1.3 Responsibility

- 1.3.1 *Provider.* Providing support for the claim of traceability of the result of a measurement or value of a standard is the responsibility of the calibration provider. Calibration reports and certificates must contain a traceability statement.
- 1.3.2 *User.* Assessing the validity of a claim of traceability is the responsibility of the user of that result or value. Verifying claims of traceability often includes obtaining a calibration directly from a national metrology institute or another laboratory that has achieved recognition or accreditation through a recognized accrediting body.
- 1.3.3 *Use of, or reference to, official test numbers of a national metrology institute.* Having an authentic test number does not provide assurance or evidence that the measurement value provided by another organization is traceable. Not only must there be an unbroken chain of comparisons, but each measurement must be accompanied by a statement of uncertainty associated with the value. Test report numbers should not be used nor required as proof of the adequacy or traceability of a test or measurement. National and international standards dealing with test and measurement quality requirements, such as ANSI/NCSL Z 540-1, ISO 10012, ISO/IEC 17025 and the ISO 9000 series do not require the use or reporting of specific test numbers to establish traceability.

1.4 Safety

No outstanding safety concerns

2. Methodology

2.1 Summary

Traceability is maintained through comparison to appropriate standards with suitable procedures and measurement uncertainties. Procedures are outlined in SOPs and GMPs. Examples of possible hierarchies of the standards leading to the traceability of a calibration are provided in this document.

2.2 Procedure

The charts in this GMP show examples of the traceability hierarchy for mass, length, volume, and temperature measurement disciplines. Each laboratory must define the exact traceability system in their operating quality manual. (A worksheet is included as an Appendix to help in the definition of the traceability system.)

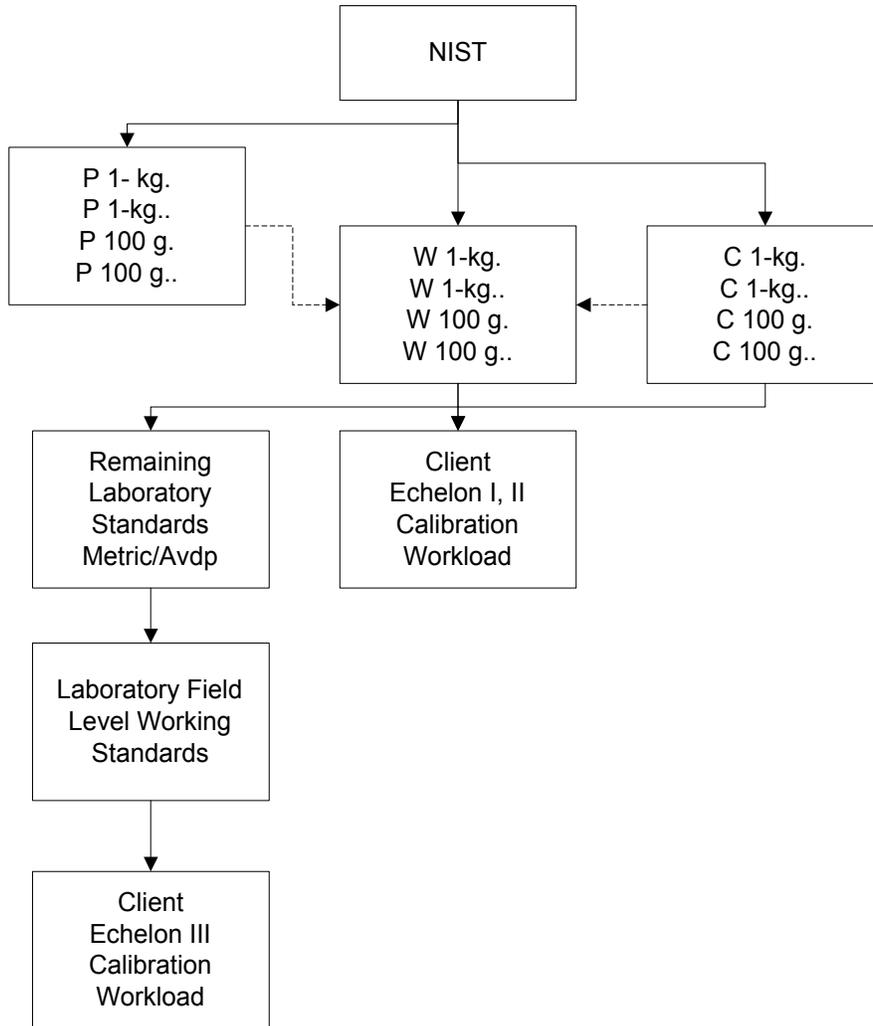
3. Calculations

There are no calculations in this GMP.

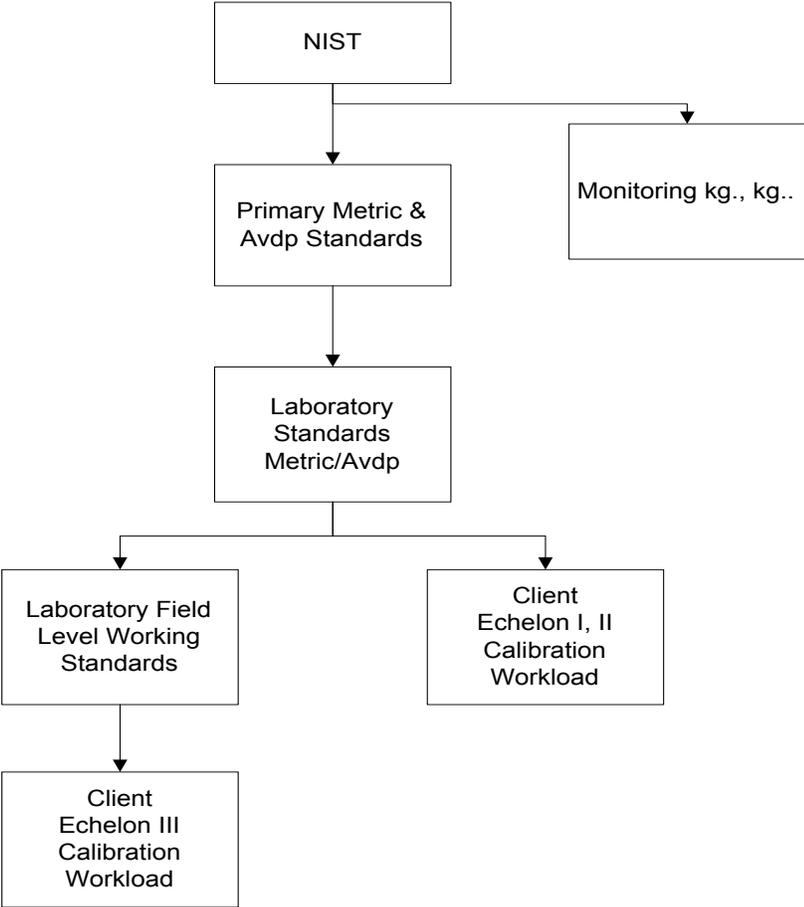
4. Assignment of Uncertainty

The uncertainty associated with reported calibration values is included within the uncertainty analysis for each SOP and in SOP 29, Calculating and Reporting Uncertainties.

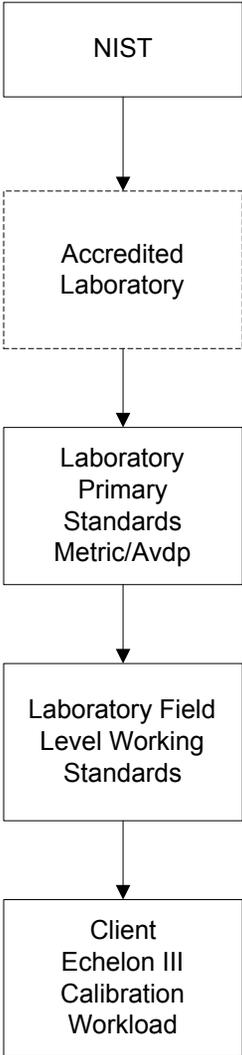
Mass - Option A



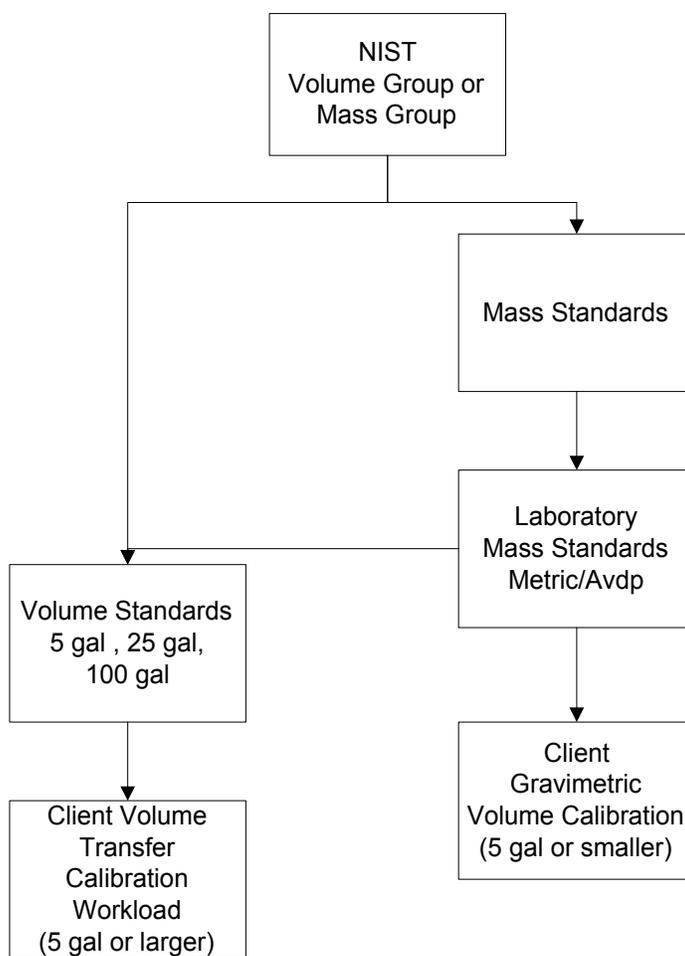
Mass - Option B



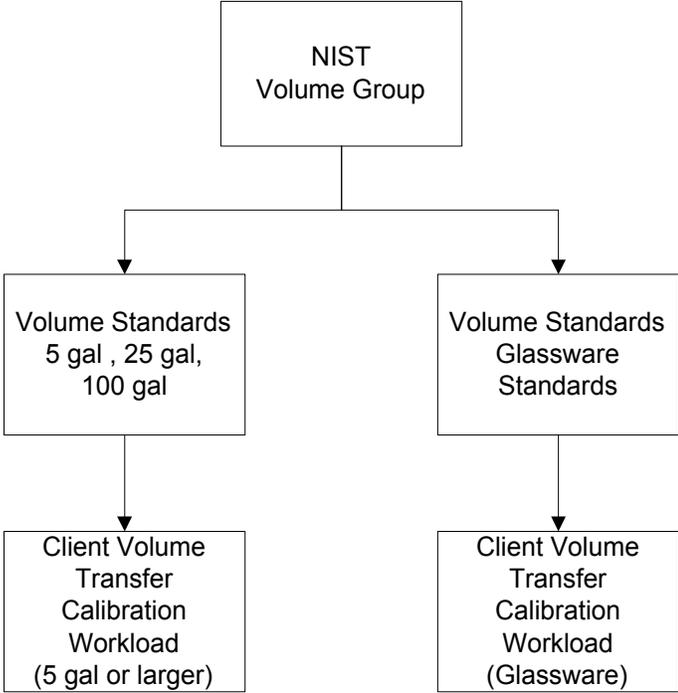
Mass - Option C



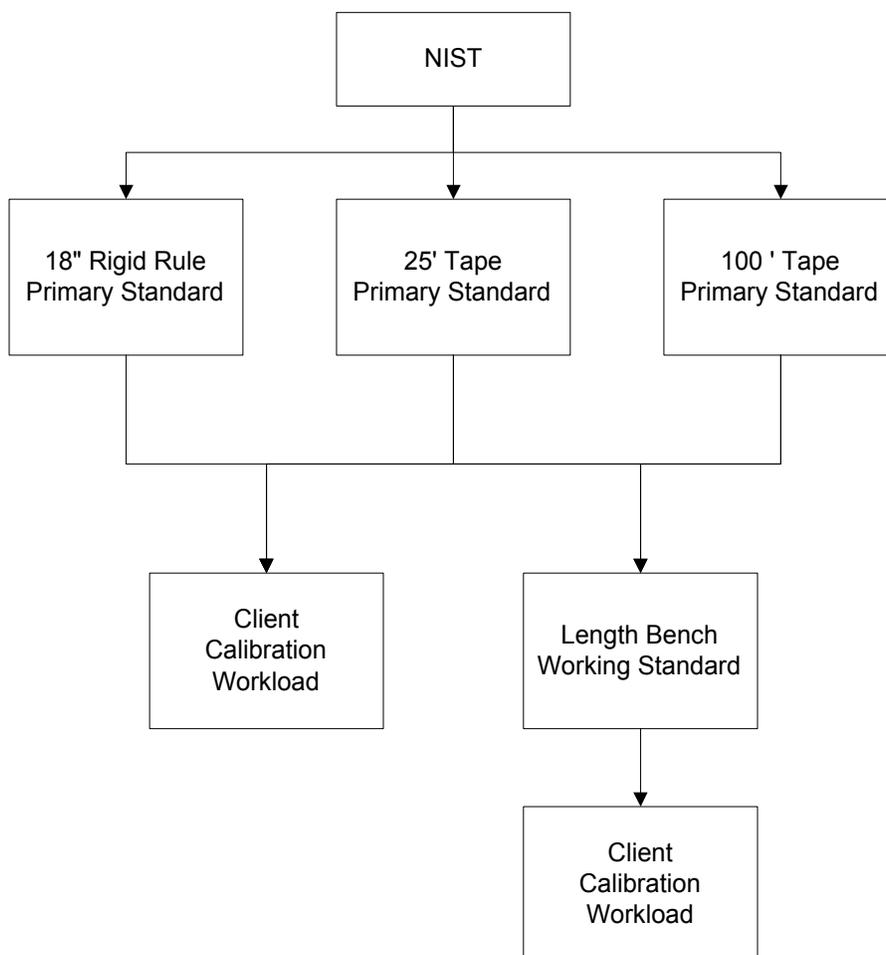
### Volume - Option A

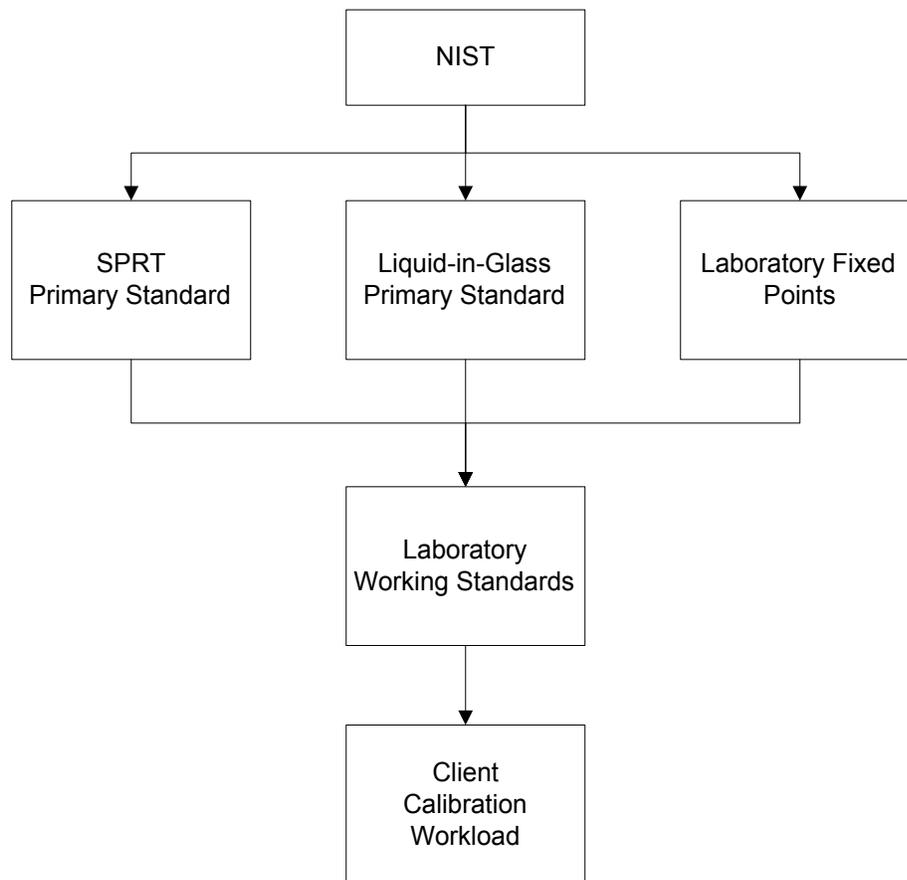


**Volume - Option B**



### Length





**Temperature**

**Appendix A - Traceability worksheet**

**Parameter: Mass, Volume, Length, Temperature, Other**

| <b>P</b> |              | <b>Metric (g, L, m, °C)</b> | <b>Cal. date</b> | <b>Cal. Lab</b> | <b>Interval</b> |
|----------|--------------|-----------------------------|------------------|-----------------|-----------------|
|          | <b>Range</b> |                             |                  |                 |                 |
|          |              |                             |                  |                 |                 |



**E.5 EPA Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Compendium Method IO-3.3.**

EPA/625/R-96/010a

**Compendium of Methods  
for the Determination of  
Inorganic Compounds  
in Ambient Air**

**Compendium Method IO-3.3**

**DETERMINATION OF METALS  
IN AMBIENT PARTICULATE  
MATTER USING  
X-RAY FLUORESCENCE (XRF)  
SPECTROSCOPY**

**Center for Environmental Research Information  
Office of Research and Development  
U.S. Environmental Protection Agency  
Cincinnati, OH 45268**

**June 1999**

## Method IO-3.3

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- James L. Cheney, U.S. Army Corps of Engineers, Omaha, NE
- Michael F. Davis, U.S. EPA, Region 7, KC, KS
- Joseph B. Elkins Jr., U.S. EPA, OAQPS, RTP, NC
- Robert G. Lewis, U.S. EPA, NERL, RTP, NC
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- William A. McClenny, U.S. EPA, NERL, RTP, NC
- Frank F. McElroy, U.S. EPA, NERL, RTP, NC
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

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#### Author(s)

- Bob Kellog, ManTech, RTP, NC
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

#### Peer Reviewers

- David Brant, National Research Center for Coal and Energy, Morgantown, WV
- John Glass, SC Department of Health and Environmental Control, Columbia, SC
- Roy Bennet, U.S. EPA, RTP, NC
- Charles Lewis, EPA, RTP, NC
- Ray Lovett, West Virginia University, Morgantown, WV
- Lauren Drees, U.S. EPA, NRMRL, Cincinnati, OH

### DISCLAIMER

***This Compendium has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.***

**Method IO-3.3**  
**Determination of Metals in Ambient Particulate Matter Using**  
**X-Ray Fluorescence (XRF) Spectroscopy**

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## Chapter IO-3 CHEMICAL SPECIES ANALYSIS OF FILTER-COLLECTED SPM

### Method IO-3.3 DETERMINATION OF METALS IN AMBIENT PARTICULATE MATTER USING X-RAY FLUORESCENCE (XRF) SPECTROSCOPY

#### 1. Scope

**1.1** During a span of more than two decades, the U. S. Environmental Protection Agency (EPA) has developed and applied x-ray fluorescence (XRF) to the analysis of ambient and source aerosols using both energy and wavelength dispersive spectrometers. Inorganic Compendium Method IO-3.3 briefly describes the agency's experience with XRF and informs the reader of its capability in elemental aerosol analysis and attempts to give a brief account of what is involved in its application. The procedures described have been in a continual state of evolution beginning with those in use on a special purpose spectrometer designed by Lawrence Berkeley Laboratory (LBL) and eventually applied to a commercially available instrument manufactured by Kevex. It is for the Kevex spectrometer to which this method applies.

**1.2** The area of toxic air pollutants has been the subject of interest and concern for many years. Recently the use of receptor models has resolved the elemental composition of atmospheric aerosol into components related to emission sources. The assessment of human health impacts resulting in major decisions on control actions by Federal, state, and local governments is based on these data. Accurate measures of toxic air pollutants at trace levels is essential to proper assessments.

**1.3** Suspended particulate matter (SPM) in air generally is considered to consist of a complex multi-phase system consisting of all airborne solid and low vapor pressure, liquified particles having aerodynamic particle sizes ranging from below 0.01 microns to 100 (0.01  $\mu\text{m}$  to 100  $\mu\text{m}$ ) microns and larger. Historically, measurement of SPM has concentrated on total suspended particulates (TSP) with no preference to size selection.

**1.4** The most commonly used device for sampling TSP in ambient air is the high-volume sampler, which consists essentially of a blower and a filter, and which is usually operated in a standard shelter to collect a 24-hour sample. The sample is weighed to determine concentration of TSP and is usually analyzed chemically to determine concentration of various inorganic compounds. When EPA first regulated TSP, the National Ambient Air Quality Standard (NAAQS) was stated in terms of SPM with aerodynamic particle size of  $<100 \mu\text{m}$  captured on a filter as defined by the high-volume TSP sampler. Therefore, the high-volume TSP sampler was the reference method. The method is codified in 40CFR50, Appendix B.

**1.5** More recently, research on the health effects of TSP in ambient air has focused increasingly on particles that can be inhaled into the respiratory system, i.e., particles of aerodynamic diameter of  $<10 \mu\text{m}$ . These particles are referred to as  $\text{PM}_{10}$ . It is now generally recognized that, except for toxic materials, it is this  $\text{PM}_{10}$  fraction of the total particulate loading that is of major significance in health effects. The reference method for  $\text{PM}_{10}$  is codified in 40CFR50, Appendix J and specifies a measurement principle based on extracting an ambient air sample with a powered sampler that incorporates inertial separation of  $\text{PM}_{10}$  size range particles and collection of these particles on a filter for a 24-hour period. Again, the sample is weighed to determine concentration of  $\text{PM}_{10}$  and is usually analyzed chemically to determine concentration of various inorganic compounds.

**1.6** Further research now strongly suggests that atmospheric particles commonly occur in two distinct modes, the fine (<2.5  $\mu\text{m}$ ) mode and the coarse (2.5 to 10.0  $\mu\text{m}$ ) mode. The fine or accumulation mode (also termed the respirable particles) is attributed to growth of particles from the gas phase and subsequent agglomeration, whereas the coarse mode is made up of mechanically abraded or ground particles. Because of their initially gaseous origin, the fine range of particle sizes includes inorganic ions such as sulfate, nitrate, and ammonium as well as combustion-form carbon, organic aerosols, metals, and other combustion products. Coarse particles, on the other hand, normally consist of finely divided minerals such as oxides of aluminum, silicon, iron, calcium, and potassium. Samplers which separate SPM into two size fractions of 0-2.5  $\mu\text{m}$  and 2.5-10  $\mu\text{m}$  are called dichotomous samplers. In 1997, the EPA promulgated a new standard with fine particles. The new  $\text{PM}_{2.5}$  standard replaced the previously NAAQS for  $\text{PM}_{10}$ .

**1.7** Airborne particulate materials retained on a sampling filter, whether TSP,  $\text{PM}_{10}$ ,  $\text{PM}_{2.5}$ , or dichotomous size fractions, may be examined by a variety of analytical methods. This method describes the procedures for XRF analysis as the analytical technique. The XRF method provides analytical procedures for determining concentration in  $\text{ng}/\text{m}^3$  for 44 elements that might be captured on typical filter materials used in fine particle or dichotomous sampling devices. With the sample as a thin layer of particles matrix effects substantially disappear so the method is applicable to elemental analysis of a broad range of particulate material. The method applies to energy dispersive XRF analysis of ambient aerosols sampled with fine particle (<2.5  $\mu\text{m}$ ) samplers, dichotomous and VAPS (versatile air pollution sampler) samplers with a 10  $\mu\text{m}$  upper cut point and  $\text{PM}_{10}$  samples.

**1.8** The analysis of ambient aerosol samples captured on filterable material should be performed by a scientist that has been trained in energy dispersive x-ray fluorescence spectroscopy and its associated data processing system. The training should be performed by a scientist with an advance degree in the physical sciences with a minimum of 5 years experience in x-ray spectroscopy.

## **2. Applicable Documents**

### **2.1 ASTM Documents**

- D4096 Application of High Volume Sample Method For Collection and Mass Determination of Airborne Particulate Matter.
- D1356 Definition of Terms Related to Atmospheric Sampling and Analysis.
- D1357 Practice For Planning the Sampling of the Ambient Atmosphere.

## 2.2 U.S. Government Documents

- U.S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: A Field Guide for Environmental Quality Assurance*, EPA-600/R-94/038a.
- U.S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition)*, EPA-600/R-94/038b.
- "Reference Method for the Determination of Particulate Matter in the Atmosphere," *Code of Federal Regulations*, 40 CFR 50, Appendix B.
- "Reference Method for the Determination of Particulate Matter in the Atmosphere (PM<sub>10</sub> Method)," *Code of Federal Regulations*, 40 CFR 50, Appendix J.
- "1978 Reference Method for the Determination of Lead in Suspended Particulate Matter Collected From Ambient Air." *Federal Register* 43 (194):46262-3.
- *Test Methods for Evaluating Solid Waste, Method 9022*, EPA Laboratory Manual, Vol. 1-A, SW-846.

## 2.3 Other Documents

- KeveX XRF TOOLBOX II Reference Manual
- KeveX 771-EDX Spectrometer User's Guide and Tutorial

## 3. Summary of Method

*[Note: This method was developed using the KeveX spectrometer. EPA has experience in the use of the KeveX spectrometer associated with various field monitoring programs involving analysis of filterable particulate matter for metals over the last two decades. The use of other manufacturers of x-ray spectrometers should work as well as long as the quality assurance and quality control specifications identified in Sections 12 through 14 of Method 10-3.3 are met. However, modifications to Compendium Method IO-3.3 procedures may be necessary if another commercial x-ray spectrometer is used.]*

The method described is x-ray fluorescence applied to PM<sub>10</sub>, fine (<2.5 μm) and coarse (2.5-10 μm) aerosols particles captured on membrane filters for research purposes in source apportionment. The samplers which collect these particles are designed to separate particles on their inertial flow characteristics producing size ranges which simplify x-ray analysis. The instrument is a commercially available KeveX EDX-771 energy dispersive x-ray spectrometer which utilizes secondary excitation from selectable targets or fluorescers and is calibrated with thin metal foils and salts for 44 chemical elements. Spectra are acquired by menu-driven procedures and stored for off-line processing. Spectral deconvolution is accomplished by a least squares algorithm which fits stored pure element library spectra and background to the sample spectrum under analysis. X-ray attenuation corrections are tailored to the fine particle layer and the discrete coarse particle fraction. Spectral interferences are corrected by a subtractive coefficient determined during calibration. The detection limits are determined by propagation of errors in which the magnitude of error from all measured quantities is calculated or estimated as appropriate. Data are reported in ng/m<sup>3</sup> for all samples. Comprehensive quality control measures are taken to provide data on a broad range of parameters, excitation conditions and elements.

**4. Significance**

**4.1** The area of toxic air pollutants has been the subject of interest and concern for many years. Recently the use of receptor models has resolved the elemental composition of atmospheric aerosol into components related to emission sources. The assessment of human health impacts resulting in major decisions on control actions by federal, state and local governments are based on these data.

**4.2** Inhalable ambient air particulate matter ( $<10\ \mu\text{m}$ ) can be collected on Teflon® filters by sampling with a dichotomous sampler and analyzed for specific metals by X-ray fluorescence. The dichotomous sampler collects particles in two size ranges - fine ( $<2.5\ \mu\text{m}$ ) and coarse ( $2.5\text{-}10\ \mu\text{m}$ ). The trace element concentrations of each fraction are determined using the nondestructive energy dispersive X-ray fluorescence spectrometer.

**4.3** The detectability and sensitivity of specific elements may vary from instrument to instrument depending upon X-ray generator frequency, multichannel analyzer sensitivity, sample interferences, etc.

## **5. Definitions**

*[Note: Definitions used in this document are consistent with ASTM Methods. All pertinent abbreviations and symbols are defined within this document at point of use.]*

**5.1 Accuracy.** The agreement between an experimentally determined value and the accepted reference value.

**5.2 Attenuation.** Reduction of amplitude or change in wave form due to energy dissipation or distance with time.

**5.3 Calibration.** The process of comparing a standard or instrument with one of greater accuracy (smaller uncertainty) for the purpose of obtaining quantitative estimates of the actual values of the standard being calibrated, the deviation of the actual value from a nominal value, or the difference between the value indicated by an instrument and the actual value.

**5.4 10  $\mu\text{m}$  Dichotomous Sampler.** An inertial sizing device that collects suspended inhalable particles ( $<10\ \mu\text{m}$ ) and separates them into coarse ( $2.5\text{-}10\ \mu\text{m}$ ) and fine ( $<2.5\ \mu\text{m}$ ) particle-size fractions.

**5.5 Emissions.** The total of substances discharged into the air from a stack, vent, or other discrete source.

**5.6 Filter.** A porous medium for collecting particulate matter.

**5.7 Fluorescent X-Rays (Fluorescent Analysis).** Characteristic X-rays excited by radiation of wavelength shorter than the corresponding absorption edge.

**5.8 Inhalable Particles.** Particles with aerodynamic diameters of  $<10\ \mu\text{m}$  which are capable of being inhaled into the human lung.

**5.9 Interference.** An undesired positive or negative output caused by a substance other than the one being measured.

**5.10 Precision.** The degree of mutual agreement between individual measurements, namely repeatability and reproducibility.

**5.11 Standard.** A concept that has been established by authority, custom, or agreement to serve as a model or rule in the measurement of quantity or the establishment of a practice or procedure.

**5.12 Traceability to NIST.** A documented procedure by which a standard is related to a more reliable standard verified by the National Institute of Standards and Technology (NIST).

**5.13 Uncertainty.** An allowance assigned to a measured value to take into account two major components of error: (1) the systematic error, and (2) the random error attributed to the imprecision of the measurement process.

**5.14 Chi-square.** A statistic which is a function of the sum of squares of the differences of the fitted and measured spectrum.

**5.15 Fluorescer.** A secondary target excited by the x-ray source and in turn excites the sample.

**5.16 FWHM.** Full width at half maximum, a measure of spectral resolution.

**5.17 NIST.** National Institute of Standards and Technology.

**5.18 Shape.** The actual shape of a background corrected pulse height spectrum for an element.

**5.19 SRMs.** Standard reference materials.

**5.20 Teflo®.** Trade name of a Teflon filter.

**5.21 Unknown.** A sample submitted for analysis whose elemental concentration is not known.

**5.22 XRF.** X-ray fluorescence.

## **6. Description of Spectrometer**

The x-ray analyzer is a KeveX EDX-771 energy dispersive spectrometer with a 200 watt rhodium target tube as an excitation source. The machine has multiple modes of excitation including direct, filtered direct, and secondary which utilizes up to 7 targets or fluorescers. To minimize radiation damage to delicate aerosol samples only the secondary mode is used. Table 1 provides a listing of the fluorescers and the elements which they excite associated with energy dispersive spectrometers. Analysis atmospheres are selectable with choices of helium, vacuum or air; helium is used for all targets except Gd where air is employed because it gives a lower background. The detector is cryogenically cooled lithium-drifted silicon with a 5  $\mu\text{m}$  Be window and a resolution of 158 eV at Fe K $\alpha$ , and comes with two manually changeable collimators. A 16 position rotating wheel accommodates the samples and provides sample changing.

The machine is operated by procedure files (or programs) written in KeveX's proprietary Job Control Language (JCL) which runs in a Windows 3.1 environment and provides setting of the analytical conditions and data acquisition. Using the JCL language, procedures have been written in-house to perform all the

functions necessary to acquire spectra and to assign to them file names in a structured manner to facilitate future spectral processing. These procedures are invoked in menu form.

## 7. Caveats

**7.1** The type of samplers mentioned in Section 1.7 must be operated in accordance with Inorganic Compendium Method IO-2.2 Sampling for Suspended Particulate Matter in Ambient Air Using a Dichotomous Sampler, or severe errors in x-ray analysis may occur. For example, errors in flow rate will not only give erroneous volumes but will cause a more serious condition of altering the cut points upon which the coarse particle x-ray attenuations are based. If samples are intended for x-ray analysis then the sampling protocol must conform to the constraints inherent within this method. Furthermore, the type of filter on which the sample is collected is very important. In general, thin membrane filters (Teflo® and Nuclepore®) are required so that the background is low and penetration of particles into the matrix of the filter is small. Thick depth filters such as quartz or glass fiber not only have high background but also allow particles to penetrate into the matrix of the filter - a condition which the spectral processing program cannot accommodate.

**7.2** Some internal contaminations consisting of Sn, Ni, Cu and Fe are present which sometimes appear in blanks. Routine analysis of blanks with samples will give the magnitude of the correction necessary to compensate for this.

**7.3** In general the elements analyzed by the Gd fluorescer have higher detection limits than the other fluorescers (see Table 2). The reason for this is due to limitations in the upper voltage limit of the x-ray tube power supply and the use of rhodium instead of a heavier element such as tungsten as a target material for the x-ray tube. As a secondary consequence of this, there are also higher detection limits for many of the elements below chromium because they overlap the elements analyzed by Gd.

**7.4** An inherent problem with a helium atmosphere is the diffusion of He through the detector window causing detector degradation and necessitating replacement. A lifetime of 3 to 4 years is expected.

**7.5** Due to an x-ray leak around the anode area of the x-ray tube the head must be shielded with additional lead cladding to prevent unwanted excitation of internal parts. This leak posed no threat to personnel but caused high background when operating at the maximum voltage. The additional shielding proved very effective at improving detection limits.

**7.6** Experience with wavelength dispersive spectrometers (WDXRF) has shown good agreement with energy dispersive instruments (EDXRF) over a broad range of elements. In spite of this agreement and the simpler spectral processing requirements of wavelength machines the preference remains with energy dispersive equipment for a variety of reasons. The very low power tubes in EDXRF machines leaves the sample intact and unaffected whereas in WDXRF the high power excitation embrittles the filter itself after 15 - 30 min exposure raising the possibility of altering particle morphology. This is a concern if electron microscopy is considered. Also, the vacuum environment, necessary for WDXRF, causes loss of some volatile materials.

**8. Sample Preparation**

**8.1** Sample preparation begins with the correct operation of the samplers employed. Inorganic Compendium Method IO-2.2, Sampling for Suspended Particulate Matter in Ambient Air Using a Dichotomous Sampler, covering the option of the samplers in the field and subsequent collection of ambient air particles on 37-mm Teflon® filter for XRF analysis. One of the greatest advantages of analyzing aerosols by XRF is that the sample can, in theory, be collected in a manner most advantageous to XRF by sampling for a duration that produces an ideal mass loading on the filter. An approximate maximum target mass is about 100 µg/cm<sup>2</sup> although much less is often collected in many environments.

**8.2** The types of filters used for aerosol sampling are 37-mm or 47-mm Teflo® with a pore size of 2 microns and, if electron microscopy is planned for the coarse fraction, then a 0.6 micron pore size Nuclepore® filter is used. The sample should be collected on the side of the Teflo® filter with the supporting ring to maintain the proper distance between the sample and detector during analysis. A properly collected sample will be a uniform deposit over the entire collection area of at least 25-mm in diameter. Samples which are not uniformly deposited over the whole collection area are not quantitatively analyzable.

**8.3** All filter samples received for analysis are removed with tweezers from their container and are checked for any invalidating conditions such as holes, tears, or a non-uniform deposit which would prevent quantitative analysis. If such a condition is found the sample is noted as invalid on the XRF data entry form; data from such samples are not reported. Teflo® filters are easily handled because of the supporting ring, however, Nuclepore® filters must have a supporting ring applied to them (after gravimetric assay) to help maintain their flatness and to securely hold them in the frame. The sample is then placed in a custom-designed commercially available two-part sample frame which snaps together holding the filter securely in place.

## **9. Spectral Acquisition and Processing**

**9.1** Spectra are acquired in sets of 15 samples each. Up to 7 spectra are acquired for each sample depending on how many secondary excitation targets are selected. Utilizing all seven fluorescers requires approximately 4 hours machine time for 44 elements analyzed plus atmospheric argon.

**9.2** Elemental intensities are determined by spectral deconvolution with a least squares algorithm which utilizes experimentally determined elemental shape functions instead of the mathematical Gaussian function. This approach has been successfully implemented for many years on an earlier machine and is described in Section 15, Citation 10. Since the spectral shape is not a pure Gaussian the experimental shapes are a more realistic representation of a spectrum. In addition to this library of elemental shape spectra there is also a background shape spectrum for each of the types of filters. It is assumed that the background on an unknown sample is due to the filter and not to the sample. (This is one of the reasons for avoiding heavily loaded filters.) The least squares algorithm synthesizes the spectrum of the sample under analysis by taking a linear combination of all the elemental shapes spectra and the background shape spectrum. The coefficients on the linear combination of elemental shapes and background spectra are scaling factors determined by minimizing chi-square thus producing the best fit possible by least square minimization. Values of the chi-square statistic are calculated for each sample and fluorescer to give an indication of the quality of the fit.

**9.3** X-ray attenuation corrections are performed as described in Section 15, Citation 10 and are briefly described here. The mass absorption coefficients for the layer of fine particles is based on a typical composition of ambient aerosol particles so the actual x-ray attenuations on a given sample are simply a function of the mass loading. Coarse particle attenuations are more complex in that they are based on x-ray

attenuation by spherical particles with compositions of common crustal minerals with various size distributions. An average attenuation and uncertainty for each coarse particle element is based on this broad range of crustal minerals and is therefore a one-time calculation giving an attenuation factor useable for all subsequent coarse (2.5-10  $\mu\text{m}$ ) particle analyses. This treatment assumes low coarse particle loading so that the particles do not shadow one another - yet another reason for assuring that the sample mass loading is not too high. Attenuation corrections on  $\text{PM}_{10}$  particles are deduced from elemental concentration data from samples taken with collocated  $\text{PM}_{10}$  and dichotomous samplers.

**9.4** The need for interference corrections arises from overlaps that are not deconvoluted by the least squares algorithm. This can best be illustrated by an example: Barium and titanium are analyzed by the gadolinium and iron fluorescers, respectively. The barium L x-rays overlap with the K x-rays of titanium and require an interference correction because the elements analyzed by gadolinium do not include titanium. The interference correction technique is described by Gilfrich in Section 15, Citation 29. The interference coefficient, determined during calibration, represents the fraction of the concentration of an affecting element (barium in the present example) which must be subtracted from the concentration of the affected element concentration (titanium) to compensate for the interference.

**9.5** When samples are collected by the dichotomous or other samplers using virtual impaction, an additional correction must be employed because these type of samplers do not perfectly separate the fine and coarse particles. Due to virtual impaction requirements, about 10% of the fine particle mass is deposited on the coarse filter. Therefore, the attenuation corrections used for the particles on the coarse filter "over-correct" the attenuation because of these residual fines on the coarse filter. These effects are compensated for by the flow fraction correction.

## **10. Data Reporting**

*[Note: In other Inorganic Compendium methods, the authors have provided detailed examples of calculations involving final metal concentration (in terms of  $\mu\text{g}/\text{m}^3$ ) from filterable materials. However, due to the nature of overlapping spectra which is characteristic of energy dispersive spectrometers, calculations are required to be performed by computer due to the complexity of the deconvolution of the recorded spectra which uses least square algorithm involving experimentally determined elemental shape functions instead of the mathematical Gaussian function. To perform by hand would require second order calculus and considerable time and manpower. Thus, the application of a computer is mandatory to determine elemental intensities and the elemental concentrations by a polynomial fit using a model based on the fundamentals of x-ray physics process (see Section 11 for further explanation).]*

The two most important data output files are an ASCII file which contains a recapitulation of the field data and the final sample concentrations in  $\text{ng}/\text{m}^3$  and a Lotus file with only the sample data. An example printout of a fine/coarse sample pair is shown in Table 3.

The uncertainty reported with each concentration is a 1F (68% confidence level) uncertainty and is determined by propagating the errors given in Section 12. Elements with concentrations below 3 times the uncertainty are flagged with an asterisk (\*) on the printed record. If the true elemental concentration is zero then the fitting procedure implies that negative and positive results are equally probable. Therefore, negative numbers may be reported.

## 11. Calibration

**11.1** Calibration is performed only when a change in fluorescers or x-ray tubes or detector is made or a serious malfunction occurs requiring significant repairs. Calibration establishes the elemental sensitivity factors and the magnitude of the interference or overlap coefficients. It takes approximately 2 weeks to complete a calibration.

**11.2** Thin film standards are used for calibration because they most closely resemble the layer of particles on a filter. There are two types of calibration standards in use. One type consists of thin films deposited on Nuclepore substrates (Micromatter Co., Eastsound, WA). These standards are available for almost all the elements analyzed ranging in atomic number from 11 (Na) to 82 (Pb) with deposit masses gravimetrically determined to  $\pm 5\%$ . Another type consists of polymer films that contain known amounts of two elements in the form of organo-metallic compounds dissolved in a polymer and are not commercially available but their preparation is described in Section 15, Citation 9. These standards have been prepared for elements with atomic numbers above 21 (titanium and heavier). The same set of standards is used every time the spectrometer is calibrated. The standards are sufficiently durable to last many years, however occasionally one must be replaced due to accidents in handling. Approximately 200 calibration standards for 44 elements are in use (see Table 4.) and the acquisition of their spectra requires several days.

**11.3** The background files which are used for background fitting are created at calibration time. Thirty clean Teflo® and Nuclepore® blanks are kept sealed in a plastic bag and are used exclusively for background measurement. After acquiring spectra for all 7 fluorescers the spectra are added together to produce a single spectrum for each fluorescer. Options are available to omit a spectrum from the sum if one shows a contamination. It is these summed spectra that are fitted to the background during spectral processing.

**11.4** The shape standards are thin film standards consisting of ultra pure elemental materials for the purpose of determining the physical shape of the pulse height spectrum. For this purpose it is not necessary for the concentration of the standard to be known - only that it be pure. A slight contaminant in the region of interest in a shape standard can have serious effect on the ability of the least squares fitting algorithm to fit the shapes to the unknown. For this reason the Se and elemental As standards, whose compounds are volatile, are kept in separate plastic bags in a freezer to prevent contamination of other standards; the Au standard, which will slowly amalgamate with atmospheric Hg, is kept in a desiccator. The shape standards are acquired for sufficiently long times to provide a large number of counts in the peaks of interest. It is these elemental shape spectra that are fitted to the peaks in an unknown sample during spectral processing.

**11.5** The spectra from the calibration standards are deconvoluted to get elemental intensities as described in Section 9.2. Using these intensities and the elemental concentration in the standards the sensitivities are determined by a polynomial fit using a model based on the fundamentals of the x-ray physics process as well as measurements on the calibration standards. This approach allows the calculation of sensitivities for elements for which there are poor or no standards such as volatile ones like Se and elemental As as well as improving on elements with good standards.

**11.6** The overlap coefficients are determined during calibration and represent the extent of interference that exists between overlapping spectral peaks. During calibration an affecting element (barium, to continue with the example of Section 9.4) is measured both at the analyte line peak for barium and at the titanium peak. The coefficient is expressed as the ratio of the concentration of the affected element (titanium) to the

concentration of the affecting element (barium). All elements requiring overlap coefficient determination are calculated in this manner.

## 12. Detection Limits

The detection limits are determined by propagation of errors. The sources of random error which are considered are: calibration uncertainty ( $\pm 5\%$ ); long term system stability ( $\pm 5\%$ ); peak and background counting statistics; uncertainty in attenuation corrections; uncertainty in overlap corrections; uncertainty in flow rate; and uncertainty in coarse fraction due to flow fraction correction (paired samples only). Table 2 outlines typical 1F (68% confidence level) detection limits on a Teflo® blank for fine particles and a Nuclepore® blank for coarse (2.5  $\mu\text{m}$ -10  $\mu\text{m}$ ) particles. These detection limits are defined in terms of the uncertainty in the blank. This ignores the effect of other elements which generally is small except for the light elements (potassium and lower) where overlapping spectral lines will increase the detection limit.

*[Note: The difference in the detection limits between the two filters in Table 2 is due more to the difference in sensitivity to fine and coarse particles and less to the difference in filter material.]*

Higher confidence levels may be chosen for the detection limits by multiplying the 1F limits by 2 for a 2F (or 95% level) or by 3 for 3F (or 99.7% level). To convert the detection limits to more useful units one can use the typical deposit areas for 37-mm and 47-mm diameter filters of 6.5  $\text{cm}^2$  and 12.0  $\text{cm}^2$  respectively.

## 13. Quality Control

**13.1** A comprehensive quality control program is in effect consisting of many measured parameters covering all measurement conditions and automatically produces control charts for all such measurements. All plotted data are normalized to the mean to give a rapid assessment of relative change.

**13.2** Run-time quality control gives an indication of instrument performance at the time of data acquisition by measurements on stable qualitative standards. The parameters which are measured and their significance are: peak areas (monitors change in sensitivity), background areas (monitors contamination or background changes), centroid (monitors gain and zero adjustment to insure that spectra are assigned the correct channel), and FWHM, (monitors degradation of the detector resolution). These four parameters are measured for elements ranging from sodium to lead and include atmospheric argon. An example of plots of run-time QC data are illustrated in Figures 1 through 4 and Table 5, for the target and tolerance values for the parameters measured.

**13.3** In addition to the run-time quality control procedure the analysis results of Standard Reference Materials SRM1833 and SRM1832 are included in the data reports. These results provide an overall check of the spectral processing program for the elements which are certified in the standards. The sole purpose of the SRMs is to provide a quality control measure; the standards are not used for calibration. Typical results of these SRMs are documented in Tables 6 and 7, and plotted in Figure 5.

**13.4** The run-time quality control procedures serve as an indicator of possible emerging problems by flagging deviations greater than 3 tolerance units as defined for each element in Table 5. Persistently increasing trends are investigated to determine their cause(s) before they impact the results of SRM analyses.

**13.5** The acceptance criteria of results for the elements certified in the SRMs is that the uncertainty intervals for the analytical results and those of the certified values should overlap each other. If any element fails this then the run of unknowns is repeated. Repeated failures indicate the need for recalibration.

**13.6** A value for chi-square is calculated and reported with the data to indicate the quality of the fit. Chi-square values that are much larger than 1.0 indicate a problem in the fitting procedure. Changes in detector resolution or gain in the amplifier produce large values for chi-square; however such changes would be detected by the run-time quality control procedure. Also, large chi-square values can accompany results for heavily loaded filters even though the relative errors are typical. In addition, elements analyzed by the titanium and the iron fluorescers may experience large chi-square values due to interferences from overlapping elements. Chi-square is a more useful measure of goodness-of-fit for the other fluorescers for this reason.

**13.7** To acquire more information about fitting problems the fitted spectra can be viewed on the screen or a hard copy printed. Such plots can be compared to the unknown spectra, background spectra, or to the library shape standards to help elucidate the suspected problem. Various statistics such as the correlation coefficient can be calculated on the fitted and measured spectra as a additional measure of the goodness-of-fit. Fitted spectrum superposed on its measured spectrum along with the associated statistics is illustrated in Figure 6.

#### 14. Precision and Accuracy

Precision varies with the element and concentration. At high concentrations (greater than 1  $\mu\text{g}/\text{cm}^2$ ) a precision of 7.1% can be expected for elements analyzed by one fluorescer and 5.0% can be expected for those analyzed by two. Refer to Table 1 for a listing of the elements and the fluorescers which analyze them. Based upon the analysis of NIST SRMs the accuracy is  $\pm 10\%$ .

#### 15. References

1. Arinc, F., Wielopolski, L., and Gardner, R. P., *The Linear Least-Squares Analysis of X-Ray Fluorescence Spectra of Aerosol Samples using Pure Element Library Standards and Photon Excitation*, X-ray Fluorescence Analysis of Environmental Samples, ed. T. G. Dzuby, Ann Arbor Science, Ann Arbor, MI p.227.
2. Bevington, P.R., *Data Reduction and Error Analysis for the Physical Sciences*, McGrawHill Book Co., New York, N.Y.
3. Billiet, J., Dams, R., and Hoste, J., X-Ray Spectrum, 9:206-211, 1980.
4. Birks, L.S., and Gilfrich, J.V., "X-Ray Spectrometry," *Anal. Chem.*, 48:273R-28R, 1976.
5. Dzuby, T. G., *Analysis of Aerosol Samples by X-Ray Fluorescence*, Env. Science Research Lab, U. S. Environmental Protection Agency, Research Triangle Park, NC, 27711, April 1986.
6. Dzuby, T. G., "Chemical Elements Balance Method Applied to Dichotomous Sampler Data," *Annals New York Academy of Sciences*, 1980.

7. Dzubay, T. G., *Development and Evaluation of Composite Receptor Methods*, EPA Project Summary, EPA-600/3-88-026, U. S. Environmental Protection Agency, Research Triangle Park, NC, 27711, September 1988.
8. Dzubay, T. G., *Quality Assurance Narrative for SARB XRF Facility*, Env. Science Research Lab, U. S. Environmental Protection Agency, Research Triangle Park, NC, 27711, March 1989.
9. Dzubay, T. G., et al., "Polymer Film Standards for X-Ray Fluorescence Spectrometers," *J. Trace and Microprobe Techniques*, 5(4):327-341, 1987-88.
10. Dzubay, T. G., Drane, E. A., Rickel, D. G., and Courtney, W. J., "Computer Code for Analyzing X-Ray Fluorescence Spectra of Airborne particulate Matter," *Advances in X-Ray Analysis*, Vol. 23.
11. Dzubay, T. G., Lamothe, P. J., and Yasuda, H., *Advances in X-Ray Analysis*, ed. H. F. McMurdie, C.S. Barrett, J. B. Newkirk, and C. O. Ruud, Plenum, New York, N.Y. 20:411-421, 1977.
12. Dzubay, T. G., Morosoff, N., Whitaker, G. L., et al., "Evaluation of Polymer Films as Standards for X-Ray Fluorescence Spectrometers," Presented at Symposium on Electron Microscopy and X-Ray Applications to Environmental and Occupational Health Analysis.
13. Dzubay, T. G., and Nelson, R. O., *Self Absorption Corrections for X-Ray Fluorescence Analysis of Aerosols*, *Advances in X-Ray Analysis*, ed. W. L. Pickles, et al., Plenum Publishing Corp., New York, N.Y. 18:619, 1975.
14. Dzubay, T. G., and Rickel, D. G., *X-Ray Fluorescence Analysis of Filter-Collected Aerosol Particles*, *Electron Microscopy and X-Ray Applications*, Ann Arbor Science, 1978.
15. Dzubay, T. G., Stevens, R. K., Gordon, G. E., Olmez, I., Sheffield, A. E., and Courtney, W. J., "A Composite Receptor Method Applied to Philadelphia Aerosol," *Environmental Science & Technology*, 22:46, January 1988.
16. Dzubay, T. G., Stevens, R. K., Gordon, G. E., Olmez, I., Sheffield, A. E., and Courtney, W. J., "A Composite Receptor Method Applied to Philadelphia Aerosol," *Environmental Science & Technology*, 22:46, January 1988.
17. Giauque, R. D., Goulding, F. S., Jaklevic, J. M., and Pehl, R. H., "Trace Element Determination with Semiconductor Detector X-Ray Spectrometers," *Anal. Chem.*, 45:671, 1973.
18. Goulding, F. S., and Jaklevic, J. M., *Fabrication of Monitoring System for Determining Mass and Composition of Aerosol as a Function of Time*, EPA-650/2-75-045, U. S. Environmental Protection Agency, Research Triangle Park, NC 27711, 1975.
19. *Inhalable Particulate Network Operations and Quality Assurance Manual*, Office of Research and Development, Env. Monitoring Systems Lab, U. S. Environmental Protection Agency, Research Triangle Park, NC 27711.
20. Jaklevic, J. M., Landis, D. A., and Goulding, F. S., "Energy Dispersive X-Ray Fluorescence Spectrometry Using Pulsed X-Ray Excitation," *Advances in X-Ray Analysis*, 19:253-265, 1976.

21. Jenkins, R., and deVries, J. L., *Practical X-Ray Spectroscopy*, Springer-Verlag, New York, NY, 1967.
22. Loo, B. W., Gatti, R. C., Liu, B. Y. H., Chong-Soong, K., and Dzubay, T. G., "Absorption Corrections for Submicron Sulfur Collected in Filters," *X-Ray Fluorescence Analysis of Environmental Samples*, Ann Arbor Science, Ann Arbor, MI p. 187, 1977.
23. NBS Standard Reference materials Catalog 1986-87, National Bureau of Standards Publ. 260, U. S. Department of Commerce, Washington, DC p. 64, June 1986.
24. Rhodes, J. R., *X-Ray Spectrom.*, 6:171-173, 1977.
25. Wagman, J., Bennett, R. L., and Knapp, K. T., "Simultaneous Multiwavelength Spectrometer for Rapid Elemental Analysis of Particulate Pollutants," *X-Ray Fluorescence Analysis of Environmental Samples*, ed. T. G. Dzubay, Ann Arbor Science Publishers, Inc. Ann Arbor, MI, pp, 35-55, 1977.
26. *Volume II: Protocols for Environmental Sampling and Analysis*, Particle Total Exposure Assessment Methodology (P-Team): Pre-Pilot Study, EPA 68-02-4544, U. S. Environmental Protection Agency, Research Triangle Park, NC 27709, January 27, 1989.
27. Jaklevic, et al., *X-ray Fluorescence Analysis of Environmental Samples*, ed. T. G. Dzubay, Ann Arbor Science, Ann Arbor, MI, p. 63.
28. Cooper, J. A., Valdovinos, L. M., Sherman, J. R., Pollard, W. L., Sarver, R. H., and Weilder, J. K., "Quantitative Analysis of Aluminum and Silicon in Air Particulate Deposits on Teflon® Membrane Filters by X-ray Fluorescence Analysis," NEA, Inc., Beaverton, OR, July 15, 1987.
29. Gilfrich, et al., *X-ray Fluorescence Analysis of Environmental Samples*, ed. T. G. Dzubay, Ann Arbor Science, Ann Arbor, MI, p. 283.

TABLE 1. EXAMPLE OF FLUORESCER USAGE

| Element | Fluorescer |    |    |    |    |    | Gd |
|---------|------------|----|----|----|----|----|----|
|         | Al         | Ti | Fe | Ge | Ag | Zr |    |
| Na      | x          |    |    |    |    |    |    |
| Mg      | x          |    |    |    |    |    |    |
| Al      | x          |    |    |    |    |    |    |
| Si      | x          |    |    |    |    |    |    |
| P       | x          | x  |    |    |    |    |    |
| S       | x          | x  |    |    |    |    |    |
| Cl      | x          | x  |    |    |    |    |    |
| Ar      | x          | x  |    |    |    |    |    |
| K       | x          | x  |    |    |    |    |    |
| Ca      | x          | x  |    |    |    |    |    |
| Sc      | x          | x  |    |    |    |    |    |
| Ti      |            |    | x  | x  |    |    |    |
| V       |            |    | x  | x  |    |    |    |
| Cr      |            |    | x  | x  |    |    |    |
| Mn      |            |    |    | x  |    | x  |    |
| Fe      |            |    |    | x  |    | x  |    |
| Co      |            |    |    | x  |    | x  |    |
| Ni      |            |    |    | x  |    | x  |    |
| Cu      |            |    |    | x  |    | x  |    |
| Zn      |            |    |    |    |    | x  |    |
| Ga      |            |    |    |    |    | x  |    |
| Ge      |            |    |    |    |    | x  |    |
| As      |            |    |    |    |    | x  |    |
| Se      |            |    |    |    |    | x  |    |
| Br      |            |    |    |    |    | x  |    |
| Rb      |            |    |    |    | x  | x  |    |
| Sr      |            |    |    |    | x  |    | x  |
| Y       |            |    |    |    | x  |    |    |
| Zr      |            |    |    |    | x  |    | x  |
| Mo      |            |    |    |    | x  |    | x  |
| Rh      |            |    |    |    |    |    | x  |
| Pd      |            |    |    |    |    |    | x  |
| Ag      |            |    |    |    |    |    | x  |
| Cd      |            |    |    |    |    |    | x  |
| Sn      |            |    |    |    |    |    | x  |
| Sb      |            |    |    |    |    |    | x  |
| Te      |            |    |    |    |    |    | x  |
| I       |            |    |    |    |    |    | x  |
| Cs      |            |    |    |    |    |    | x  |
| Ba      |            |    |    |    |    |    | x  |
| La      |            |    |    |    |    |    | x  |
| W       |            |    |    |    | x  | x  |    |
| Au      |            |    |    |    | x  | x  |    |
| Hg      |            |    |    |    | x  | x  |    |
| Pb      |            |    |    |    | x  | x  |    |

[Note: The 'x' marks the fluorescers that analyze each element.]

TABLE 2. METHOD DETECTION LIMITS (MDL) FOR  
TEFLO® AND NUCLEPORE® BLANK FILTERS (1F)

| Teflo® - fine element<br>Method Detection Limits (MDL) |                    |                                | Nuclepore® - coarse element<br>Method Detection Limits (MDL) |                    |                                |
|--|--------------------|--------------------------------|--|--------------------|--------------------------------|
|  | ng/cm <sup>2</sup> | ng/m <sup>3</sup> <sup>1</sup> |  | ng/cm <sup>2</sup> | ng/m <sup>3</sup> <sup>2</sup> |
| Na   | 5.3                | 1.59                           | Na   | 17.4               | 47.12                          |
| Mg   | 3.2                | 0.96                           | Mg   | 7.9                | 21.34                          |
| Al   | 17.6               | 5.29                           | Al   | 46.7               | 126.48                         |
| Si   | 8.0                | 2.41                           | Si   | 21.2               | 50.40                          |
| P  | 2.6                | 0.78                           | P  | 4.1                | 11.10                          |
| S  | 2.6                | 0.78                           | S  | 6.9                | 16.56                          |
| Cl   | 4.8                | 1.44                           | Cl   | 5.6                | 13.44                          |
| K  | 6.3                | 1.89                           | K  | 5.6                | 15.17                          |
| Ca   | 9.0                | 2.71                           | Ca   | 8.7                | 23.56                          |
| Sc   | 1.5                | 0.45                           | Sc   | 1.3                | 3.52                           |
| Ti   | 16.9               | 5.08                           | Ti   | 18.7               | 42.52                          |
| V  | 5.3                | 1.59                           | V  | 5.5                | 14.89                          |
| Cr   | 3.0                | 0.90                           | Cr   | 3.0                | 8.12                           |
| Mn   | .8                 | 0.24                           | Mn   | .8                 | 2.17                           |
| Fe   | .7                 | 0.21                           | Fe   | 1.0                | 2.71                           |
| Co   | .4                 | 0.12                           | Co   | .4                 | 1.08                           |
| Ni   | .6                 | 0.18                           | Ni   | .7                 | 1.89                           |
| Cu   | .7                 | 0.21                           | Cu   | .8                 | 2.17                           |
| Zn   | 1.0                | 0.30                           | Zn   | 1.1                | 2.98                           |
| Ga   | 1.6                | 0.48                           | Ga   | 1.5                | 4.06                           |
| Ge   | 1.1                | 0.33                           | Ge   | 1.0                | 2.71                           |
| As   | .8                 | 0.24                           | As   | .9                 | 2.44                           |
| Se   | .7                 | 0.21                           | Se   | .6                 | 1.62                           |
| Br   | .6                 | 0.18                           | Br   | .7                 | 1.89                           |
| Rb   | .7                 | 0.21                           | Rb   | .7                 | 1.89                           |
| Sr   | 1.1                | 0.33                           | Sr   | .9                 | 2.44                           |
| Y  | 1.2                | 0.36                           | Y  | 1.1                | 2.98                           |
| Zr   | 1.2                | 0.36                           | Zr   | 1.1                | 2.98                           |
| Mo   | 1.6                | 0.48                           | Mo   | 1.5                | 4.06                           |
| Rh   | 25.9               | 7.79                           | Rh   | 26.5               | 71.70                          |
| Pd   | 22.9               | 6.89                           | Pd   | 18.7               | 50.65                          |
| Ag   | 20.2               | 6.02                           | Ag   | 20.3               | 54.98                          |
| Cd   | 22.0               | 6.62                           | Cd   | 19.2               | 52.00                          |
| Sn   | 30.5               | 9.18                           | Sn   | 31.5               | 85.31                          |
| Sb   | 31.4               | 9.45                           | Sb   | 26.7               | 72.31                          |
| Te   | 26.3               | 7.91                           | Te   | 27.6               | 66.62                          |
| I  | 35.5               | 10.68                          | I  | 34.4               | 93.17                          |
| Cs   | 48.9               | 14.62                          | Cs   | 50.9               | 137.85                         |
| Ba   | 51.8               | 15.59                          | Ba   | 58.3               | 157.89                         |
| La   | 70.6               | 2.12                           | La   | 68.9               | 186.60                         |
| W  | 3.4                | 10.23                          | W  | 3.3                | 8.93                           |
| Au   | 1.7                | 0.51                           | Au   | 1.5                | 4.06                           |
| Hg   | 1.5                | 0.45                           | Hg   | 1.4                | 3.79                           |
| Pb   | 1.5                | 0.45                           | Pb   | 1.5                | 4.06                           |

<sup>1</sup>Based upon dichotomous sampling for 24-hrs. using a 37-mm Teflo® filter at a sampling rate of 0.9 m<sup>3</sup>/hr.<sup>2</sup>Based upon dichotomous sampling for 24-hrs using a 37-mm Nuclepore® filter at a sampling rate of 0.1 m<sup>3</sup>/hr.

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TABLE 3. DATA REPORT FORMAT FOR A FINE/COARSE PAIRED SAMPLE

KEVEX SUMMARY: ADOBE FLATS URBAN PARTICULATE STUDY

|                |        |                         |              |        |                           |
|----------------|--------|-------------------------|--------------|--------|---------------------------|
| SITE           | =      | ADB                     | SAMPLE DATE  | =      | 3/20/92 AND 1900 HOURS    |
| DURATION (MIN) | =      | 714.0                   | FLOW (L/MIN) | =      | 37.105 +- .500            |
| FLOW FRAC      | =      | .0869                   | XRF ID       | =      | 999956                    |
| XRF ID         | =      | 999906                  | SAMPLE ID    | =      | NU0033                    |
| SAMPLE ID      | =      | T0033                   |              |        |                           |
|                |        | FINE, NG/M <sup>3</sup> |              |        | COARSE, NG/M <sup>3</sup> |
| MASS           | 77912. | +- 1962.                | MASS         | 11347. | +- 812.                   |
| *NA            | 211.9  | +- 71.4                 | *NA          | 53.3   | +- 27.1                   |
| MG             | 564.6  | +- 89.4                 | MG           | 443.9  | +- 40.8                   |
| *AL            | 162.2  | +- 74.1                 | AL           | 539.9  | +- 173.8                  |
| SI             | 213.4  | +- 40.4                 | SI           | 909.5  | +- 232.7                  |
| * P            | 12.1   | +- 18.5                 | * P          | -5.5   | +- 11.3                   |
| S              | 2653.4 | +- 183.7                | S            | 285.7  | +- 84.9                   |
| CL             | 1164.4 | +- 79.3                 | *CL          | 34.8   | +- 24.6                   |
| K              | 193.6  | +- 13.8                 | K            | 63.5   | +- 8.9                    |
| CA             | 43.4   | +- 5.6                  | CA           | 181.7  | +- 13.9                   |
| *SC            | 3.6    | +- 4.1                  | *SC          | -1.3   | +- 2.2                    |
| *TI            | 17.6   | +- 6.6                  | TI           | 54.7   | +- 9.6                    |
| * V            | 4.6    | +- 2.3                  | * V          | 3.2    | +- 1.7                    |
| *CR            | 2.0    | +- 1.0                  | CR           | 9.8    | +- 1.6                    |
| MN             | 10.0   | +- 1.4                  | MN           | 10.1   | +- 1.3                    |
| FE             | 243.7  | +- 21.9                 | FE           | 783.5  | +- 78.2                   |
| *CO            | 2.8    | +- 1.8                  | *CO          | 4.8    | +- 1.7                    |
| NI             | 3.8    | +- 1.2                  | *NI          | .3     | +- .6                     |
| CU             | 14.3   | +- 1.9                  | CU           | 8.8    | +- 1.3                    |
| ZN             | 167.5  | +- 14.9                 | ZN           | 27.6   | +- 4.9                    |
| *GA            | 2.4    | +- 1.0                  | *GA          | -.0    | +- .4                     |
| *GE            | 3.3    | +- 1.3                  | *GE          | .0     | +- .6                     |
| AS             | 24.7   | +- 3.6                  | *AS          | 1.8    | +- 1.2                    |
| SE             | 4.7    | +- .8                   | *SE          | .7     | +- .4                     |
| BR             | 29.0   | +- 2.8                  | BR           | 7.9    | +- 1.1                    |
| *RB            | 1.7    | +- .8                   | *RB          | 1.0    | +- .4                     |
| SR             | 2.9    | +- .9                   | SR           | 2.2    | +- .5                     |
| * Y            | 12.4   | +- 6.1                  | * Y          | 3.9    | +- 2.9                    |
| *ZR            | 2.9    | +- 4.8                  | *ZR          | 4.3    | +- 2.6                    |
| *MO            | 7.3    | +- 4.8                  | *MO          | -3.2   | +- 2.2                    |
| *RH            | .0     | +- 3.2                  | *RH          | -1.2   | +- 1.6                    |
| *PD            | -3.6   | +- 3.1                  | *PD          | -1.0   | +- 1.7                    |
| *AG            | -6.4   | +- 3.4                  | *AG          | 1.2    | +- 1.9                    |
| *CD            | 8.5    | +- 4.5                  | *CD          | -.7    | +- 2.2                    |
| SN             | 54.3   | +- 9.4                  | *SN          | 2.3    | +- 3.9                    |
| *SB            | -1.6   | +- 6.4                  | *SB          | -.6    | +- 3.3                    |
| *TE            | 2.5    | +- 7.5                  | *TE          | -7.2   | +- 3.8                    |
| * I            | 25.0   | +- 9.6                  | * I          | 2.4    | +- 4.7                    |
| *CS            | -4.0   | +- 11.2                 | *CS          | 12.4   | +- 5.9                    |
| *BA            | -7.7   | +- 13.7                 | BA           | 25.1   | +- 7.4                    |
| *LA            | -4.8   | +- 34.5                 | *LA          | 22.6   | +- 17.9                   |
| * W            | -1.1   | +- 2.6                  | * W          | 1.5    | +- 1.3                    |

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|     |          |      |     |         |     |
|-----|----------|------|-----|---------|-----|
| *AU | -.9 +-   | 1.8  | *AU | .2 +-   | .9  |
| *HG | -.4 +-   | 1.9  | *HG | 1.5 +-  | 1.0 |
| PB  | 221.6 +- | 19.7 | PB  | 46.0 +- | 6.2 |

\* INDICATES THAT THE CONCENTRATION IS BELOW 3 TIMES THE UNCERTAINTY.  
XRF DATE= 04/29/1992 16:35 RBK (F): 04/29/1992 20:35 RBK (C)  
SPECTRAL ANALYSIS DATE= 5/20/1992

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TABLE 4. CALIBRATION STANDARDS AND CONCENTRATIONS

| Standard ID | Element | µg/cm <sup>2</sup> |
|-------------|---------|--------------------|-------------|---------|--------------------|-------------|---------|--------------------|-------------|---------|--------------------|
| CaF237      | F       | 18.00              | CuS1124     | S       | 31.90              | Cr 85       | Cr      | 85.00              | RbNO311     | Rb      | 69.00              |
| CaF2 29     | F       | 14.10              | CuS58.6     | S       | 16.50              | Cr 84       | Cr      | 84.00              | RbNO322     | Rb      | 12.90              |
| CaF2 90     | F       | 43.80              | CuS57.6     | S       | 13.90              | Cr 75       | Cr      | 75.00              | RbNO3 a     | Rb      | 24.90              |
| CaF2 91     | F       | 44.30              | CuS58.2     | S       | 14.00              | Cr 74       | Cr      | 74.00              | RbNO3 b     | Rb      | 24.90              |
| CaF2102     | F       | 49.60              | NaCl 57     | Cl      | 34.60              | Cr 122      | Cr      | 122.00             | RbNO3 c     | Rb      | 24.90              |
| CaF2 66     | F       | 32.10              | NaCl 87     | Cl      | 52.80              | CrCu32a     | Cr      | 9.19               | SrF2 57     | Sr      | 39.80              |
| CaF2 28     | F       | 13.60              | NaCl446     | Cl      | 27.10              | CrCu26g     | Cr      | 8.14               | SbSr29z     | Sr      | 4.97               |
| CaF2 33     | F       | 16.10              | NaCl1715    | Cl      | 43.40              | MnZn24b     | Mn      | 8.57               | SrF2 50     | Sr      | 34.90              |
| CaF2 39     | F       | 19.00              | NaCl497     | Cl      | 30.20              | Mn 57       | Mn      | 57.00              | SbSr31y     | Sr      | 5.14               |
| CaF2 54     | F       | 26.30              | NaCl501     | Cl      | 30.40              | Mn 183      | Mn      | 183.00             | SrF2137     | Sr      | 95.60              |
| CaF2291     | F       | 14.10              | NaCl 51     | Cl      | 31.00              | MnZn27x     | Mn      | 9.10               | SrF2184     | Sr      | 12.80              |
| CaF2 30     | F       | 14.60              | NaCl512     | Cl      | 31.10              | Mn 43       | Mn      | 43.00              | SrF2 92     | Sr      | 64.20              |
| CaF2 52     | F       | 25.30              | NaCl519     | Cl      | 31.50              | Mn 46.9     | Mn      | 46.90              | SrF2103     | Sr      | 71.80              |
| CaF2 48     | F       | 23.40              | KCl 45      | Cl      | 21.40              | Mn 44.5     | Mn      | 44.50              | YF3 46      | Y       | 28.00              |
| CaF2 45     | F       | 21.90              | KCl53.3     | Cl      | 25.40              | Mn 46.6     | Mn      | 46.60              | ZrCd24c     | Zr      | 9.85               |
| CaF2 36     | F       | 17.50              | KCl 70      | Cl      | 33.30              | Mn 43.7     | Mn      | 43.70              | ZrCd20w     | Zr      | 10.77              |
| CaF2134     | F       | 65.20              | KCl 49      | Cl      | 23.30              | Mn 69       | Mn      | 69.00              | MoO3145     | Mo      | 96.70              |
| CaF2110     | F       | 53.50              | KCl48.7     | Cl      | 23.20              | FePb37y     | Fe      | 7.72               | MoO3106     | Mo      | 70.70              |
| NaCl 57     | Na      | 22.40              | KCl47.9     | Cl      | 22.80              | Fe 107      | Fe      | 107.00             | MoO3110     | Mo      | 73.30              |
| NaCl 87     | Na      | 34.20              | KCl 48      | Cl      | 22.80              | Fe 127      | Fe      | 127.00             | MoO3 59     | Mo      | 39.30              |
| NaCl446     | Na      | 17.60              | KCl47.6     | Cl      | 22.60              | Fe 46       | Fe      | 46.00              | MoO3 54     | Mo      | 36.00              |
| NaCl1715    | Na      | 28.10              | KCl 45      | K       | 23.60              | Fe 88       | Fe      | 88.00              | Rh 16       | Rh      | 16.00              |
| NaCl497     | Na      | 19.60              | KCl53.3     | K       | 28.00              | FePb38y     | Fe      | 7.71               | Pd 33       | Pd      | 33.00              |
| NaCl501     | Na      | 19.70              | KCl 70      | K       | 36.70              | Co 45a      | Co      | 45.00              | Pd 198      | Pd      | 198.00             |
| NaCl 51     | Na      | 20.10              | KCl 49      | K       | 25.70              | Co 45b      | Co      | 45.00              | Ag 35       | Ag      | 35.00              |
| NaCl512     | Na      | 20.10              | KCl48.7     | K       | 25.50              | RbCo29c     | Co      | 7.43               | Ag 132      | Ag      | 132.00             |
| NaCl519     | Na      | 20.40              | KCl47.9     | K       | 25.10              | RbCo25b     | Co      | 7.65               | Cd 83       | Cd      | 83.00              |
| Mg 81       | Mg      | 81.00              | KCl 48      | K       | 25.20              | Ni 54       | Ni      | 54.00              | ZrCd20w     | Cd      | 9.15               |
| Mg 41       | Mg      | 41.00              | KCl47.6     | K       | 25.00              | Ni 88       | Ni      | 88.00              | ZrCd24c     | Cd      | 8.38               |
| Mg 41.3     | Mg      | 41.30              | CaF2 37     | Ca      | 19.00              | NiV 21c     | Ni      | 5.77               | Cd 77       | Cd      | 77.00              |
| Mg 43       | Mg      | 43.00              | CaF2 29     | Ca      | 14.90              | Ni 101      | Ni      | 101.00             | Sn 40       | Sn      | 40.00              |
| Mg 43.8     | Mg      | 43.80              | CaF2 90     | Ca      | 46.20              | Cu 96       | Cu      | 96.00              | Sn 185      | Sn      | 185.00             |
| Mg 60.2     | Mg      | 60.20              | CaF2 91     | Ca      | 46.70              | Cu 104      | Cu      | 104.00             | Sn 97a      | Sn      | 97.00              |
| Al 57       | Al      | 57.00              | CaF2102     | Ca      | 52.40              | Cu 128      | Cu      | 128.00             | Sn 97b      | Sn      | 97.00              |
| Al 37.9     | Al      | 37.90              | CaF2 66     | Ca      | 33.90              | CrCu26g     | Cu      | 7.65               | Sn 79       | Sn      | 79.00              |
| Al 37.4     | Al      | 37.40              | CaF2 28     | Ca      | 14.40              | CrCu32a     | Cu      | 8.63               | Sb 194      | Sb      | 194.00             |
| Al 29       | Al      | 29.00              | CaF2 33     | Ca      | 16.90              | Cu 38       | Cu      | 38.00              | Sb 47       | Sb      | 47.00              |
| Al 43.2     | Al      | 43.20              | CaF2 39     | Ca      | 20.00              | Zn 51       | Zn      | 51.00              | Sb 147      | Sb      | 147.00             |
| Al 62       | Al      | 62.00              | CaF2 54     | Ca      | 27.20              | Zn 125      | Zn      | 125.00             | Sb 42       | Sb      | 42.00              |
| Al 75       | Al      | 75.00              | CaF2291     | Ca      | 14.90              | MnZn27x     | Zn      | 8.46               | SbSr29z     | Sb      | 5.01               |
| SiO 46      | Si      | 29.30              | CaF2 30     | Ca      | 15.40              | MnZn24b     | Zn      | 7.97               | SbSr31y     | Sb      | 5.18               |
| SiO 47      | Si      | 29.90              | CaF2 52     | Ca      | 26.70              | GaP 34      | Ga      | 23.50              | Te 53       | Te      | 53.00              |
| SiO 51a     | Si      | 32.50              | CaF2 48     | Ca      | 24.60              | GaP 40      | Ga      | 27.70              | KI 46       | I       | 35.20              |
| SiO 51b     | Si      | 32.50              | CaF2 45     | Ca      | 23.10              | GaP 70      | Ga      | 48.50              | CsBr 53     | Cs      | 33.10              |
| SiO 56      | Si      | 35.70              | CaF2 36     | Ca      | 18.50              | GaP 105     | Ga      | 72.70              | CsBr 54     | Cs      | 33.70              |
| SiO 80      | Si      | 51.00              | CaF2134     | Ca      | 68.60              | Ge 37       | Ge      | 37.00              | CsBr 51     | Cs      | 31.90              |
| SiO27.6     | Si      | 17.60              | CaF2110     | Ca      | 56.50              | TiGe33d     | Ge      | 6.22               | BaF2108     | Ba      | 84.60              |
| SiO46.1     | Si      | 29.40              | ScF3 57     | Sc      | 25.10              | TiGe29x     | Ge      | 5.94               | BaF2 48     | Ba      | 37.60              |
| SiO72.2     | Si      | 46.00              | Ti 39       | Ti      | 39.00              | Ge 140      | Ge      | 140.00             | BaF2 60     | Ba      | 47.00              |
| GaP 34      | P       | 10.50              | Ti 95       | Ti      | 95.00              | BaAs23y     | As      | 5.60               | BaF2 57     | Ba      | 44.70              |
| GaP 40      | P       | 12.30              | TiGe33d     | Ti      | 2.46               | BaAs36w     | As      | 5.52               | BaF2143     | Ba      | 112.00             |
| GaP 70      | P       | 21.50              | TiGe29x     | Ti      | 2.36               | CsBr 53     | Br      | 19.90              | BaF2114     | Ba      | 89.40              |
| GaP 105     | P       | 32.30              | V 45        | V       | 45.00              | CsBr 54     | Br      | 20.30              | BaAs23y     | Ba      | 4.98               |

TABLE 4. (continued)

| Standard ID | Element | $\mu\text{g}/\text{cm}^2$ |
|-------------|---------|---------------------------|-------------|---------|---------------------------|-------------|---------|---------------------------|-------------|---------|---------------------------|
| CuS1052     | S       | 30.80                     | V 53        | V       | 53.00                     | CsBr 51     | Br      | 19.10                     | BaAs36w     | Ba      | 4.91                      |
| CuS 48      | S       | 13.00                     | NiV 21c     | V       | 6.64                      | RbNO346     | Rb      | 26.60                     | LaF3157     | La      | 111.30                    |
| CuS 136     | S       | 33.00                     | Cr 30       | Cr      | 30.00                     | RbCo25b     | Rb      | 7.88                      | LaF3 62     | La      | 44.00                     |
| CuS39.6     | S       | 10.20                     | Cr 53       | Cr      | 53.00                     | RbCo29c     | Rb      | 7.65                      |             |         |                           |

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TABLE 5. TARGET AND TOLERANCE VALUES FOR QC RESULTS

| FILE: 0:QCBEGTGT |    |           |          |          | (TARGET VALUES)        |    |           |          |          |
|------------------|----|-----------|----------|----------|------------------------|----|-----------|----------|----------|
| STDEL            | EL | AREA      | CENTROID | FWHM     | STD                    | EL | AREA      | CENTROID | FWHM     |
| ID               |    | (cts)     | (keV)    | (ev)     | ID                     |    | (cts)     | (keV)    | (ev)     |
| 1833             | Pb | 31112.12  | 10.5449  | 207.4653 | 1832                   | Cu | 17548.85  | 8.0411   | 174.1389 |
| 1833             | Zn | 31772.52  | 8.6306   | 179.6835 | 1832                   |    | 5303.84   | 6.9247   | 167.1478 |
| 1833             | Fe | 313475.41 | 6.3935   | 159.4537 | 1832                   | Mn | 86202.33  | 5.8891   | 154.6347 |
| 1833             | Ti | 216978.09 | 4.5037   | 142.4946 | 1832                   | Ca | 217562.00 | 3.6847   | 135.3520 |
| 1833             | Si | 69021.60  | 1.7322   | 121.7406 | 1832                   | V  | 99761.96  | 4.9443   | 146.1904 |
| 1833             | K  | 220344.80 | 3.3069   | 132.4137 | 1832                   | Al | 16562.45  | 1.4779   | 119.5793 |
| BLKt             | Sn | 111.52    | 0.0000   | 0.0000   | 1832                   | Si | 67688.42  | 1.7319   | 118.4960 |
| BLKt             | Pb | 85.82     | 0.0000   | 0.0000   | 1832                   | Na | 10332.21  | 1.0256   | 114.4485 |
| BLKt             | Cu | 497.06    | 0.0000   | 0.0000   | BLKt                   | Ba | 183.14    | 0.0000   | 0.0000   |
| BLKt             | Sr | 72.92     | 0.0000   | 0.0000   | BLKt                   | W  | 241.42    | 0.0000   | 0.0000   |
| BLKt             | Ni | 648.99    | 0.0000   | 0.0000   | BLKt                   | Zn | 148.48    | 0.0000   | 0.0000   |
| BLKt             | Fe | 459.10    | 0.0000   | 0.0000   | BLKt                   | Sr | 83.00     | 0.0000   | 0.0000   |
| BLKt             | S  | 266.76    | 0.0000   | 0.0000   | BLKt                   | Ni | 654.44    | 0.0000   | 0.0000   |
| BLKt             | Al | 396.30    | 0.0000   | 0.0000   | BLKt                   | Fe | 603.55    | 0.0000   | 0.0000   |
| BLKt             | Ar | 747.74    | 0.0000   | 0.0000   | BLKt                   | S  | 3047.53   | 0.0000   | 0.0000   |
| BLKt             | Na | 120.85    | 0.0000   | 0.0000   | BLKt                   | Si | 936.48    | 0.0000   | 0.0000   |
| BaNa             | Na | 27711.44  | 1.0278   | 107.2698 | BLKt                   | Ar | 751.18    | 0.0000   | 0.0000   |
| BaNa             | Ba | 7369.12   | 32.0701  | 670.6336 | BLKt                   | Mg | 3622.12   | 0.0000   | 0.0000   |
|                  |    |           |          |          | BaSr                   | Sr | 210871.20 | 14.1410  | 227.8625 |
|                  |    |           |          |          | BaSr                   | Ba | 7464.85   | 32.0692  | 671.0372 |
| FILE: 0:QCBEGTOL |    |           |          |          | (TOLERANCE UNITS in %) |    |           |          |          |
| STDEL            | EL | AREA      | CENTROID | FWHM     | STD                    | EL | AREA      | CENTROID | FWHM     |
| ID               |    | (cts)     | (keV)    | (ev)     | ID                     |    | (cts)     | (keV)    | (ev)     |
| 1833             | Pb | 1.66      | 0.0313   | 0.9901   | 1832                   | Cu | 1.66      | 0.0104   | 1.9331   |
| 1833             | Zn | 1.66      | 0.0131   | 1.7328   | 1832                   | Co | 1.70      | 0.0308   | 2.4345   |
| 1833             | Fe | 1.66      | 0.0224   | 0.9361   | 1832                   | Mn | 1.66      | 0.0198   | 1.3536   |
| 1833             | Ti | 1.66      | 0.0259   | 0.9768   | 1832                   | Ca | 1.66      | 0.0253   | 1.1311   |
| 1833             | Si | 1.66      | 0.0616   | 1.4120   | 1832                   | V  | 1.66      | 0.0243   | 1.1031   |
| 1833             | K  | 1.66      | 0.0323   | 0.9235   | 1832                   | Al | 2.02      | 0.1173   | 3.3722   |
| BLKt             | Sn | 12.98     | 0.0000   | 0.0000   | 1832                   | Si | 1.66      | 0.0481   | 0.8888   |
| BLKt             | Pb | 8.93      | 0.0000   | 0.0000   | 1832                   | Na | 1.78      | 0.1560   | 1.5333   |
| BLKt             | Cu | 4.95      | 0.0000   | 0.0000   | BLKt                   | Ba | 9.92      | 0.0000   | 0.0000   |
| BLKt             | Sr | 17.61     | 0.0000   | 0.0000   | BLKt                   | W  | 8.20      | 0.0000   | 0.0000   |
| BLKt             | Ni | 3.81      | 0.0000   | 0.0000   | BLKt                   | Zn | 11.45     | 0.0000   | 0.0000   |
| BLKt             | Fe | 7.57      | 0.0000   | 0.0000   | BLKt                   | Sr | 10.88     | 0.0000   | 0.0000   |
| BLKt             | S  | 8.71      | 0.0000   | 0.0000   | BLKt                   | Ni | 6.55      | 0.0000   | 0.0000   |
| BLKt             | Al | 7.23      | 0.0000   | 0.0000   | BLKt                   | Fe | 5.63      | 0.0000   | 0.0000   |
| BLKt             | Ar | 17.39     | 0.0000   | 0.0000   | BLKt                   | S  | 2.88      | 0.0000   | 0.0000   |
| BLKt             | Na | 16.00     | 0.0000   | 0.0000   | BLKt                   | Si | 6.75      | 0.0000   | 0.0000   |
| BaNa             | N  | 1.66      | 0.1103   | 1.2599   | BLKt                   | Ar | 22.14     | 0.0000   | 0.0000   |
| BaNa             | Ba | 2.53      | 0.0979   | 3.9782   | BLKt                   | Mg | 5.64      | 0.0000   | 0.0000   |
|                  |    |           |          |          | BaSr                   | Sr | 1.66      | 0.0073   | 0.4538   |
|                  |    |           |          |          | BaSr                   | Ba | 1.86      | 0.0279   | 2.8094   |

**Chapter IO-3**  
**Chemical Analysis**

**Method IO-3.3**  
**X-Ray Analysis**

TABLE 6. EXAMPLE PRINTOUT OF SRM 1833

KEVEX SUMMARY: TEFLON® BLANKS LOT #457803 (NEW TUBE)

SITE =  
 DURATION (MIN) = .0      SAMPLE DATE = 99/99/99 AND 9999 HOURS  
 FLOW FRAC = .0000      FLOW (L/MIN) = .000 +/- .200  
 XRF ID = 112141  
 SAMPLE ID = SRM1833

| FINE, NG/CM <sup>2</sup> |         |    |        | NIST CERTIFIED VALUES |         |    |        |
|--------------------------|---------|----|--------|-----------------------|---------|----|--------|
| MASS                     | 0.      | +- | 398.   | MASS                  | 15447   |    |        |
| *NA                      | -801.2  | +- | 326.4  | NA                    | .0      | +- | .0     |
| MG                       | 161.3   | +- | 18.2   | MG                    | .0      | +- | .0     |
| AL                       | 1027.5  | +- | 102.2  | AL                    | .0      | +- | .0     |
| SI                       | 34806.8 | +- | 3023.4 | SI                    | 33366.0 | +- | 2163.0 |
| P                        | 79.8    | +- | 19.9   | P                     | .0      | +- | .0     |
| *S                       | -28.2   | +- | 782.8  | S                     | .0      | +- | .0     |
| *CL                      | -68.6   | +- | 113.8  | CL                    | .0      | +- | .0     |
| K                        | 16734.7 | +- | 1018.7 | K                     | 17147.0 | +- | 1699.0 |
| *CA                      | -3.9    | +- | 61.4   | CA                    | .0      | +- | .0     |
| *SC                      | -17.1   | +- | 5.4    | SC                    | .0      | +- | .0     |
| TI                       | 12852.9 | +- | 822.1  | TI                    | 12821.0 | +- | 1854.0 |
| *V                       | 46.0    | +- | 52.2   | V                     | .0      | +- | .0     |
| CR                       | 108.2   | +- | 12.7   | CR                    | .0      | +- | .0     |
| MN                       | 13.8    | +- | 2.9    | MN                    | .0      | +- | .0     |
| FE                       | 14332.4 | +- | 872.4  | FE                    | 14212.0 | +- | 463.0  |
| *CO                      | -2.6    | +- | 2.9    | CO                    | .0      | +- | .0     |
| NI                       | 62.5    | +- | 4.6    | NI                    | .0      | +- | .0     |
| *CU                      | 3.8     | +- | 1.5    | CU                    | .0      | +- | .0     |
| ZN                       | 3800.9  | +- | 327.7  | ZN                    | 3862.0  | +- | 309.0  |
| *GA                      | -30.9   | +- | 7.7    | GA                    | .0      | +- | .0     |
| *GE                      | 5.9     | +- | 3.6    | GE                    | .0      | +- | .0     |
| *AS                      | 5.7     | +- | 14.6   | AS                    | .0      | +- | .0     |
| *SE                      | -2.0    | +- | 2.6    | SE                    | .0      | +- | .0     |
| *BR                      | -2.3    | +- | 2.5    | BR                    | .0      | +- | .0     |
| *RB                      | .5      | +- | 1.4    | RB                    | .0      | +- | .0     |
| *SR                      | -5.0    | +- | 2.9    | SR                    | .0      | +- | .0     |
| *Y                       | -2.6    | +- | 7.5    | Y                     | .0      | +- | .0     |
| *ZR                      | -7.6    | +- | 3.5    | ZR                    | .0      | +- | .0     |
| MO                       | 45.4    | +- | 5.6    | MO                    | .0      | +- | .0     |
| *RH                      | 156.7   | +- | 69.5   | RH                    | .0      | +- | .0     |
| *PD                      | 79.2    | +- | 67.1   | PD                    | .0      | +- | .0     |
| *AG                      | 114.0   | +- | 69.7   | AG                    | .0      | +- | .0     |

TABLE 6. (continued)

| FINE, NG/CM <sup>2</sup> |         |    |        | NIST CERTIFIED VALUES |         |    |       |
|--------------------------|---------|----|--------|-----------------------|---------|----|-------|
| *CD                      | 24.7    | +- | 66.3   | CD                    | .0      | +- | .0    |
| *SN                      | -1496.1 | +- | 188.1  | SN                    | .0      | +- | .0    |
| *SB                      | 88.2    | +- | 96.2   | SB                    | .0      | +- | .0    |
| *TE                      | 240.8   | +- | 93.8   | TE                    | .0      | +- | .0    |
| *I                       | 134.8   | +- | 107.5  | I                     | .0      | +- | .0    |
| *CS                      | -209.3  | +- | 106.6  | CS                    | .0      | +- | .0    |
| *BA                      | -5098.1 | +- | 517.8  | BA                    | .0      | +- | .0    |
| *LA                      | -1416.4 | +- | 202.2  | LA                    | .0      | +- | .0    |
| W                        | 59.9    | +- | 17.6   | W                     | .0      | +- | .0    |
| *AU                      | 8.7     | +- | 6.8    | AU                    | .0      | +- | .0    |
| *HG                      | -30.6   | +- | 5.9    | HG                    | .0      | +- | .0    |
| PB                       | 16886.2 | +- | 1028.1 | PB                    | 16374.0 | +- | 772.0 |

\* INDICATES THAT THE CONCENTRATION IS BELOW 3 TIMES THE UNCERTAINTY.

XRF DATE= 28-SEP-93 10:58:37 RBK

SPECTRAL ANALYSIS DATE= 12/14/1993

**Chapter IO-3**  
**Chemical Analysis**

**Method IO-3.3**  
**X-Ray Analysis**

TABLE 7. EXAMPLE PRINTOUT OF SRM 1832

KEVEX SUMMARY: TEFLO® BLANKS LOT #457803 (NEW TUBE)

SITE =  
 DURATION (MIN) = .0 SAMPLE DATE = 99/99/99 AND 9999 HOURS  
 FLOW FRAC = .0000 FLOW (L/MIN) = .000 +/- .200  
 XRF ID = 112191  
 SAMPLE ID = SRM1832

| FINE, NG/CM <sup>2</sup> |         |     |        | NIST CERTIFIED VALUES |         |     |        |
|--------------------------|---------|-----|--------|-----------------------|---------|-----|--------|
| MASS                     | 0.      | + - | 398.   | MASS                  | 16431   |     |        |
| NA                       | 11891.5 | + - | 1035.0 | NA                    | 11173.0 | + - | .0     |
| MG                       | 92.2    | + - | 13.0   | MG                    | .0      | + - | .0     |
| AL                       | 15856.5 | + - | 1373.2 | AL                    | 14953.0 | + - | 986.0  |
| SI                       | 34398.8 | + - | 2964.2 | SI                    | 35491.0 | + - | 1150.0 |
| P                        | 492.0   | + - | 32.1   | P                     | .0      | + - | .0     |
| S                        | 402.1   | + - | 27.3   | S                     | .0      | + - | .0     |
| CL                       | 156.8   | + - | 15.9   | CL                    | .0      | + - | .0     |
| * K                      | 18.5    | + - | 18.0   | K                     | .0      | + - | .0     |
| CA                       | 20011.7 | + - | 1218.2 | CA                    | 19225.0 | + - | 1315.0 |
| *SC                      | -21.8   | + - | 5.6    | SC                    | .0      | + - | .0     |
| *TI                      | -4.7    | + - | 130.6  | TI                    | .0      | + - | .0     |
| V                        | 4593.6  | + - | 281.1  | V                     | 4272.0  | + - | 493.0  |
| *CR                      | 7.4     | + - | 7.3    | CR                    | .0      | + - | .0     |
| MN                       | 4959.3  | + - | 302.4  | MN                    | 4437.0  | + - | 493.0  |
| FE                       | 30.5    | + - | 3.9    | FE                    | .0      | + - | .0     |
| CO                       | 1055.1  | + - | 64.7   | CO                    | 970.0   | + - | 66.0   |
| *NI                      | -6.8    | + - | 1.8    | NI                    | .0      | + - | .0     |
| CU                       | 2400.1  | + - | 146.3  | CU                    | 2300.0  | + - | 164.0  |
| ZN                       | 9.3     | + - | 2.7    | ZN                    | .0      | + - | .0     |
| *GA                      | 2.1     | + - | 2.1    | GA                    | .0      | + - | .0     |
| *GE                      | .3      | + - | 2.4    | GE                    | .0      | + - | .0     |
| *AS                      | -3.7    | + - | 2.2    | AS                    | .0      | + - | .0     |
| *SE                      | 1.0     | + - | 1.2    | SE                    | .0      | + - | .0     |
| BR                       | 10.7    | + - | 1.8    | BR                    | .0      | + - | .0     |
| *RB                      | -.2     | + - | .9     | RB                    | .0      | + - | .0     |
| *SR                      | 2.8     | + - | 2.3    | SR                    | .0      | + - | .0     |
| * Y                      | -5.0    | + - | 1.6    | Y                     | .0      | + - | .0     |
| *ZR                      | -6.5    | + - | 1.8    | ZR                    | .0      | + - | .0     |
| MO                       | 26.8    | + - | 4.2    | MO                    | .0      | + - | .0     |
| *RH                      | 25.2    | + - | 58.2   | RH                    | .0      | + - | .0     |

TABLE 7. (continued)

|     | FINE, NG/CM <sup>2</sup> |    |       | NIST CERTIFIED VALUES |    |    |    |
|-----|--------------------------|----|-------|-----------------------|----|----|----|
| *PD | -69.0                    | +- | 54.7  | PD                    | .0 | +- | .0 |
| *AG | 151.2                    | +- | 63.4  | AG                    | .0 | +- | .0 |
| *CD | 24.2                     | +- | 58.2  | CD                    | .0 | +- | .0 |
| *SN | -640.8                   | +- | 138.6 | SN                    | .0 | +- | .0 |
| *SB | -73.5                    | +- | 81.3  | SB                    | .0 | +- | .0 |
| *TE | -9.3                     | +- | 73.9  | TE                    | .0 | +- | .0 |
| *I  | -46.6                    | +- | 91.6  | I                     | .0 | +- | .0 |
| *CS | 3.6                      | +- | 96.7  | CS                    | .0 | +- | .0 |
| *BA | -2352.9                  | +- | 328.6 | BA                    | .0 | +- | .0 |
| *LA | -509.9                   | +- | 156.5 | LA                    | .0 | +- | .0 |
| W   | 40.0                     | +- | 12.9  | W                     | .0 | +- | .0 |
| *AU | -5.6                     | +- | 2.5   | AU                    | .0 | +- | .0 |
| *HG | -5.4                     | +- | 3.0   | HG                    | .0 | +- | .0 |
| *PB | -10.4                    | +- | 4.2   | PB                    | .0 | +- | .0 |

\* INDICATES THAT THE CONCENTRATION IS BELOW 3 TIMES THE UNCERTAINTY.

XRF DATE= 29-SEP-93 13:27:55 RBK

SPECTRAL ANALYSIS DATE= 12/14/1993

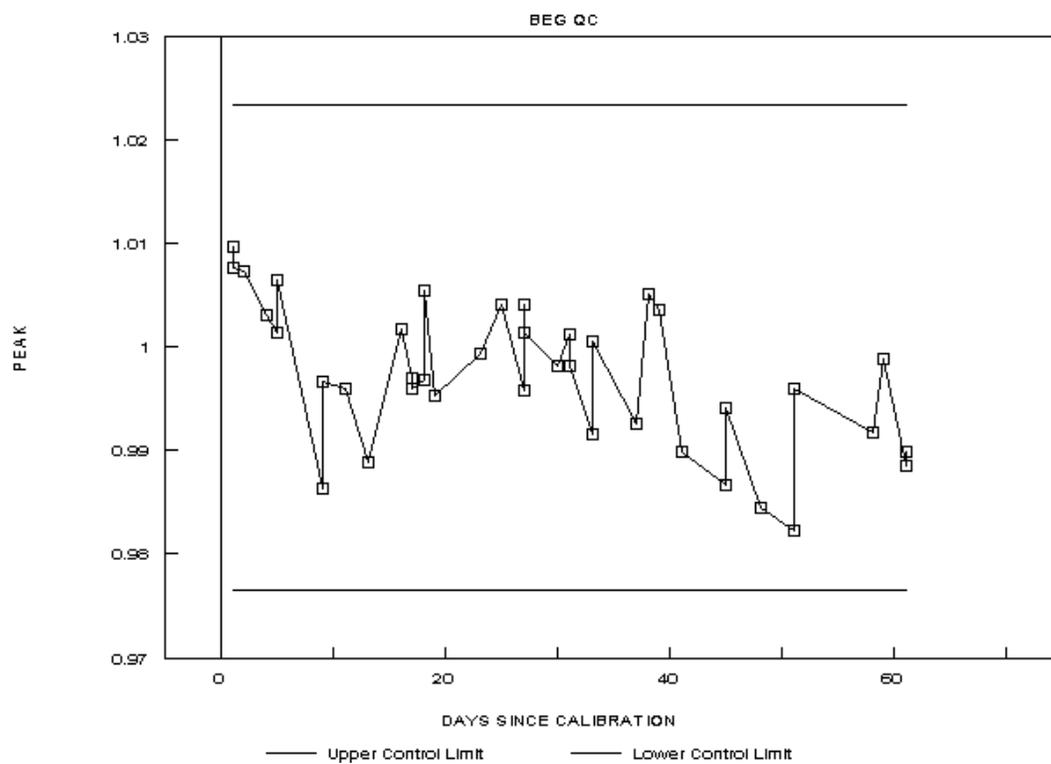


Figure 1. Quality control indicator associated with Fe peak area.

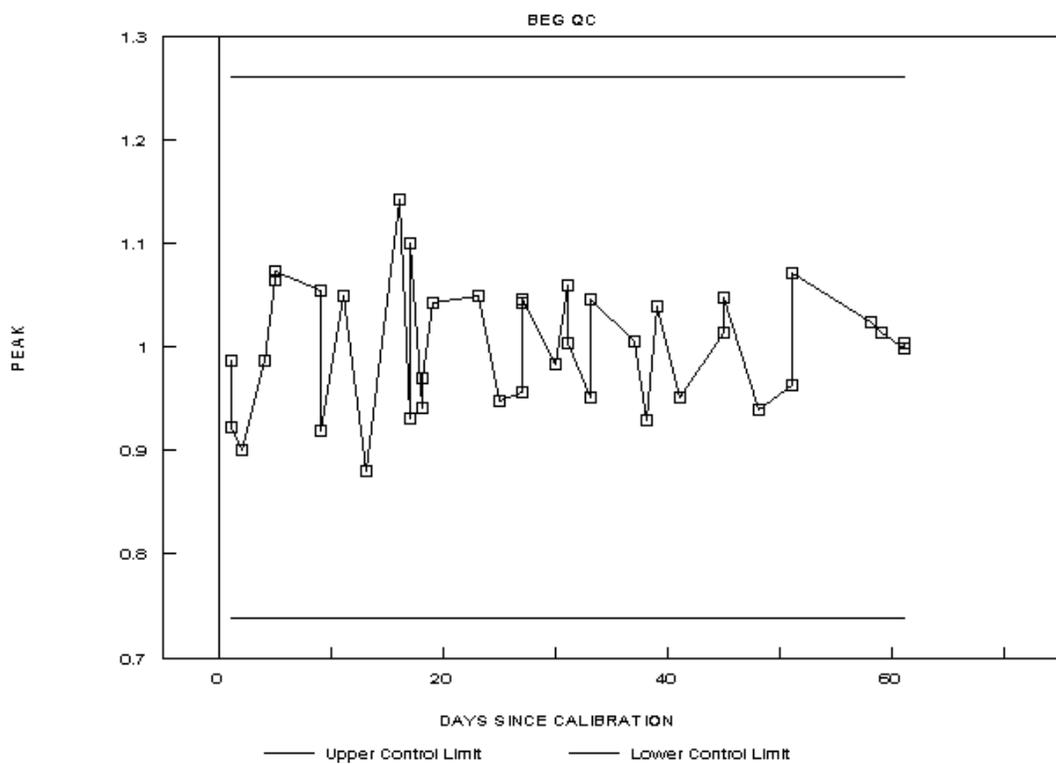


Figure 2. Quality control indicator associated with S background area.

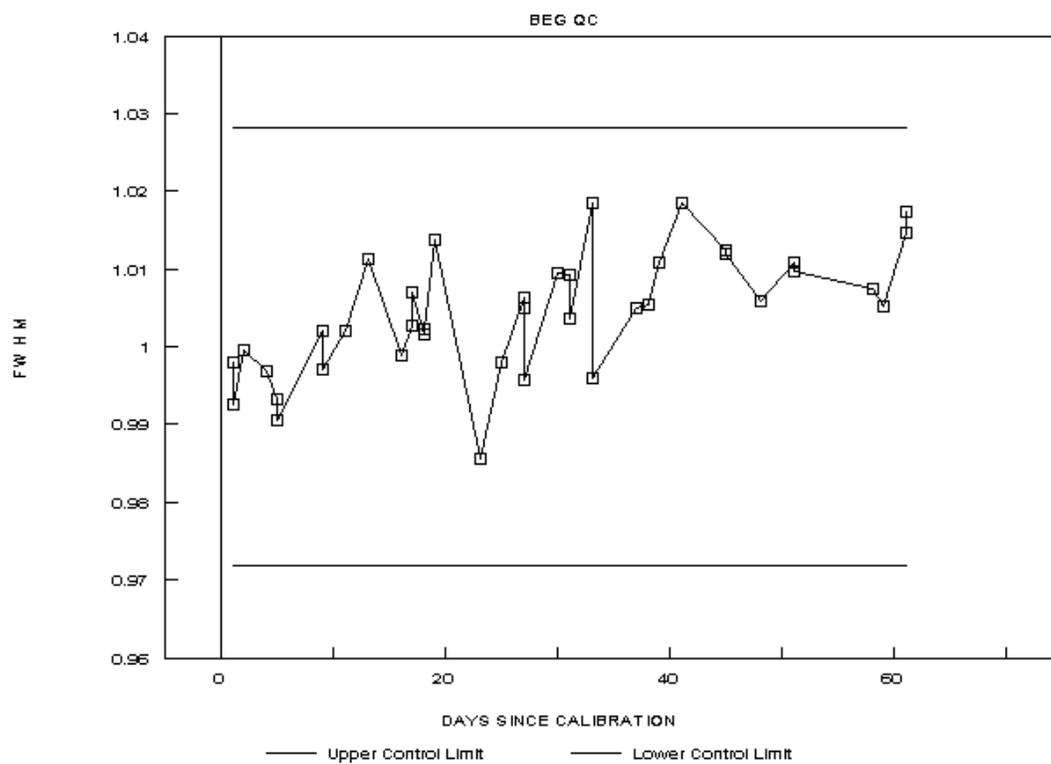


Figure 3. Quality control indicator associated with Fe FWHM.

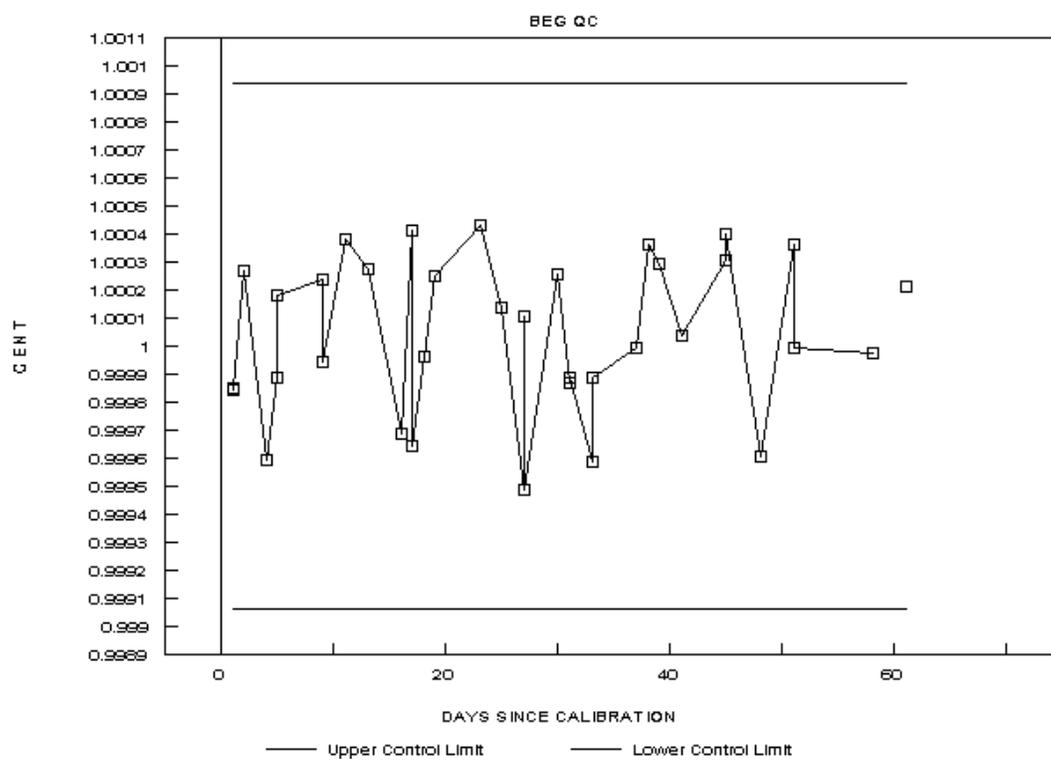


Figure 4. Quality control indicator associated with Pb centroid.

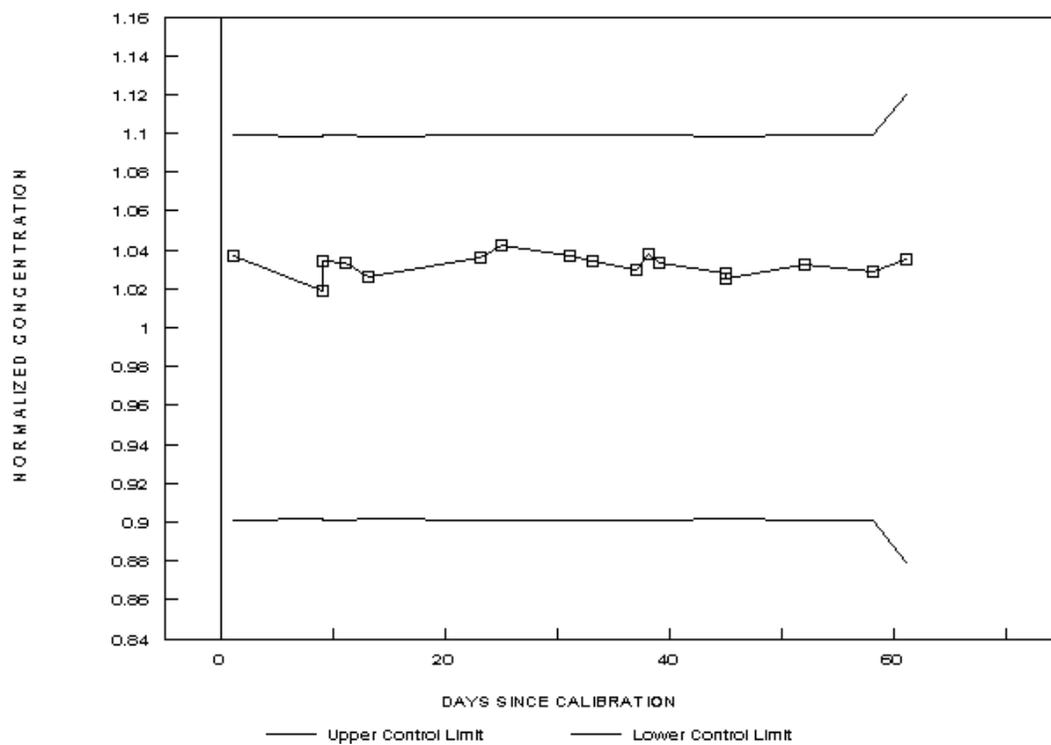


Figure 5. Quality control indicator associated with Pb in SRMs.

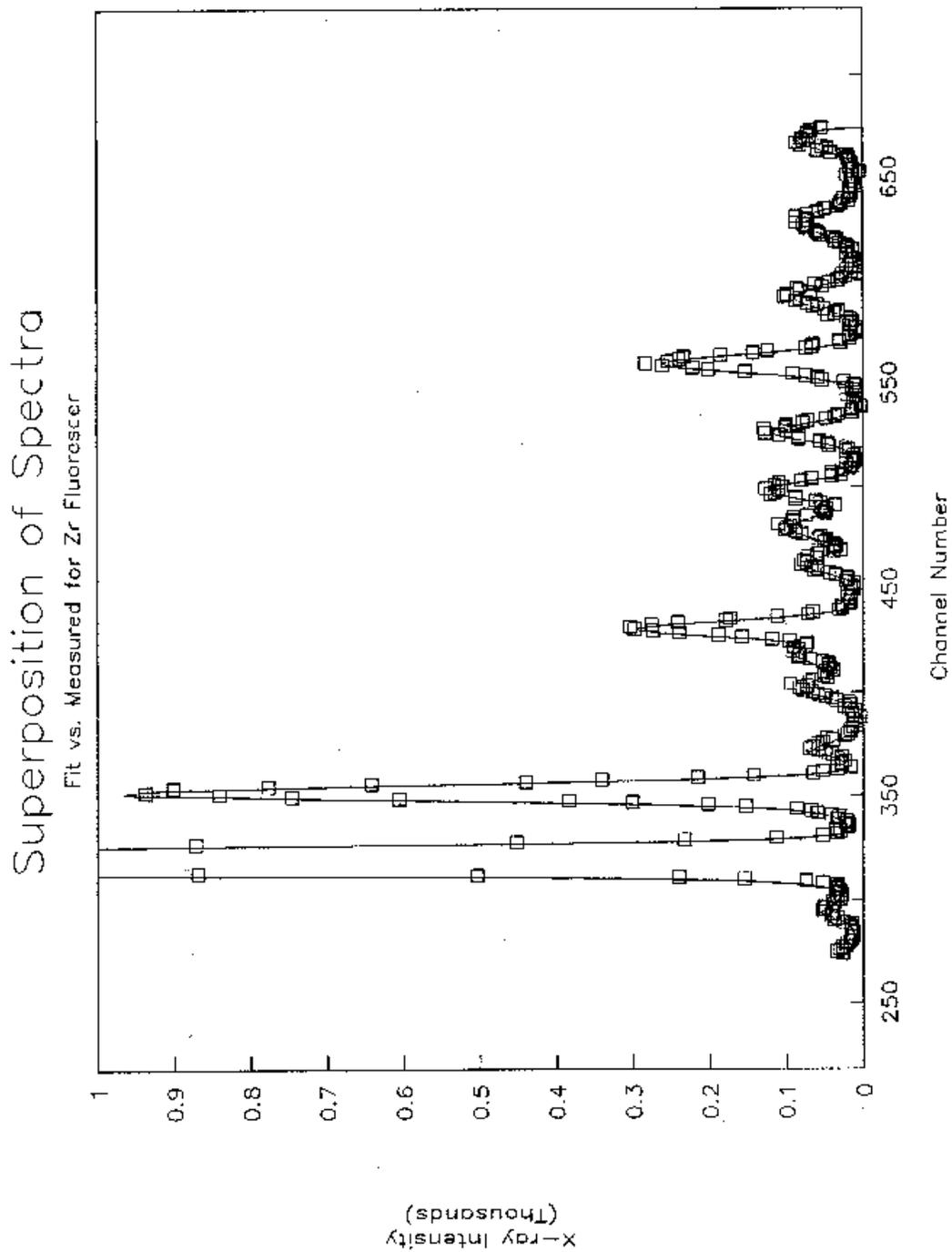


Figure 6. Quality control indicator associated with superposition of fitted and measured spectrum.



## E6 Determination of Ring Thickness

**Designation: D 1777 – 96 (Reapproved 2007)**

### Standard Test Method for Thickness of Textile Materials<sup>1</sup>

This standard is issued under the fixed designation D 1777; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

#### 1. Scope

1.1 This test method covers the measurement of the thickness of most textile materials.

1.2 This test method applies to most fabrics including woven fabrics, air bag fabrics, blankets, napped fabrics, knitted fabrics, layered fabrics, and pile fabrics. The fabrics may be untreated, heavily sized, coated, resin-treated, or otherwise treated. Instructions are provided for testing thickness, except as provided for in another standard such as listed in Section 2.

1.3 The values stated in SI units are to be regarded as the standard. The values stated in inch-pound may be approximate.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

#### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

**D 123** Terminology Relating to Textiles

**D 1776** Practice for Conditioning and Testing Textiles

**D 2904** Practice for Interlaboratory Testing of a Textile Test Method that Produces Normally Distributed Data

**D 2906** Practice for Statements on Precision and Bias for Textiles<sup>3</sup>

2.2 *ASTM Adjuncts:*

TEX-PAC<sup>4</sup>

#### 3. Terminology

3.1 Definitions—For definitions of other textile terms used in this test method, see Terminology **D 123**.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D13 on Textiles and is the direct responsibility of Subcommittee D13.59 on Fabric Test Methods, General.

Current edition approved Jan. 1, 2007. Published January 2007. Originally approved in 1960. Discontinued in November 1995 and reinstated as D 1777 – 96. Last previous edition approved in 2002 as D 1777 – 96(2002).

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Withdrawn.

<sup>4</sup> A PC program on floppy disk for analyzing Committee D-13 interlaboratory data are available from ASTM Headquarters. For a 3½-in. disk, request PCN:12-429040-18. For a 5¼-in. disk, request PCN:12-429041-18.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *cross-machine direction, CD, n*—the direction in the plane of the fabric perpendicular to the direction of manufacture.

3.2.1.1 *Discussion*—This term is used to refer to the direction analogous to coursewise or filling direction in knitted or woven fabrics, respectively.

3.2.2 *machine direction, MD, n*—the direction in the plane of the fabric parallel to the direction of manufacture.

3.2.2.1 *Discussion*—This term is used to refer to the direction analogous to walewise or warp direction in knitted or woven fabrics, respectively.

3.2.3 *pressure, n*—the force exerted to a surface per unit area.

3.2.3.1 *Discussion*—Pressure may be expressed in any appropriate or specified units, such as pascals (Pa), newtons per square metre (N/m<sup>2</sup>), or pounds-force per square inch (psi).

3.2.4 *thickness, n*—the distance between one surface of a material and its opposite.

3.2.4.1 *Discussion*—In textiles, thickness is the distance between the upper and lower surfaces of the material as measured under a specified pressure. It is usually determined as

the distance between an anvil or base and a presser foot used to apply the specified pressure.

## 4. Summary of Test Method

4.1 A specimen is placed on the base of a thickness gauge and a weighted presser foot lowered. The displacement between the base and the presser foot is measured as the thickness of the specimen.

## 5. Significance and Use

5.1 This test method is considered satisfactory for acceptance testing of commercial shipments since current estimates of between-laboratory precision are acceptable, and this test method is used extensively in the trade for acceptance testing.

5.1.1 In case of a dispute arising from differences in reported test results when using this test method for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine if there is a statistical bias between their laboratories. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the two parties should take a group of test specimens that are as homogeneous as possible and that are

from a lot of material of the type in question. Test specimens then should be randomly assigned in equal numbers to each laboratory for testing. The average results from the two laboratories should be compared using the appropriate statistical analysis and an acceptable probability level chosen by the two parties before testing is begun. If a bias is found, either its cause must be found and corrected, or the purchaser and the supplier must agree to interpret future test results with consideration to the known bias.

5.2 Thickness is one of the basic physical properties of textile materials. In certain industrial applications, the thickness may require rigid control within specified limits. Bulk and warmth properties of textile materials are often estimated from their thickness values, and thickness is also useful in measuring some performance characteristics, such as before and after abrasion and shrinkage.

5.3 The thickness value of most textile materials will vary considerably depending on the pressure applied to the specimen at the time the thickness measurement is taken. In all cases, the apparent thickness varies inversely with the pressure applied. For this reason, it is essential that the pressure be specified when discussing or listing any thickness value.

5.4 When using this test method for measuring the thickness of textile materials, the primary method for the specific material such as listed in the Referenced Document section shall take precedence over the directions described in this test method, unless specifically provided for in that test method. This test method is used in its entirety when no test method for measuring thickness is available for the specific material to be tested or unless otherwise specified in a material specification or contract order.

## 6. Apparatus

6.1 *Thickness Gauge*, having dimensions appropriate to the material to be tested as specified in Table 1, unless otherwise specified in a material specification or contract order. A circular presser foot commonly is used for most materials; however, for

certain materials, such as narrow tapes, a rectangular foot is more appropriate when agreed upon between the purchaser and the supplier.

6.1.1 *Automatic Microprocessor Data Gathering Systems*, optional.

6.1.2 *Spring Force or Compression Test Apparatus*, may be substituted for the dead-weight-type thickness gauge providing they meet the specified conditions cited in Table 1.

6.2 *Cutting Dies or Templates*, to cut specimens having minimum dimensions at least 20 % greater than any dimension of the presser foot to be used in measuring the thickness (optional).

## 7. Sampling and Test Specimens

7.1 *Lot Sample*—As a lot sample for acceptance testing, randomly select the number of rolls or pieces of fabric directed in an applicable material specification or other agreement between the purchaser and the supplier. Consider the rolls or pieces of fabric to be the primary sampling units. In the absence of such an agreement, take the number of fabric rolls specified in Table 2.

NOTE 1—An adequate specification or other agreement between the purchaser and the supplier requires taking into account the variability between rolls or pieces of fabric and between specimens from a swatch from a roll or piece of fabric to provide a sampling plan with a meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

7.2 *Laboratory Sample*—For acceptance testing, take a swatch extending the width of the fabric and approximately 1 m (1 yd) along the machine direction from each roll or piece in the lot sample. For rolls of fabric, take a sample that will exclude fabric from the outer wrap of the roll or the inner wrap around the core of the roll of fabric. For finished garments, take a piece of sufficient size to provide the number of specimens required in 7.3.

7.3 *Test Specimens*—From each laboratory sampling unit, take ten specimens. Use the cutting die or template described in

**TABLE 1 Designated Gauges and Gauge Specifications for Measuring Thickness of Textiles**

| Testing Option <sup>A</sup> | Material Type  | Gauge Type <sup>B</sup> | Presser Foot Diameter   | Anvil                     | Anvil/Foot Parallelism  | Foot to Anvil Surface Parallelism | Applied Pressure  | Readability            |
|-----------------------------|--|-------------------------|-------------------------|---------------------------|-------------------------|-----------------------------------|---|------------------------|
| 1                           | Woven fabrics  | dead-weight mm          | 28.7 $\pm$ 0.02         | 38 mm D, or greater       | 0.01 mm                 | 0.002 mm                          | 4.14 $\pm$ 0.21 kPa   | 0.02 mm                |
|                             | Knitted fabrics<br>Textured fabrics                      |                         | (1.129 $\pm$ 0.001 in.) | (1.629 in. D, or greater) | (0.0005 in.)            | (0.0001 in.)                      | (0.60 $\pm$ 0.03 psi)   | (0.001 in.)            |
| 2                           | Coated fabrics   | dead-weight mm          | 9.5 $\pm$ 0.02          | 38 mm D, or greater       | 0.01 mm                 | 0.002 mm                          | 23.4 $\pm$ 0.7 kPa  | 0.02 mm                |
|                             | Narrow fabrics<br>Webbings<br>Tapes<br>Ribbons<br>Braids |                         | (0.375 $\pm$ 0.001 in.) | (1.629 in. D, or greater) | (0.0005 in.)            | (0.0001 in.)                      | (3.4 $\pm$ 0.1 psi)   | (0.001 in.)            |
| 3                           | Films  | dead-weight mm          | 6.3 $\pm$ 0.02          | 19 mm D, or greater       | 0.002 mm                | 0.002 mm                          | 172 $\pm$ 14 kPa  | 0.002 mm               |
|                             | Glass cloths<br>Glass tapes                              |                         | (0.250 $\pm$ 0.001 in.) | (0.750 in. D, or greater) | (0.0001 in.)            | (0.0001 in.)                      | (25 $\pm$ 2 psi)  | (0.0001 in.)           |
| 4                           | Glass fiber mat  | dead-weight             | (2.25 $\pm$ 0.001 in.)  | (2.75 in. D, or greater)  | 0.01 mm<br>(0.0005 in.) | 0.002 mm<br>(0.0001 in.)          | 18.9 $\pm$ 0.7 kPa<br>(2.75 $\pm$ 0.1 psi)                            | 0.02 mm<br>(0.001 in.) |
| 5                           | Blankets   | dead-weight mm          | 28.7 $\pm$ 0.02         | 38 mm D, or greater       | 0.01 mm                 | 0.002 mm                          | 0.7 $\pm$ 0.07 kPa  | 0.02 mm                |
|                             | Pile fabrics<br>Napped fabrics                           |                         | (1.129 $\pm$ 0.001 in.) | (1.629 in. D, or greater) | (0.0005 in.)            | (0.0001 in.)                      | (0.1 $\pm$ 0.01 psi)<br>also 7.58 $\pm$ 0.21 kPa (1.1 $\pm$ 0.03 psi) | (0.001 in.)            |

<sup>A</sup> When testing fabrics made with textured yarns or open-end spun yarns, primary consideration should be given to the pressure applied in Option 1, with respect to the size of the presser foot used.

<sup>B</sup> Other spring force or compression test apparatus that meet the stated specifications can be used.



**D 1777 - 96 (2007)**

**TABLE 2 Number of Rolls or Pieces of Fabric in the Lot Sample**

| Number of Rolls or Pieces in Lot, Inclusive | Number of Rolls or Pieces in Lot Sample |
|---|---|
| 1 to 3                                      | all                                     |
| 4 to 24                                     | 4                                       |
| 25 to 50                                    | 5                                       |
| over 50                                     | 10 % to a maximum of 10 rolls or pieces |

6.2. It is permissible to make thickness tests of a textile material without cutting, providing it can be maintained without distortion in a plane parallel to the presser foot and anvil while making measurements.

7.3.1 *Cutting Test Specimens*—When cutting specimens, cut having minimum dimensions at least 20 % greater than any dimension of the presser foot to be used. Label to maintain specimen identity.

7.3.1.1 Take specimens, representing a broad distribution across the width and length, and preferably along the diagonal, of the laboratory sample and no nearer the edge than one tenth its width. Ensure specimens are free of folds, creases, or wrinkles. Avoid getting oil, water, grease, and so forth, on the specimens when handling.

## 8. Preparation of Test Apparatus and Calibration

8.1 Use [Table 1](#) to select the thickness gauge designated for the material to be tested.

8.2 Verify calibration of the thickness gauge as directed in the manufacturer's instructions.

8.3 When using microprocessor automatic data gathering systems, set the appropriate parameters as defined in the manufacturer's instructions.

## 9. Conditioning

9.1 Precondition the specimens by bringing them to approximate moisture equilibrium in the standard atmosphere for preconditioning textiles as specified in [Practice D 1776](#), unless otherwise specified in a material specification or contract order.

9.2 After preconditioning, bring the test specimens to moisture equilibrium for testing in the standard atmosphere for testing textiles as specified in [Practice D 1776](#) or, if applicable, in the specified atmosphere in which the testing is to be performed, unless otherwise specified in a material specification or contract order.

## 10. Procedure

10.1 Test the conditioned specimens in the standard atmosphere for testing textiles, which is 21 ± 1°C (70 ± 2°F) and 65 ± 2 % relative humidity, unless otherwise specified in a material specification or contract order.

10.2 Handle the test specimens carefully to avoid altering the natural state of the material.

10.3 Place the specimen face side up, or otherwise as specified, on the anvil of the thickness gauge. Gradually lower the presser foot into contact with the specimen, taking 5 to 6 s to apply full pressure for most textiles and 3 to 4 s for glass material textiles.

10.4 Read and record the thickness value to a readability shown in [Table 1](#).

**TABLE 3 Thickness of Fabrics, mils**

| Test Options and Materials | Grand Average | Components of Variance Expressed as Standard Deviations |                             |                              |
|----------------------------|---------------|---|-----------------------------|------------------------------|
|                            |               | Single-Operator Component                               | Within-Laboratory Component | Between-Laboratory Component |
| Option 1                   |               |   |                             |                              |
| Mat 6, S/0002H             | 7.13          | 0.19  | 0.17                        | 0.66                         |
| Mat 7, S/28305             | 11.49         | 1.46  | 0.55                        | 0.02                         |
| Mat 9, S/Denim             | 38.65         | 0.75  | 0.41                        | 1.54                         |
| Option 2                   |               |   |                             |                              |
| Mat 6, S/0002H             | 7.04          | 0.23  | 0.00                        | 0.72                         |
| Mat 7, S/28305             | 11.84         | 0.23  | 0.27                        | 0.48                         |
| Mat 9, S/Denim             | 38.33         | 0.64  | 0.00                        | 1.64                         |
| Option 3                   |               |   |                             |                              |
| Mat 6, S/0002H             | 6.68          | 0.32  | 0.18                        | 0.58                         |
| Mat 7, S/28305             | 11.50         | 0.37  | 0.13                        | 0.49                         |
| Mat 9, S/Denim             | 36.50         | 0.60  | 0.19                        | 2.41                         |
| Option 4                   |               |   |                             |                              |
| Mat 6, S/0002H             | 10.20         | 0.40  | 0.00                        | ...                          |
| Mat 7, S/28305             | 12.85         | 0.36  | 0.00                        | ...                          |
| Mat 9, S/Denim             | 43.95         | 1.08  | 0.54                        | ...                          |
| Option 5 (0.1 psi)         |               |   |                             |                              |
| Mat 10, Pile               | 178.08        | 8.79  | 0.00                        | 3.30                         |
| Mat 14, Pile               | 46.91         | 1.15  | 0.16                        | 0.69                         |
| Option 5 (1.1 psi)         |               |   |                             |                              |
| Mat 10, Pile               | 101.67        | 4.63  | 0.00                        | 10.08                        |
| Mat 14, Pile               | 38.37         | 1.51  | 0.33                        | 0.28                         |

<sup>A</sup> The square roots of the components of variance are being reported to express the variability in the appropriate units of measure rather than as the squares of those units of measure.

10.5 Continue as directed in [10.2-10.4](#) until ten specimens have been tested from each laboratory sampling unit.

## 11. Calculation

11.1 *Thickness, Individual Specimens*—The thickness for individual specimens using the readability column of [Table 1](#) is read directly from the thickness gauge or data collection system, unless otherwise specified in a material specification or contract order.

11.2 *Thickness Average*—Calculate the average thickness for each laboratory sampling unit and for the lot.

11.3 *Standard Deviation, Coefficient of Variation*—Calculate when requested.

11.4 *Computer-Processed Data*—When data are automatically computer-processed, calculations generally are contained in the associated software. Record values as read from the direct-reading scale designated in the readability column of [Table 1](#), unless otherwise specified. In any event, it is recommended that computer-processed data be verified against known property values and its software described in the report.

## 12. Report

12.1 Report that the thickness was determined in accordance with Test Method D 1777. Describe the material or product sampled and the method of sampling used.

12.2 Report the following information for each laboratory sampling unit and for the lot as applicable to a material specification or contract order:

12.2.1 Thickness.

12.2.2 Testing option selected from [Table 1](#).

12.2.3 When calculated, the standard deviation or the coefficient of variation.

**TABLE 4 Thickness of Fabrics, mils**

| Test Options and Materials | Critical Differences for the Conditions Noted <sup>A</sup> |                           |                             |                              |
|----------------------------|--|---------------------------|-----------------------------|------------------------------|
|                            | Number of Observations in Each Average                     | Single-Operator Precision | Within-Laboratory Precision | Between-Laboratory Precision |
| Option 1                   |  |                           |                             |                              |
| Mat 6, S/0002H             | 1  | 0.53                      | 0.72                        | 1.96                         |
|                            | 2  | 0.37                      | 0.61                        | 1.93                         |
|                            | 5  | 0.24                      | 0.54                        | 1.91                         |
|                            | 10   | 0.17                      | 0.51                        | 1.90                         |
| Mat 7, S/28305             | 1  | 4.05                      | 4.33                        | 4.33                         |
|                            | 2  | 2.86                      | 3.25                        | 3.25                         |
|                            | 5  | 1.81                      | 2.37                        | 2.37                         |
|                            | 10   | 1.28                      | 2.00                        | 2.00                         |
| Mat 9, S/Denim             | 1  | 2.10                      | 2.39                        | 4.89                         |
|                            | 2  | 1.48                      | 1.88                        | 4.66                         |
|                            | 5  | 0.94                      | 1.48                        | 4.52                         |
|                            | 10   | 0.66                      | 1.32                        | 4.47                         |
| Option 2                   |  |                           |                             |                              |
| Mat 6, S/0002H             | 1  | 0.63                      | 0.63                        | 2.10                         |
|                            | 2  | 0.44                      | 0.44                        | 2.05                         |
|                            | 5  | 0.28                      | 0.28                        | 2.02                         |
|                            | 10   | 0.20                      | 0.20                        | 2.01                         |
| Mat 7, S/28305             | 1  | 0.63                      | 0.99                        | 1.65                         |
|                            | 2  | 0.45                      | 0.88                        | 1.59                         |
|                            | 5  | 0.28                      | 0.81                        | 1.55                         |
|                            | 10   | 0.20                      | 0.79                        | 1.54                         |
| Mat 9, S/Denim             | 1  | 1.76                      | 1.76                        | 4.88                         |
|                            | 2  | 1.25                      | 1.25                        | 4.72                         |
|                            | 5  | 0.79                      | 0.79                        | 4.62                         |
|                            | 10   | 0.56                      | 0.56                        | 4.59                         |
| Option 3                   |  |                           |                             |                              |
| Mat 6, S/0002H             | 1  | 0.90                      | 1.03                        | 1.91                         |
|                            | 2  | 0.64                      | 0.81                        | 1.80                         |
|                            | 5  | 0.40                      | 0.64                        | 1.73                         |
|                            | 10   | 0.29                      | 0.58                        | 1.71                         |
| Mat 7, S/28305             | 1  | 1.04                      | 1.10                        | 1.74                         |
|                            | 2  | 0.73                      | 0.82                        | 1.58                         |
|                            | 5  | 0.46                      | 0.58                        | 1.47                         |
|                            | 10   | 0.33                      | 0.48                        | 1.43                         |
| Mat 9, S/Denim             | 1  | 1.67                      | 1.75                        | 6.90                         |
|                            | 2  | 1.18                      | 1.29                        | 6.79                         |
|                            | 5  | 0.75                      | 0.91                        | 6.73                         |
|                            | 10   | 0.53                      | 0.74                        | 6.71                         |
| Option 4                   |  |                           |                             |                              |
| Mat 6, S/0002H             | 1  | 1.12                      | 1.12                        | ...                          |
|                            | 2  | 0.79                      | 0.79                        | ...                          |
|                            | 5  | 0.50                      | 0.50                        | ...                          |
|                            | 10   | 0.36                      | 0.36                        | ...                          |
| Mat 7, S/28305             | 1  | 1.00                      | 1.00                        | ...                          |
|                            | 2  | 0.71                      | 0.71                        | ...                          |
|                            | 5  | 0.45                      | 0.45                        | ...                          |
|                            | 10   | 0.32                      | 0.32                        | ...                          |
| Mat 9, S/Denim             | 1  | 2.99                      | 3.34                        | ...                          |
|                            | 2  | 2.11                      | 2.58                        | ...                          |
|                            | 5  | 1.34                      | 2.00                        | ...                          |
|                            | 10   | 0.94                      | 1.76                        | ...                          |
| Option 5 (0.1 psi)         |  |                           |                             |                              |
| Mat 10, Pile               | 1  | 24.37                     | 24.37                       | 26.03                        |
|                            | 2  | 17.23                     | 17.23                       | 19.50                        |
|                            | 5  | 10.90                     | 10.90                       | 14.22                        |
|                            | 10   | 9.71                      | 7.71                        | 11.95                        |
| Mat 14, Pile               | 1  | 3.18                      | 3.21                        | 3.75                         |
|                            | 2  | 2.25                      | 2.30                        | 3.00                         |
|                            | 5  | 1.42                      | 1.49                        | 2.44                         |
|                            | 10   | 1.01                      | 1.10                        | 2.22                         |
| Option 5 (1.1 psi)         |  |                           |                             |                              |
| Mat 10, Pile               | 1  | 12.84                     | 12.84                       | 30.75                        |
|                            | 2  | 9.08                      | 9.08                        | 29.38                        |
|                            | 5  | 5.74                      | 5.74                        | 28.52                        |
|                            | 10   | 4.06                      | 4.06                        | 28.23                        |
| Mat 14, Pile               | 1  | 4.18                      | 4.28                        | 4.35                         |
|                            | 2  | 2.95                      | 3.09                        | 3.19                         |

**TABLE 4 Continued**

| Test Options and Materials | Critical Differences for the Conditions Noted <sup>A</sup> |                           |                             |                              |
|----------------------------|--|---------------------------|-----------------------------|------------------------------|
|                            | Number of Observations in Each Average                     | Single-Operator Precision | Within-Laboratory Precision | Between-Laboratory Precision |
|                            | 5  | 1.87                      | 2.08                        | 2.23                         |
|                            | 10   | 1.32                      | 1.61                        | 1.79                         |

<sup>A</sup> The critical differences were calculated using  $t = 1.960$ , which is based on infinite degrees of freedom.

12.2.4 For computer-processed data, identify the program (software) used.

12.2.5 Any modification of this test method.

### 13. Precision and Bias <sup>5</sup>

13.1 *Summary*—In comparing two averages, the differences should not exceed the single-operator precision values shown in **Table 3** for the respective number of tests and for materials having averages similar to those shown in **Table 1** in 95 out of 100 cases when all the observations are taken by the same well-trained operator using the same piece of equipment and specimens randomly drawn from the sample of material. Larger differences are likely to occur under all other circumstances.

13.2 *Interlaboratory Test Data*—An interlaboratory test was run in 1994–1995 in which randomly drawn samples were tested as specified in the five options of this test method. Two operators in each laboratory each tested ten specimens of each material. Five of the ten specimens were tested on one day, and five specimens were tested on a second day. Analysis of the data was conducted in accordance with Practices **D 2904** and **D 2906**, as well as the adjunct Tex-Pac. The components of variance for thickness, expressed as standard deviations, were calculated to be the values listed in **Table 3**. The fabric types are shown in **Tables 3 and 4**. The designated options and number of participating laboratories are shown as follows:

| Option | Number of Laboratories |
|--------|------------------------|
| 1      | 6                      |
| 2      | 4                      |
| 3      | 3                      |
| 4      | 1                      |
| 5      | 2                      |

13.3 *Precision*—For the components of variance reported in **Table 3**, two averages of observed values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed in **Table 4**. There were sufficient differences related to the material type and structure to warrant listing the components of variance and the critical differences separately. Consequently, no multi-material comparisons were made.

NOTE 2—Since the interlaboratory tests for Options 2, 3, 4, and 5 included less than five laboratories, estimates of between-laboratory precision should be used with special caution.

NOTE 3—The tabulated values of the critical differences should be

<sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D13–1110.

considered to be a general statement, particularly with respect to between-laboratory precision. Before a meaningful statement can be made about two specific laboratories, the amount of statistical bias, if any, between them must be established, with each comparison being based on recent data obtained on specimens taken from a lot of material to the type being evaluated so as to be as nearly homogeneous as possible, and then randomly assigned in equal numbers to each of the laboratories.

13.4 *Bias*—The value of thickness of fabrics only can be defined in terms of a test method. Within this limitation, this test method has no known bias.

#### **14. Keywords**

14.1 fabric; thickness

 **D 1777 - 96 (2007)**  
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## Standard Test Methods for Pore Size Characteristics of Membrane Filters by Bubble Point and Mean Flow Pore Test<sup>1</sup>

This standard is issued under the fixed designation F316; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reappraisal. A superscript epsilon (  $\epsilon$  ) indicates an editorial change since the last revision or reappraisal.

### 1. Scope

1.1 These test methods cover the determination of two of the pore size properties of membrane filters with maximum pore sizes from 0.1 to 15.0  $\mu\text{m}$ .

1.2 Test Method A presents a test method for measuring the maximum limiting pore diameter of nonfibrous membranes. The limiting diameter is the diameter of a circle having the same area as the smallest section of a given pore (Fig. 1).

1.3 Test Method B measures the relative abundance of a specified pore size in a membrane, defined in terms of the limiting diameter.

1.4 The analyst should be aware that adequate collaborative data for bias statements as required by Practice D2777 is not provided. See the precision and bias section for details.

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

E128 Test Method for Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use

### 3. Terminology

3.1 *Definitions*—For definitions of other terms used in these test methods, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.08 on Membranes and Ion Exchange Materials.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

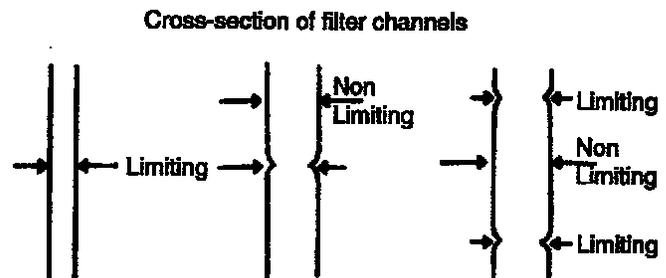


FIG. 1 Examples of Limiting Diameters

3.2.1 *pore size*—capillary equivalent pore diameter.

3.2.2 *limiting pore diameter*—diameter of a circle having the same area as the smallest section of a given pore.

### TEST METHOD A—MAXIMUM PORE SIZE

#### 4. Summary of Test Method

4.1 The bubble point test for maximum pore size is performed by prewetting the filter, increasing the pressure of gas upstream of the filter at a predetermined rate and watching for gas bubbles downstream to indicate the passage of gas through the maximum diameter filter pores.

4.2 The pressure required to blow the first continuous bubbles detectable by their rise through a layer of liquid covering the filter is called the  $\text{\textcircled{b}}$  bubble point $\text{\textcircled{b}}$ , and is used to calculate maximum pore size.

#### 5. Significance and Use

5.1 This test method may be used to:

5.1.1 Determine the maximum pore size of a filter,

5.1.2 Compare the maximum pore sizes of several filters, and

5.1.3 Determine the effect of various processes such as filtration, coating, or autoclaving on the maximum pore size of a membrane.

5.2 Membrane filters have discrete pores from one side to the other of the membrane, similar to capillary tubes. The bubble point test is based on the principle that a wetting liquid is held in these capillary pores by capillary attraction and surface tension, and the minimum pressure required to force liquid from these pores is a function of pore diameter. The



pressure at which a steady stream of bubbles appears in this test is the bubble point pressure. The bubble point test is significant not only for indicating maximum pore size, but may also indicate a damaged membrane, ineffective seals, or a system leak.

5.3 The results of this test method should not be used as the sole factor to describe the limiting size for retention of particulate contaminants from fluids. The effective pore size calculated from this test method is based on the premise of capillary pores having circular cross sections, and does not refer to actual particle size retention. See Test Method E128 for additional information.

### 6. Apparatus

6.1 *Filter Holder*, as shown in Fig. 2, consisting of a base A, a locking ring B, O-ring seal C, support disk D, and gas inlet E. The support disk shall be 2-ply construction, consisting of a 100 by 100 mesh or finer screen and a perforated metal plate for rigidity. The diameter of the test filter may be either 25 or 47 mm, as appropriate to the holder being used for the test.

6.2 *Manifold*, as shown in Fig. 3, a micrometric flow control valve capable of providing a linear rise in pressure and a gas ballast of at least 16 000-cm<sup>3</sup> capacity.

NOTE 1—For less accurate determinations, the simplified apparatus shown in Fig. 4 may be used.

6.3 *Pressure Gages* (and mercury manometer if required), covering the range of pressures needed for the pore sizes under investigation (see Table 1).

6.4 *Metal Punch*, used to cut a suitable size filter from the test sheet to fit the test filter holder.

### 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society

where such specifications are available.<sup>3</sup> Other grades may be used provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

7.2 *Water*, conforming to Specification D1193, Type IV or higher purity.

7.3 *Denatured Alcohol*.

7.4 *Petroleum Distillate*, with surface tension of 30 dynes/cm at 25°C.

7.5 *Mineral Oil*, such as USP liquid petrolatum heavy, with surface tension of 34.7 dynes/cm at 25°C.

7.6 *1,1,2-trichloro-1,2,2-trifluoroethane (Freon TFt)*, available from commercial chemical supply houses.

7.7 *Clean Gas Pressure Source*, with regulation (filtered air or nitrogen).

NOTE 2—Table 1 lists the nominal surface tension of these liquids at 25°C. Table 2 lists the simplified maximum pore size formulas based on these values, where the liquid completely wets the membrane.

### 8. Procedure

8.1 Wet the test membrane completely by floating it on a pool of the liquid. Use a vacuum chamber to assist in wetting the filter, if needed.

8.2 Place the wet membrane in the filter holder.

8.3 Close the filter holder and apply slight gas pressure to eliminate possible liquid back flow.

8.4 Cover the perforated metal plate with 2 to 3 mm of test liquid.

8.5 Increase the gas pressure slowly. Record the lowest pressure at which a steady stream of bubbles rises from the central area of the liquid reservoir.

NOTE 3—Faulty sealing may cause erroneous bubbling from the sealing edge of the liquid reservoir. Be sure to record the bubble point pressure with bubbles from the central area of the reservoir (see Fig. 5).

### 9. Calculation

9.1 If the test liquid is known to wet the membrane completely, calculate the maximum pore size from the following equation:

$$d = 5 Cg/p \tag{1}$$

where:

$d$  = limiting diameter,  $\mu\text{m}$ ,

$g$  = surface tension,  $\text{mN/m}$ , (dynes/cm),

$p$  = pressure, Pa or cm Hg, and

$C$  = constant, 2860 when  $p$  is in Pa, 2.15 when  $p$  is in cm Hg, and 0.415 when  $p$  is in psi units.

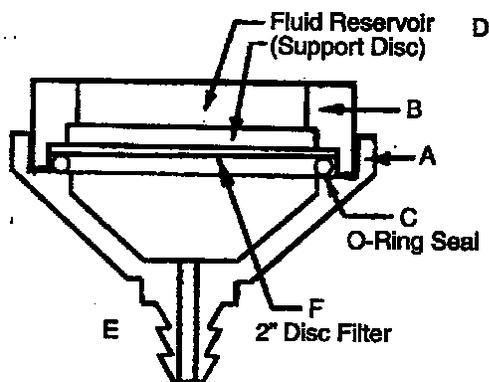
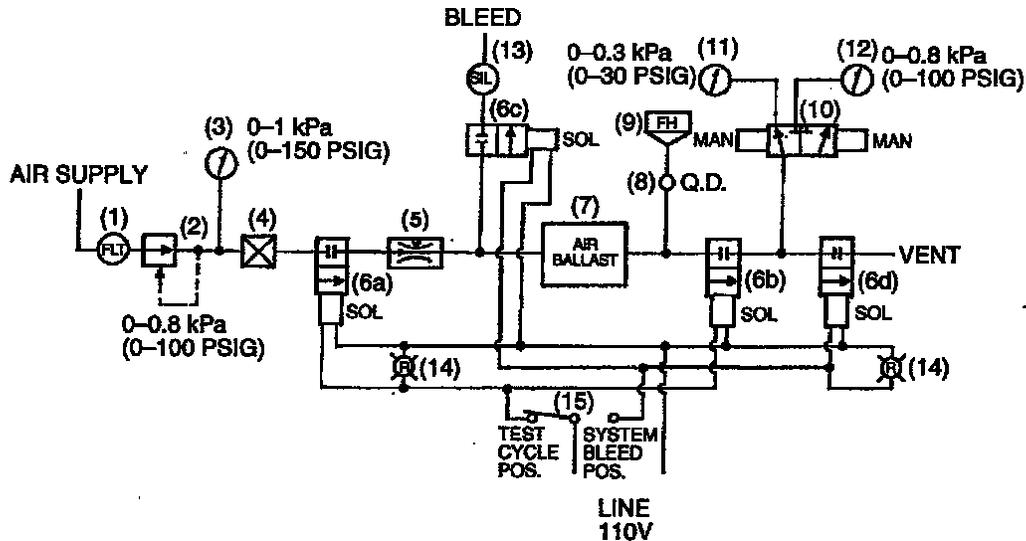


FIG. 2 Filter Holder

<sup>3</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.



| Key | Quantity | Component                         |
|-----|----------|-----------------------------------|
| 1   | 1        | Filter                            |
| 2   | 1        | Pressure regulator                |
| 3   | 1        | Pressure gage                     |
| 4   | 1        | Valve shutoff, manual             |
| 5   | 1        | Valve, flow control, manual       |
| 6   | 4        | Valve, solenoid, nc               |
| 7   | 1        | Air ballast                       |
| 8   | 1        | Quick disconnect fitting          |
| 9   | 2        | Open filter holder, 47 mm         |
| 10  | 1        | Valve, 3-way, manual              |
| 11  | 1        | Test gage, 0-0.3 kPa (0-30 psig)  |
| 12  | 1        | Test gage, 0-0.8 kPa (0-100 psig) |
| 13  | 1        | Exhaust silencer                  |
| 14  | 2        | Pilot light, red, elec.           |
| 15  | 1        | Switch, spdt, elec.               |

FIG. 3 Manifold for Bubble Point Testing

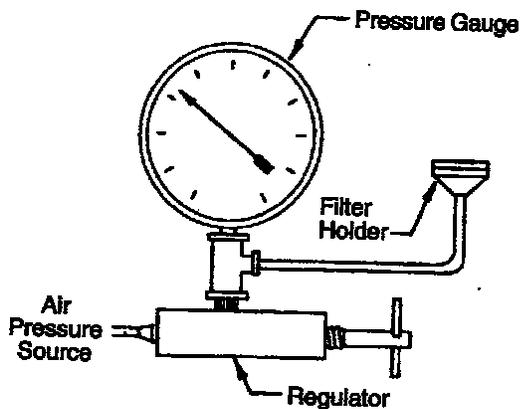


FIG. 4 Test Setup (Simplified)

NOTE 4—The fluid must completely wet the membrane filter with the contact angle being zero. If the contact angle is greater than zero, the calculated effective pore size will be larger than the actual effective pore size rating.

### 10. Reporting Results

10.1 Record the minimum pressure for gas passage as indicated by continuous bubbles. Record the maximum pore

size calculated, along with identification of the membrane tested and the liquid used.

### TEST METHOD B—DETERMINATION OF PORE SIZE DISTRIBUTION

#### 11. Summary of Test Method

11.1 A fluid-wet filter will pass air when the applied air pressure exceeds the capillary attraction of the fluid in the pores. Smaller pores will exhibit similar behavior at higher pressures. The relationship between pore size and pressure has been established, as indicated in Table 2.

11.2 By comparing the gas flow rates of both a wet and dry filter at the same pressures, the percentage of the flow passing through the filter pores larger than or equal to the specified size may be calculated from the pressure-size relationship. By increasing pressure in small steps, it is possible to determine the flow contribution of very small pore size increments by difference.

11.3 To determine the mean flow pore size, a pressure is sought at which the wet filter flow is one half of the dry filter flow.

#### 12. Significance and Use

12.1 This test method may be used to:

**TABLE 1 Pressure Ranges Required**

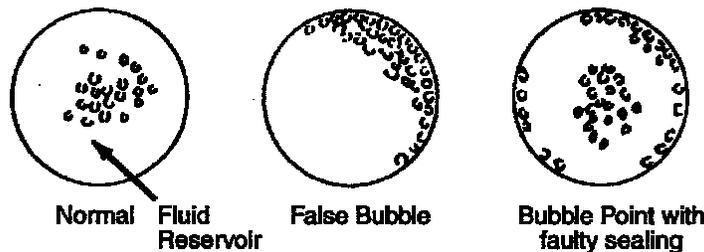
| Fluid Used           | Pore Size Range to be Investigated (Alternative psi) |                                 |                                  |                                   | Surface Tension, dynes/cm at 25°C |
|----------------------|--|---------------------------------|----------------------------------|-----------------------------------|-----------------------------------|
|                      | \$1 μm   | \$0.5 μm                        | \$0.1 μm                         | \$0.05 μm                         |                                   |
| Water                | 0 to 155 cm Hg<br>(0 to 30 psi)                      | 0 to 310 cm Hg<br>(0 to 60 psi) | 0 to 775 cm Hg<br>(0 to 150 psi) | 0 to 3100 cm Hg<br>(0 to 600 psi) | 72.0                              |
| Petroleum distillate | 0 to 65 cm Hg<br>(0 to 15 psi)                       | 0 to 130 cm Hg<br>(0 to 25 psi) | 0 to 325 cm Hg<br>(0 to 65 psi)  | 0 to 1300 cm Hg<br>(0 to 250 psi) | 30.0                              |
| Denatured alcohol    | 0 to 50 cm Hg<br>(0 to 10 psi)                       | 0 to 100 cm Hg<br>(0 to 20 psi) | 0 to 250 cm Hg<br>(0 to 50 psi)  | 0 to 1000 cm Hg<br>(0 to 200 psi) | 22.3                              |
| Mineral oil          | 0 to 75 cm Hg<br>(0 to 15 psi)                       | 0 to 150 cm Hg<br>(0 to 30 psi) | 0 to 305 cm Hg<br>(0 to 75 psi)  | 0 to 1500 cm Hg<br>(0 to 300 psi) | 34.7                              |
| Freon TFI            | 0 to 40 cm Hg<br>(0 to 8 psi)                        | 0 to 80 cm Hg<br>(0 to 16 psi)  | 0 to 200 cm Hg<br>(0 to 40 psi)  | 0 to 800 cm Hg<br>(0 to 100 psi)  | 17.3                              |

**TABLE 2 Calculation of Maximum Pore Size from Bubble Point Pressure,<sup>A</sup>  $d = C1/P$**

NOTE—Refer to [Appendix X1](#) for derivation of maximum pore size formula.

| Fluid Used           | Pressure Units of Bubble Point |                   |                                  |
|----------------------|--------------------------------|-------------------|----------------------------------|
|                      | cm Hg                          | psi               | Pa                               |
| Water                | 155/Bubble Point               | 30.0/Bubble Point | 2.06 $\times 10^5$ /Bubble Point |
| Petroleum distillate | 64.6/Bubble Point              | 12.5/Bubble Point | 8.58 $\times 10^4$ /Bubble Point |
| Denatured alcohol    | 47.75/Bubble Point             | 9.25/Bubble Point | 6.38 $\times 10^4$ /Bubble Point |
| Mineral oil          | 74.5/Bubble Point              | 14.4/Bubble Point | 9.92 $\times 10^4$ /Bubble Point |
| Freon TFI            | 37.2/Bubble Point              | 7.2/Bubble Point  | 4.95 $\times 10^4$ /Bubble Point |

<sup>A</sup> Example—A certain filter was observed to have a bubble point of 41.0 psi with petroleum distillate:  
 Size (μm) = 12.5/41.0 psi  
 Therefore, maximum pore size = 0.305 μm.



**FIG. 5 Examples of Erroneous Bubble Points**

- 12.1.1 Determine the pore size distribution of a membrane filter,
- 12.1.2 Determine the mean flow pore size of a membrane filter,
- 12.1.3 Determine the effect of processes such as sterilization, upon the pore size distribution,
- 12.1.4 Measure the maximum pore size of a membrane filter, and
- 12.1.5 Determine the gas flow rate of a filter, and thereby its gas flow capability.

**13. Apparatus**

- 13.1 *Clean Gas Pressure Source*, with regulation (filtered air or nitrogen).
- 13.2 *Pressure Gage* (or set of gages), covering the necessary pressure range for the pore sizes under study. (See [Table 1](#).)
- 13.3 *Closed Filter Holder*, (see [Fig. 6](#)).

NOTE 5—Two 1-in. (25-mm) filter holders, instead of one holder, eliminate the task of cleaning the filter holder after a fluid-wet air flow

determination. A two-position gas valve to switch the gas pressure from dry to wet holders may be used (see [Fig. 7](#)).

- 13.4 *Set of Rotameters*, covering the range from 0 to 100 L/min.
- 13.5 *Test Tube and Capillary Tube*, to serve for detection of the bubble point.
- 13.6 *In-Line Fluid Trap*, to protect the rotameters from the fluid.
- 13.7 *Appropriate Fittings, Hose, Connectors, Piping*, to assemble apparatus as shown in [Figs. 6 and 7](#).
- 13.8 *x-y Recorder*, to assist in graphing results of mean flow pore test.

**14. Reagents**

- 14.1 *Mineral Oil*, such as USP liquid petrolatum heavy.

**15. Procedure for One Holder ( [Fig. 6](#) )**

- 15.1 Place a dry filter sample disk in the filter holder.
- 15.2 Close the holder and apply gas pressure in increments.

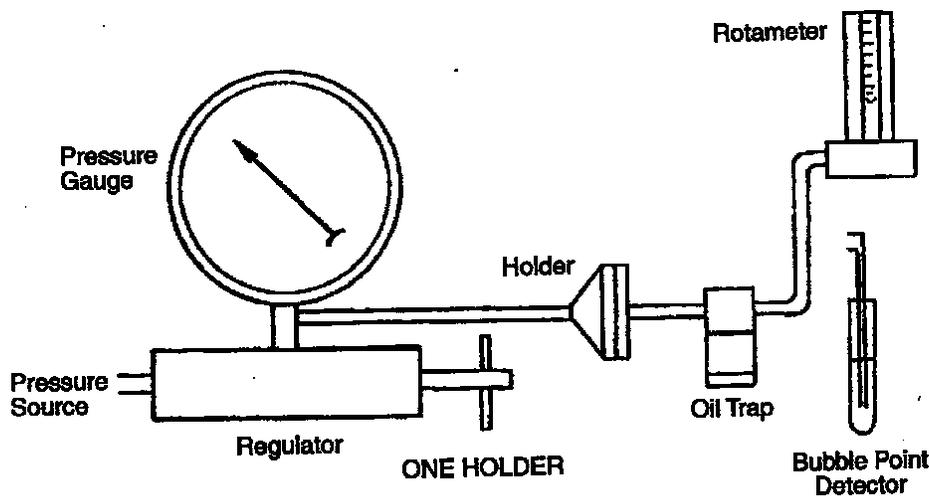


FIG. 6 Setup for One Holder

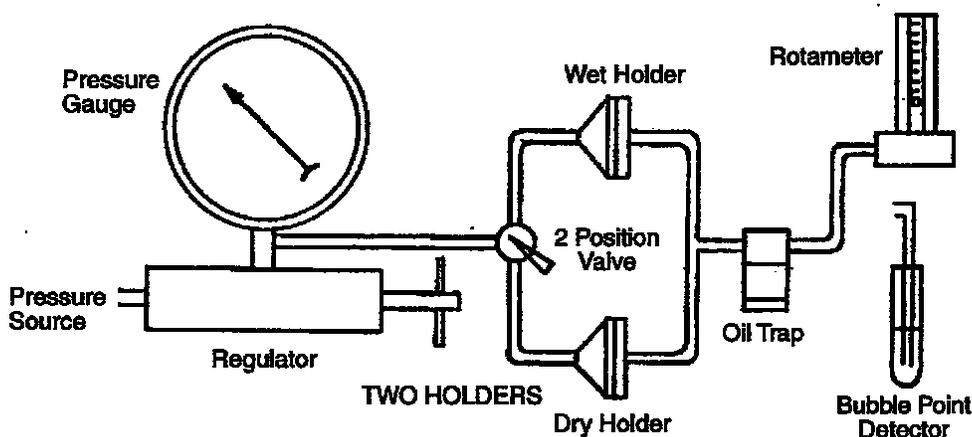


FIG. 7 Setup for Two Holders

15.3 Plot gas flow versus gas pressure over the intended range of use. (Estimate from pore size-pressure formula, see Table 1.)

15.4 Reduce gas pressure and remove the filter from the holder.

15.5 Completely wet the filter in mineral oil, replace the wet filter in the holder, and apply a slight pressure, checking to see that a tight seal exists.

15.6 Increase pressure gradually and record the pressure of the first gas flow detected by the capillary tube tip immersed in the test tube half-filled with mineral oil.

15.7 Change to rotameters and plot the fluid-wet filter gas flow versus pressure on the same coordinates as for the plot made in accordance with 15.3.

15.8 Reduce the pressure, remove the filter, and clean the holder for the next test.

**16. Procedure for Two Holders ( Fig. 7 )**

16.1 Place a dry filter in the holder to be used exclusively for dry filters.

16.2 Wet a filter of the same type and lot as the filter used in 16.1, in mineral oil and place it in a holder to be used exclusively for wet filters.

16.3 Apply gas pressure to the dry filter and plot gas flow versus gas pressure.

16.4 Change the two position valve to apply gas pressure to the wet filter holder and record the first gas flow as detected by the capillary tube as the bubble point. Switch to rotameters and plot fluid-wet gas flow versus gas pressure.

**17. Calculation of Mean Flow Pore Size**

17.1 Record the minimum pressure for gas passage (bubble point pressure). Calculate the maximum pore size from the formula in 9.1 and correlation data in Table 2.

17.2 Using the graph from 16.3, draw the line (or curve) corresponding to one half the dry gas-flow rate of the tested filter. Find the intersection of this (one-half dry-flow) line and the wet flow line (or curve), as shown in Fig. 8. Determine the pressure coordinate of the intersection and substitute into the pressure-pore size formula. (See Table 2 for mineral oil.)

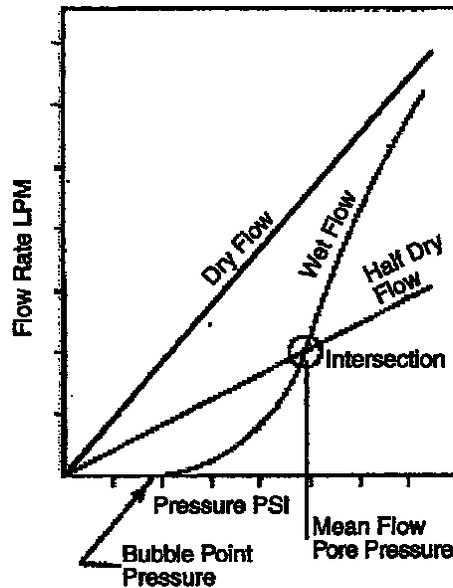


FIG. 8 Example of Mean Flow Pore Determination

18. Calculation of Pore Size Frequency

18.1 Select the limits of the pore size range being evaluated. Substitute the limits individually into the pore-size versus pressure formula and obtain their respective pressures. From the test graph, determine the wet and dry flows at the pressure limits (pore size limits of the range) as shown in Fig. 9.

18.2 Substitute the values found in the following equation:

$$Q = FS \left( \frac{\text{wet flow}_h}{\text{dry flow}_h} \right) \geq S \left( \frac{\text{wet flow}_l}{\text{dry flow}_l} \right) \geq 100 \quad (2)$$

where:

- Q = filter flow, %
- l = lower pressure limit, and
- h = higher pressure limit.

18.3 Report Q as the percentage of the filter flow passing through pores within the range specified.

NOTE 6—Example—Determine the percentage of the filter flow passing

through a pore size range from 0.8 to 0.2 μm of the filter described in Fig. 9.

$$Q = FS \left( \frac{7 \text{ L/min}}{21 \text{ L/min}} \right) \geq S \left( \frac{2 \text{ L/min}}{15 \text{ L/min}} \right) \geq 100$$

$$= 0.333 \geq 0.133 \geq 100$$

$$= 20\% \quad (3)$$

Twenty percent of the gas passing through the filter described in Fig. 9 moves through pores between 0.8 and 0.2 μm.

19. Precision and Bias

19.1 Results should not differ from the mean by more than the following amounts:

| Pore Size Range   | Repeatability, %<br>Same Operator<br>and Apparatus | Reproducibility, %<br>Different Operator<br>and Apparatus |
|-------------------|--|---|
| Greater than 1 μm | 2  | 4   |
| 1 to 0.5 μm       | 1  | 2.5   |
| Less than 0.5 μm  | 0.5  | 1   |

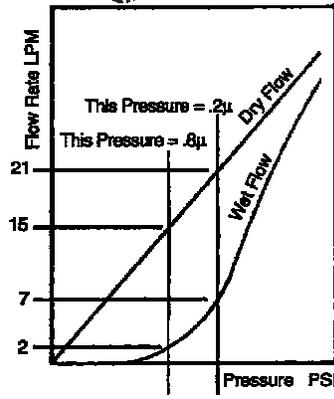


FIG. 9 Example of Pore Size Frequency Determination

FIG. 9 Example of Pore Size Frequency Determination

19.2 Since the change in pore size per unit change in applied pressure is greatest at large pore sizes and diminishes hyperbolically, repeatability and reproducibility increase accordingly with applied pressure.

## APPENDIX

### (Nonmandatory Information)

#### X1. PORE SIZE FORMULA DERIVATION

X1.1 The pore size formula is derived as follows:

Surface tension formula:

$$g = \frac{2 \cos \theta}{d} \frac{B}{r} \tag{X1.1}$$

Rearranging for diameter (2r):

$$d = \frac{4 \cos \theta B}{g} \tag{X1.2}$$

At bubble point pressure  $P = hgp$ :

$$d = \frac{4 \cos \theta B g}{P} \tag{X1.3}$$

When a fluid wets a filter  $\cos \theta = 1$ :

$$d = \frac{4 B g}{P} \tag{X1.4}$$

where:

$h$  = height of rise,  
 $p$  = density of fluid,

$g$  = gravitational constant,

$r$  = radius of tube,

$\theta$  = contact angle,

$B$  = capillary constant, and

$g$  = surface tension.

Substituting pressure conversion factors and the capillary constant:

For centimetres of Hg:

$$d = \frac{2.145 \text{ g-dynes/cm}^2}{P \text{ cm Hg}} \tag{X1.5}$$

For psi:

$$d = \frac{0.415 \text{ g-dynes/cm}^2}{P \text{ psi}} \tag{X1.6}$$

For Pa:

$$d = \frac{2860 \text{ g-dynes/cm}^2}{P \text{ Pa}} \tag{X1.7}$$

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**E8 Federal Ambient EPA Filter Requirements: 2.12. Quality Assurance  
Guidance Document 2.12: Monitoring PM<sub>2.5</sub> in Ambient Air using  
Designated Reference or Class I Equivalent Methods**

United States  
Environmental Protection  
Agency

Human Exposure and Atmospheric Sciences Division  
National Exposure Research Laboratory  
Research Triangle Park NC 27711

Research and Development

November 1998



# Quality Assurance Guidance Document 2.12

## Monitoring PM<sub>2.5</sub> in Ambient Air Using Designated Reference or Class I Equivalent Methods

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## List of Acronyms and Abbreviations

|                   |   |
|-------------------|---|
| AIRS              | Aerometric Information Retrieval System             |
| ANSI              | American National Standards Institute               |
| ASQC              | American Society for Quality Control                |
| ASTM              | American Society for Testing and Materials          |
| CFR               | <i>Code of Federal Regulations</i>                  |
| CV                | coefficient of variation                            |
| DOP               | dioctylphthalate                                    |
| DQO               | data quality objective                              |
| EPA               | Environmental Protection Agency                     |
| FEM               | Federal Equivalent Method                           |
| FR                | flow rate   |
| FRM               | Federal Reference Method                            |
| HEPA              | high-efficiency particulate air                     |
| NAAQS             | National Ambient Air Quality Standards              |
| NIST              | National Institute of Standards and Technology      |
| NVLAP             | National Voluntary Laboratory Accreditation Program |
| OAQPS             | Office of Air Quality Planning and Standards        |
| $P_a$             | pressure, ambient                                   |
| PAMS              | Photochemical Assessment Monitoring Stations        |
| $PM_{10}$         | particulate matter #10 $\mu\text{m}$                |
| $PM_{2.5}$        | particulate matter #2.5 $\mu\text{m}$               |
| PMP               | polymethylpentene                                   |
| $^{210}\text{Po}$ | polonium-210  |
| PSD               | prevention of significant deterioration             |
| PTFE              | polytetrafluoroethylene                             |
| QA/QC             | quality assurance/quality control                   |
| QA                | quality assurance                                   |
| QAD               | Quality Assurance Division                          |
| QAPP              | quality assurance project plan                      |
| QC                | quality control                                     |
| RH                | relative humidity                                   |
| SLAMS             | State and local air monitoring stations             |
| SOP               | standard operating procedure                        |
| SRM               | Standard reference material                         |
| SVOC              | semivolatile organic compound                       |
| $T_a$             | temperature, ambient                                |
| TSP               | total suspended particulate                         |
| WINS              | well impactor ninety-six                            |

## 1.0 Scope and Applicability

### 1.1 Overview and Purpose

This document is intended to assist personnel of air monitoring agencies that use reference methods or Class I equivalent methods to monitor ambient air for particles with an aerodynamic diameter equal to or less than 2.5  $\mu\text{m}$ , known as  $\text{PM}_{2.5}$ . The formal specifications for  $\text{PM}_{2.5}$  reference method samplers and for obtaining valid reference method measurements are set forth in the U.S. Environmental Protection Agency's (EPA's) monitoring regulations at 40 *Code of Federal Regulations* Part 50, Appendix L (EPA 1997a).<sup>\*</sup> This document reviews those formal requirements and provides clarifications and supplemental information in greater detail than can be provided in the formal regulatory requirements. Because this supplemental information is intended for method users, it emphasizes the operational aspects of the method, rather than the design and performance specifications for the sampler, which are of more concern to the sampler manufacturer. This document also provides recommended quality assurance (QA) procedures and guidance to help monitoring agencies reliably achieve the data quality objectives (DQOs) established for  $\text{PM}_{2.5}$  monitoring. The goal for acceptable measurement uncertainty has been defined as 10 percent coefficient of variation (CV) for total precision and  $\pm 10$  percent for total bias (Papp et al. 1998). The information provided here, together with the more sampler-specific information and instructions provided by the manufacturer of the selected  $\text{PM}_{2.5}$  sampler and contained in the sampler's operation or instruction manual, should be used by each monitoring agency to develop its own agency-specific standard operating procedure (SOP) to govern its individual  $\text{PM}_{2.5}$  monitoring activity.

### 1.2 Applicability

This document is primarily applicable to  $\text{PM}_{2.5}$  ambient air monitoring with reference methods carried out by State and local air monitoring agencies in their State and local air monitoring stations' (SLAMS's) air surveillance networks under the air monitoring requirements of 40 CFR Part 58 (EPA 1997b). The document is also applicable to other organizations required to carry out SLAMS or SLAMS-related  $\text{PM}_{2.5}$  monitoring and to any  $\text{PM}_{2.5}$  monitoring activity for which the  $\text{PM}_{2.5}$  reference method measurements will be entered into the Aerometric Information and Retrieval System (AIRS) database. The recommendations and guidance in this document—to the extent that they go beyond the specific regulatory requirements set forth in the method or in the sampler-specific requirements contained in a particular sampler's operation or instruction manual—are not mandatory or binding. However, monitoring agencies are strongly encouraged to adopt and follow these recommendations and guidance to help ensure that monitoring data are of acceptable quality. Following the recommendations and guidance herein is also strongly encouraged for any  $\text{PM}_{2.5}$  monitoring activity using reference or Class I equivalent methods where a high level of data quality is needed.

This document is not a full description of a  $\text{PM}_{2.5}$  monitoring method and does not substitute for the formal method description as set forth in the  $\text{PM}_{2.5}$  reference method (EPA 1997a) or for the

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<sup>\*</sup>All references are listed in Section 14.0.

sampler-specific requirements contained in the associated operation or instruction manual, which is an official part of each designated reference or equivalent method for PM<sub>2.5</sub>.

### 1.3 Conventions

Where this document refers to mandatory method requirements, the terms “shall” and “must” are used, and a supporting reference to the applicable section of the reference method regulation is generally provided. Use of the word “should” indicates an activity or procedure that is strongly recommended to help achieve a high level of measurement data quality but is not formally required by the method itself. Finally, the word “may” is used to indicate activities or suggestions that are optional or discretionary. The use of these terms is generally consistent with American National Standards Institute/American Society for Quality Control (ANSI/ASQC) E4-1994 guidance (ANSI/ASQC 1994).

### 1.4 Format and Structure

The format and structure of this document are somewhat different from the format and structure of previously published handbook documents. The page format has been updated to a new uniform standard that will be used for future sections of the *Quality Assurance Handbook for Air Pollution Measurement Systems*. It features a common, easily read font for text and a new standard bold sans serif font for headings. New uniform header and footer information indicates the organizational position of each page in the overall handbook structure, giving the volume number, document number, and subject identification; the major section number and title; the section page number; and the revision date.

The organization of the document has also been modified somewhat from that used previously, with some new or changed section titles and some entirely new section topics. These changes are intended to align the structure more closely with the organizational structure and topics of SOPs recommended by EPA’s Quality Assurance Division (QAD) in its G-6 guidance (EPA 1995). This closer alignment will help monitoring agencies use this document to prepare their individual SOPs for PM<sub>2.5</sub> monitoring. It should be clearly understood, however, that this document is not a ready-made, generic SOP as is and that it must be adapted and tailored specifically to each monitoring agency’s individual policies and circumstances to become an official agency SOP.

### 1.5 Overview of Reference and Equivalent Methods for PM<sub>2.5</sub>

A “method” for PM<sub>2.5</sub> generally consists of the following:

- A PM<sub>2.5</sub> sampler or analyzer designed, built, and sold by a particular manufacturer
- An operation or instruction manual provided by the instrument manufacturer that describes the proper use and operation of the sampler or analyzer
- The other operational and quality assurance requirements necessary to obtain reliable PM<sub>2.5</sub> concentration measurements.

Methods used for monitoring PM<sub>2.5</sub> in SLAMS or SLAMS-related air monitoring are required to be either reference or equivalent methods (EPA 1997c) as designated by EPA under the requirements and provisions of 40 CFR Part 53 (EPA 1997d). For most SLAMS purposes, reference and equivalent methods may be used interchangeably, and a particular method’s

identity as either a reference method or an equivalent method is irrelevant. However, for some purposes, such as collocated auditing, a reference method may be specifically required. A current publication, *List of Designated Reference and Equivalent Methods*, identifying all methods that have been designated as reference or equivalent methods by EPA is available from any EPA Regional Office or by writing to Department E (MD-46), U.S. Environmental Protection Agency, Research Triangle Park, NC 27711. The U.S. EPA's Ambient Monitoring Technology Information Center (AMTIC) web site, <http://www.epa.gov/ttn/amtic/>, contains several sites with information on all the designated reference and equivalent methods for criteria pollutants and will include PM<sub>2.5</sub> methods as they are designated and updated. Refer to the "what's new" topic for the latest updates.

### 1.5.1 Reference Methods

All formal sampler design and performance requirements and the operational requirements applicable to reference methods for PM<sub>2.5</sub> are specified in Appendix L of 40 CFR Part 50 (EPA 1997a). These requirements are very specific and include explicit design specifications for the type of sampler, the type of filter, the sample flow rate, and the construction of the sample's collecting components. However, various designs for the flow rate control system, the filter holder, the operator interface controls, and the exterior housing are possible. Hence, various reference method samplers from different manufacturers may vary considerably in appearance and operation. Also, a reference method may have a single filter capability (single sample sampler) or a multiple filter capability (sequential sample sampler), provided there are no deviations in the design and construction of the sample collection components specified in the reference method regulation. A PM<sub>2.5</sub> method is not a reference method until it has been demonstrated to meet all the reference method regulatory requirements and has been officially designated by EPA as a reference method for PM<sub>2.5</sub>.

### 1.5.2 Equivalent Methods

Equivalent methods for PM<sub>2.5</sub> have much wider latitude in their design, configuration, and operating principles than reference methods. These methods are not required to be based on filter collection of PM<sub>2.5</sub>; therefore, continuous or semicontinuous analyzers and new types of PM<sub>2.5</sub> measurement technologies are not precluded as possible equivalent methods. Equivalent methods are not necessarily required to meet all the requirements specified for reference methods, but they must demonstrate both comparability to reference method measurements and similar PM<sub>2.5</sub> measurement precision.

The requirements that some (but not all) candidate methods must meet to be designated by EPA as equivalent methods are specified in 40 CFR Part 53. To minimize the difficulty of meeting equivalent method designation requirements, three classes of equivalent methods have been established in the 40 CFR Part 53 regulations, based on the degree to which a candidate method deviates from the reference method requirements. All three classes of equivalent methods are acceptable for SLAMS or SLAMS-related PM<sub>2.5</sub> monitoring. But not all types of equivalent methods may be equally suited to specific PM<sub>2.5</sub> monitoring requirements or applications.

Class I equivalent methods are very similar to reference methods, with only minor deviations, and must meet nearly all of the reference method specifications and requirements. The requirements for designation as Class I equivalent methods are only slightly more extensive than the

designation requirements for reference methods. Also, because of their substantial similarity to reference methods, Class I equivalent methods operate very much the same as reference methods, and most of the information and guidance in this document is applicable to Class I equivalent methods.

Class II equivalent methods are filter-collection-based methods that differ more substantially from the reference method requirements. The requirements for designation as Class II methods may be considerably more extensive than for reference or Class I equivalent methods, depending on the specific nature of the variance from the reference method requirements. The information and guidance in this document may be only partially applicable to Class II equivalent methods. The operation or instruction manual associated with Class II equivalent methods will, therefore, have to be more detailed in some areas than for reference or Class I equivalent methods to provide information and guidance not covered by this document.

Class III equivalent methods cover any  $PM_{2.5}$  methods that cannot qualify as reference or Class I or II equivalent methods because of more profound differences from the reference method requirements. This class encompasses  $PM_{2.5}$  methods such as continuous or semicontinuous  $PM_{2.5}$  analyzers and potential new  $PM_{2.5}$  measurement technologies. The requirements for designation as Class III equivalent methods are the most extensive, and, because of the wide variety of  $PM_{2.5}$  measurement principles that could be employed for candidate Class III equivalent methods, the designation requirements are not explicitly provided in 40 CFR Part 53. For similar reasons, much of the information and guidance in this document may not be applicable to operation of Class III equivalent methods.

## 1.6 Limitations of $PM_{2.5}$ Reference and Class I Equivalent Methods

There are several conditions or effects that limit the degree to which a  $PM_{2.5}$  reference or Class I equivalent method can precisely determine the mass concentration of particulate matter in the atmosphere. Procedures to control such effects are discussed throughout this document. One limitation is maintaining the specified ambient air flow rate through the sampling inlet and filter assembly. The flow rate determines the size of the particles that are collected. The effects of this limitation are minimized by following sampler construction requirements and by employing procedures and checks to ensure the proper flow rate is maintained within close tolerances. Refer to Section 6.0, "Calibration Procedures," and to Section 8.0, "Field Operation," for guidance.

Another important limitation involves changes in the weight of a collected sample due to mishandling, chemical reactions, and volatilization. Handling procedures, choice of filter media, humidity and temperature control of the filter and sample during collection and subsequent processing, and promptness in weighing the sample following collection all help control filter artifacts. The chemical makeup of the  $PM_{2.5}$  particulate matter will vary with sampling location and source. Thus, the magnitude of  $PM_{2.5}$  weight changes due to chemical and physical processes will also vary with site location.

Weight loss due to mechanical removal of particles from the filter is minimized by carefully removing the filter, in its cassette, from the sampler and storing the filter/cassette in a protective container during transit to the weighing laboratory. Once the filter arrives at the laboratory, weight loss can be minimized by carefully removing the filter from the cassette, conditioning the filter, and neutralizing the static charge on the filter before weighing. Refer to Section 7.0, "Filter

### Preparation and Analysis.”

The choice of an essentially neutral Teflon<sup>®</sup> media filter as the collecting surface minimizes the weight gain that occurs when sulfate-containing particles form by chemical reactions of sulfur dioxide gas at the surface of alkaline media such as glass fiber filters. Appendix A to this document discusses this effect and includes a procedure for measuring the alkalinity of filters. If nitric acid vapor is present at a sampling location, it can deposit on a Teflon<sup>®</sup> filter and cause small weight gains in proportion to the amount of nitric acid present in the atmosphere (Lipfert 1994). This weight gain may not be controllable. Weight losses can occur due to thermal or chemical decomposition or evaporation of compounds like ammonium nitrate (NH<sub>4</sub>NO<sub>3</sub>), which releases ammonia and nitric acid as gases. Semivolatile organic compounds (SVOCs) may be part of the sample makeup; if so, they may evaporate and cause sample weight losses. Such weight losses are minimized or standardized by maintaining the sampler filter compartment's temperature near ambient conditions during the sampling process, keeping the sample cool during transport to the laboratory, and promptly conditioning and weighing the sample following its receipt in the laboratory. Weight gain or loss due to absorption or desorption of water vapor on the filter or on the particulate matter is minimized by specifying low moisture pickup for manufactured filters and by conditioning the filters within specified humidity and temperature ranges, both before use and after receipt from the field. Refer to Section 7.0, “Filter Preparation and Analysis,” for further guidance.

Errors in the gravimetric analysis of samples can also result from the buildup of electrostatic charges on filters during their manufacture or during sampling (Engelbrecht et al. 1980). This static buildup will interfere with microbalance weighing, but it can be reduced or eliminated by using Polonium-210 (<sup>210</sup>Po) antistatic strips before beginning the weighing process.

## 2.0 Prerequisites

### 2.1 Overview and Definitions

The air pollutant known as PM<sub>2.5</sub> is the most recent addition to the ambient air criteria pollutants, which are required by Federal law to be measured and reported on a nationwide basis. Regulations governing its measurement were effective September 16, 1997.

This document provides a review of monitoring requirements and offers guidance on ensuring the quality of the collection of samples, the determination of the volume of air sampled, and the gravimetric determination of the amount of PM<sub>2.5</sub> collected. From these data, the concentration of PM<sub>2.5</sub> particles in the ambient air can be calculated and expressed as micrograms per cubic meter ( $\mu\text{g}/\text{m}^3$ ) of air. This document addresses procedures that apply mainly to reference and Class I equivalent methods for sampling PM<sub>2.5</sub>. Although some of the terminology associated with the PM<sub>2.5</sub> sampling method may be familiar to those who have operated dichotomous samplers, an annotated listing of acronyms, abbreviations, specialized terms, and other expressions is given in Table 2-1 to familiarize the new user of PM<sub>2.5</sub> samplers with key terms.

### 2.2 Personnel Qualifications

General aspects of personnel qualifications, training, and guidance are discussed in Volume II, Part I, Section 4 of the *Quality Assurance Handbook for Air Pollution Measurement Systems* (EPA 1997). State and Local Air Monitoring Stations (SLAMS) reporting organizations or State agencies can use information from this source and specific information about PM<sub>2.5</sub> sampling and analysis to develop a training program for their employees.

The responsibility for ensuring adequate training of personnel rests with the organization's management. The Quality Assurance Coordinator of the organization should be involved as well. He or she should stress to management the need for adequate training and recommend that employees be tested or examined to evaluate the success of training and identify where further emphasis is needed.

#### 2.2.1 Laboratory Personnel Qualifications

All laboratory personnel should be familiar with general environmental laboratory procedures and techniques. Those who operate the microbalance in the laboratory must be very conscientious and attentive to details in order to report complete and high-quality PM<sub>2.5</sub> data. Persons involved with PM<sub>2.5</sub> laboratory operations should be trained to perform the following operations:

- Use common methods to determine temperature, pressure, and relative humidity (RH) in the laboratory
- Use microbalance and antistatic devices
- Label, organize, and archive filters and samples in the laboratory.

**Table 2-1. Definitions of Key Terms**

| Term  | Definition  |
|---|---|
| Accuracy  | The degree of agreement between an observed value and an accepted reference value; includes a combination of random error (precision) and systematic error (bias) components due to sampling and analytical operations. It is estimated by collocated sampling.   |
| Actual conditions                                 | The actual ambient temperature and pressure of a gas at the time its volume (or volumetric flow rate) is measured.  |
| Analyst   | A staff member who weighs the new and used filters and computes the concentration of PM <sub>2.5</sub> in µg/m <sup>3</sup> .   |
| American National Standards Institute (ANSI)      | Administrator and coordinator of the U.S. private-sector voluntary standardization system.  |
| American Society for Testing and Materials (ASTM) | A professional organization that develops and distributes protocols for testing and provides reference standards.   |
| ASTM Class 1 standards                            | The standards for weighing operations with a microbalance. Certified by ASTM.   |
| Bias  | The systematic or persistent distortion of a measurement process that causes errors in one direction.   |
| Cassette  | A device supplied with PM <sub>2.5</sub> samplers to allow a weighed Teflon® filter to be held in place in the sampler and manipulated before and after sampling without touching the filter and to minimize damage to the filter and/or sample during such activities.                                     |
| Class I equivalent method                         | An equivalent method for PM <sub>2.5</sub> based on a sampler that closely meets the specifications for reference method samplers (40 <i>Code of Federal Regulations</i> [CFR] Part 58, Appendix L), with only minor deviations, as defined by the U.S. Environmental Protection Agency (EPA).              |
| Class II equivalent method                        | An equivalent method for PM <sub>2.5</sub> , as defined by the U.S. EPA, that is based on a sampler having more significant deviations from reference method sampler specifications than a Class I equivalent method.   |
| Class III equivalent method                       | An equivalent method for PM <sub>2.5</sub> , as defined by EPA, that is based on a device that incorporates major modifications to the reference method sampler or on a fundamentally different design or principle of measurement.   |
| Collocation                                       | The placement of a second PM <sub>2.5</sub> sampler (known as the duplicate sampler) near a permanent network sampler (known as the primary sampler). Comparison of results from the two samplers is used to estimate the precision and bias of the primary sampler.  |
| Control chart                                     | A graphical presentation of quality control (QC) information over a period of time. If a procedure is "in control," the results usually fall within established control limits. The chart is useful in detecting defective performance and abnormal trends or cycles, which can then be corrected promptly. |

(continued)

**Table 2-1. Continued**

| Term                         | Definition   |
|------------------------------|--|
| Downtube                     | The vertically oriented tube that connects the PM <sub>2.5</sub> sampler inlet to sampler components inside the sampler case. To check the sample flow rate, the sampler inlet is removed from the downtube and a flow rate standard is connected in its place.  |
| Electrostatic charge buildup | A buildup of static electrical charge on an item, such as the PM <sub>2.5</sub> filter, which makes it difficult to handle, attracts or repels particles, and can influence its proper weighing.   |
| Equilibration chamber        | A clean chamber usually constructed of plastic or glass, held at near constant temperature and humidity, used to store and condition PM <sub>2.5</sub> filters until they and their collected particulate sample (if the filters have been exposed) have reached a steady state of moisture equilibration.   |
| Field blank filter           | New filters, selected at random, that are weighed at the same time that presampling weights are determined for a set of PM <sub>2.5</sub> filters and used for quality assurance (QA) purposes. These field blank filters are transported to the sampling site in the same manner as filters intended for sampling, installed in the sampler, removed from the sampler without sampling, stored in their protective containers inside the sampler's case at the sampling site until the corresponding exposed filter(s) is (are) retrieved, and returned for postsampling weighing in the laboratory, where they are handled in the same way as an actual sample filter and reweighed as a QC check to detect weight changes due to filter handling. |
| Flow adapter device          | A tight-fitting connecting device that is inserted in place of the PM <sub>2.5</sub> sampler inlet on the upper end of a sampler's downtube and used to connect a flow rate calibration or audit device to check the sample flow rate.   |
| Flow calibration device      | A National Institute of Standards and Technology (NIST)-traceable flow-determining apparatus (also called a flow rate standard) that is attached to the flow adapter device and used to assist in measuring and setting the volumetric flow rate of air into the sampler. Bubble flowmeters, piston flowmeters, and orifice devices are the most common types of flow rate standards.  |
| Flow check filter            | A standard PM <sub>2.5</sub> filter that is placed in the sampler during the flow check procedure; it is later discarded and not used for sampling.  |
| Impactor                     | An inertial particle-size separator. A PM <sub>2.5</sub> reference or Class I equivalent method sampler uses a specially shaped inlet followed by an impactor that allows only particulate matter of well-defined size ranges to penetrate to the filter collection portion of the collector.  |
| Impactor well                | That portion of the sampler inlet where particles larger than 2.5 μm impact and are held by a filter soaked with oil (tetramethyl tetraphenyltri-siloxane) such that they are removed from the sample air stream.  |

(continued)

Table 2-1. Continued

| Term   | Definition   |
|--|--|
| Laboratory blank filter                            | New filters that are weighed at the time of determination of the pre-sampling (tare) weight of each set of PM <sub>2.5</sub> filters intended for field use. These laboratory blank filters remain in the laboratory in protective containers during the field sampling and are reweighed in each weighing session as a QC check.  |
| Leak check   | Checks made to ensure there are no significant leaks into or within the sampler. An external leak check, which can be carried out automatically by the sampler, is used to detect leakage into the sampler system. An internal bypass leak check is used to determine if any portion of the flow is bypassing the sampler's filter.  |
| Mass reference standard                            | ASTM- or NIST-traceable weighing standards, generally in the range of weights expected for the filters.  |
| Microbalance                                       | A type of analytical balance that can weigh to the nearest 0.001 mg (that is, 1 µg or one-millionth g).  |
| Operator   | A staff member who services a PM <sub>2.5</sub> sampler in routine operation at a field site and who may also be responsible for transporting the sample filters to and from the laboratory.   |
| Orifice flow rate check device                     | One type of flow rate calibration or check device (transfer standard), often used in the field, based on an established relationship between flow rate and pressure drop across the orifice plate and often used in the field. An orifice's operating characteristics are determined in the laboratory using a flow rate standard such as a soap film flowmeter. Orifice devices generally require temperature and pressure corrections.   |
| $P_a$  | Local ambient (barometric) pressure.   |
| PM <sub>2.5</sub>                                  | Particulate matter (suspended in the atmosphere) having an aerodynamic diameter less than or equal to a nominal 2.5 µm, as measured by a reference method based on 40 CFR Part 50, Appendix L, and designated in accordance with 40 CFR Part 53.   |
| PM <sub>2.5</sub> sampler                          | A sampler used for monitoring PM <sub>2.5</sub> in the atmosphere that collects a sample of particulate matter from the air based on principles of inertial separation and filtration. The sampler also maintains a constant sample flow rate and may record the actual flow rate and the total volume sampled. PM <sub>2.5</sub> mass concentration is calculated as the weight of the filter catch divided by the sampled volume. A sampler cannot calculate PM <sub>2.5</sub> concentration directly. |
| Polonium-210 ( <sup>210</sup> Po) antistatic strip | A device containing a small amount of <sup>210</sup> Po that emits $\alpha$ particles (He <sup>2+</sup> ) that neutralize the static charge on filters, making them easier to handle and their weights more accurate.  |
| Precision  | A measure of random variation among individual measurements of the same property, usually under prescribed identical conditions. For ambient particulate concentration measurements, precision is usually expressed in terms of a standard deviation estimated by collocated sampling when data are reported to the Aerometric Intervention and Retrieval System (AIRS) database.  |

(continued)

Table 2-1. Continued

| Term  | Definition   |
|---|--|
| Primary National Ambient Air Quality Standard (NAAQS) for PM <sub>2.5</sub> | The NAAQS for PM <sub>2.5</sub> is defined in 40 CFR Part 50 as follows: (a) 15 µg/m <sup>3</sup> , annual mean, based on the 3-year average of the annual arithmetic mean PM <sub>2.5</sub> concentrations, spatially averaged across an area; (b) 65 µg/m <sup>3</sup> , 24-hour average, based on the 3-year average at the 98th percentile of 24-hour PM <sub>2.5</sub> concentrations at each population-oriented monitor within an area. |
| Polytetrafluoroethylene (PTFE)  | The polymer that is used to manufacture the 47-mm-diameter filters for PM <sub>2.5</sub> Federal Reference Method (FRM) and Federal Equivalent Method (FEM) samplers. Also known as Teflon®.   |
| Q <sub>a</sub>  | The sampler flow rate expressed at ambient (actual) conditions of temperature and pressure.  |
| QA supervisor or coordinator  | A staff member who assists in preparation of the reporting organization's quality plan, makes recommendations to management on quality issues (including training), oversees the quality system's control and audit components, and reports the results.   |
| Readability   | The smallest difference between two measured values that can be read on the microbalance display. The term "resolution" is a commonly used synonym.  |
| Repeatability   | A measure of the ability of a microbalance to display the same result in repetitive weighings of the same mass under the same measurement conditions. The term "precision" is sometimes used as a synonym.   |
| Standard conditions (EPA)   | EPA-designated atmospheric conditions of 1 atm pressure (760 mmHg) and 25 EC (298 K). Standard conditions are not used for reporting PM <sub>2.5</sub> data; actual [uncorrected] conditions must be used for reporting.   |
| T <sub>a</sub>  | The ambient temperature.   |
| Traceable   | A term signifying that a local standard has been compared and certified, either directly or by not more than one intermediate standard, to a NIST-certified primary standard, such as a thermometer or standard reference material (SRM).  |
| Transfer standard   | A reference standard device (for temperature, flow rate, and so on) that has been compared to a NIST reference standard and is subsequently used to calibrate, audit, or check other measurement systems or devices.   |
| Total suspended particulate (TSP)   | Particulate matter collected by a high-volume sampler, which has no particular particle size selectivity.  |
| V <sub>a</sub>  | An air volume measured or expressed at ambient (actual) conditions of temperature and pressure.  |
| WINS  | The name given to the design of the particle impactor specified by EPA for reference method samplers for PM <sub>2.5</sub> ; an acronym for "Well Impactor Ninety Six."  |

### 2.2.2 Field Personnel Qualifications

All field operations personnel should be familiar with environmental field measurement techniques. Those who service the PM<sub>2.5</sub> sampler in the field must be very conscientious and attentive to detail in order to report complete and high-quality PM<sub>2.5</sub> data. Persons qualified to perform PM<sub>2.5</sub> field operations should be able to

- Operate the PM<sub>2.5</sub> sampler
- Calibrate, audit, and troubleshoot the PM<sub>2.5</sub> sampler
- Use common methods to determine temperature, pressure, flow rate, and RH in the field
- Enter data into the AIRS and other databases.

### 2.2.3 Training for Laboratory and Field Personnel

Available means for training laboratory and field personnel are listed below. Each individual should receive training appropriate to his or her duties on the PM<sub>2.5</sub> monitoring program.

- Initially, train field personnel who are already familiar with high-volume and dichotomous sampler operations and laboratory personnel who are already familiar with weighing room techniques and requirements.
- Have all personnel study the relevant sections of this QA handbook, the operating manuals for the samplers and/or the microbalance, and the CFR citations.
- Develop a training manual and operations checklist.
- Have personnel attend relevant State- or regionally sponsored training workshops.
- Have personnel attend relevant EPA-sponsored training programs.
- Have personnel watch and study instructional videos that may become available.
- Have experienced operators instruct others through on-the-job training.
- Have newly trained operators pass a hands-on test of procedures.

## 2.3 Health and Safety Warnings

To prevent personal injury, all employees must heed any warnings that are associated with operation of the microbalance, the PM<sub>2.5</sub> sampler, and any supporting equipment and supplies. Specific health and safety warnings are generally found at the point in the operating manual or troubleshooting guide where they are most applicable.

In general, health and safety warnings fall into these categories:

- Electrical
- Chemical
- Equipment placement and stability.

Electrical safety considerations that apply to the PM<sub>2.5</sub> sampler include the following:

- Make all electrical connections in accordance with national codes. Always use a third-wire grounding arrangement on samplers and any electrical appliances or test rigs. To minimize the possibility of electrical shock and injury, always use a grounded outlet and cord. This process avoids the possibility of electrocution.
- Electrical supply lines to the PM<sub>2.5</sub> sampler must be installed so that they are protected from degradation and hazards. Inspect the electrical cords and connections for signs of wear and have an electrician repair or replace them as needed.
- Always unplug the power to the sampler when servicing or replacing parts in areas requiring removal of protective panels.
- Avoid contact of jewelry with electrical circuits. Remove rings, watches, bracelets, and necklaces to prevent shorting and electrical burns.
- Use caution when working near moving parts (such as pumps) to avoid injury.
- Be aware of weather patterns; leave the area if lightning storms approach because the sampler is elevated and made of conductive metal.

Chemical safety considerations that apply to PM<sub>2.5</sub> operations include the following:

- Use care in the application of cleaning solvents, greases for O-rings, and silicone oil for the impactor well. Use of gloves is recommended. Wash hands thoroughly after working with chemicals. Provide good ventilation if organic solvents are used. Dispose of chemicals and shop towels properly.
- Mercury metal, a poisonous material, is present in some types of barometers and RH indicators. If liquid mercury is spilled, it must be cleaned up and disposed of properly. Use protective equipment to avoid inhalation of vapors and impermeable gloves to avoid skin contact. Mercury cleanup kits are available. Avoid its use, if possible, to preclude mercury exposure.
- Exercise caution when using antistatic devices containing radioactive polonium sources. Keep an inventory of the location and size of antistatic devices. Dispose of the devices in accordance with State and local regulations. Also avoid touching pump or fan motors, which may be hot. Avoid injury from moving parts, such as fans and filter-changing mechanisms.

Equipment placement and stability are important as well. Ensure that the PM<sub>2.5</sub> sampler(s) and associated equipment are stably mounted to the ground or another surface so that they do not tip over. Electrical wires should be installed so no one will trip over them and be injured. If the sampler is placed on the roof of a building or on another elevated location, ensure that railings are installed and warning signs are placed to prevent falls.

## 2.4 Cautions

Damage to the PM<sub>2.5</sub> sampler may result if caution is not taken to properly install and maintain the device. Follow the manufacturer's instructions for maintaining the pump, for cleaning the

interior and exterior surfaces, and for safe, secure installation.

The weighing room must be maintained in a state of good order so that samples are not contaminated, misplaced, or misidentified. The room air must be relatively dust-free and controlled within specified temperature and humidity ranges. The microbalance must be placed on a vibration-free mounting. Care must be taken to avoid bumping the balance and disturbing its calibration settings. The calibration weights used to check balance precision and accuracy must be stored in a secure location away from sources of corrosion and should be used only for filter weighing by a knowledgeable analyst and handled only with plastic tweezers to prevent scoring and weight changes.

Finally, exercise care in handling new and used filters. If details concerning weighing, labeling, and transporting filters are not followed precisely, errors will result. Rough handling of used filters may dislodge  $PM_{2.5}$  material. Inadequate conditioning of filters at the specified temperatures and humidities or long delays between sample retrieval and sample weighing may lead to positive or negative weight changes and, thus, inaccurate  $PM_{2.5}$  concentrations.

(continued)

## 3.0 Summary

### 3.1 Overview

The procedures in this document are designed to serve as guidelines for the development of State or local agency quality assurance (QA) programs in support of measurements of  $PM_{2.5}$  using either Federal Reference Method (FRM) samplers or Class I equivalent method samplers.

Section 3.1 gives background information on the development and intended uses of FRM and Class I equivalent method samplers; Section 3.2 illustrates the design of the  $PM_{2.5}$  sampler and describes how  $PM_{2.5}$  particles are captured. Tables in Section 3.3 summarize the required field and laboratory quality control (QC) checks and their frequencies. Section 3.4 contains a checklist of the typical steps involved in servicing a sampler in the field.

As described in 40 CFR Part 50, Appendix L (EPA 1997a), the reference method for  $PM_{2.5}$  sampling employs a sampler to draw a measured quantity of ambient air at a constant volumetric flow rate (16.67 L/min) through a specially designed particle-size discrimination inlet.  $PM_{2.5}$  particles are those particles with an aerodynamic diameter less than or equal to a nominal 2.5  $\mu\text{m}$ . Particles in the 2.5  $\mu\text{m}$  and smaller size range are collected on a 46.2-mm-diameter Teflon<sup>®</sup> filter during the specified 23- to 25-hour sampling period. Each filter is weighed before use and after sampling. From these measurements, the mass of the collected  $PM_{2.5}$  sample can be calculated.

The total volume of air sampled is determined from the measured volumetric flow rate and the sampling time. The mass concentration of  $PM_{2.5}$  in the ambient air is computed as the total mass of collected particles in the  $PM_{2.5}$  size range divided by the total volume of air sampled and measured under ambient (actual) conditions of temperature and pressure. The  $PM_{2.5}$  concentration is thus expressed as  $\mu\text{g}/\text{m}^3$  of air. The particle-size discrimination characteristics of the sampler inlet, downtube, fractionator, and filter holder are all specified by design in 40 CFR Part 50, Appendix L (EPA 1997a). In addition, specific performance characteristics of the sampler are tested in accordance with the procedures in 40 CFR Part 53, Subpart E (EPA 1997b). Sampling methods for  $PM_{2.5}$  that meet all requirements in both Parts 50 and 53 are designated as  $PM_{2.5}$  FRMs for use in State and Local Air Monitoring Stations (SLAMS) and Prevention of Significant Deterioration (PSD) monitoring networks. These designated methods are identified by a specific number and can also be identified by the manufacturer and model number of the sampler.

### 3.2 Illustrations of Sampler Inlet Components and Sample Flow Paths

Figure 3-1 illustrates the inlet of the  $PM_{2.5}$  sampler. This inlet is designed to representatively extract ambient aerosols from the surrounding airstream, remove particles with aerodynamic diameters greater than 10  $\mu\text{m}$ , and send the remaining smaller particles to the next stage. Figure 3-2 illustrates the impactor and filter holder assembly that first removes those particles less than 10  $\mu\text{m}$  but greater than 2.5  $\mu\text{m}$  in diameter but allows particles of 2.5  $\mu\text{m}$  in diameter to pass and collect on a Teflon<sup>®</sup> filter surface. Downstream of the inlet, particles less than 10  $\mu\text{m}$  but greater than 2.5  $\mu\text{m}$  are removed by a single-stage, single-flow, single-jet impactor assembly. The well of the impactor assembly contains a 37-mm-diameter glass fiber filter immersed in 1 mL of low-

volatility, low-viscosity diffusion oil. The oiled glass fiber filter removes particles between 10 and 2.5  $\mu\text{m}$  in diameter by preventing bouncing of the incoming particles.

### 3.3 Summary of Field and Laboratory QC Checks

Tables 3-1 and 3-2 summarize the field and laboratory QC checks required to collect and process  $\text{PM}_{2.5}$  according to U.S. EPA regulations. The frequencies of the checks are listed and cross-references are made to the *Code of Federal Regulations* and to this document.

### 3.4 Summary of Sampling Procedures for $\text{PM}_{2.5}$

To illustrate the steps to install a filter/cassette in the sampler to begin a run and the steps to shut down the

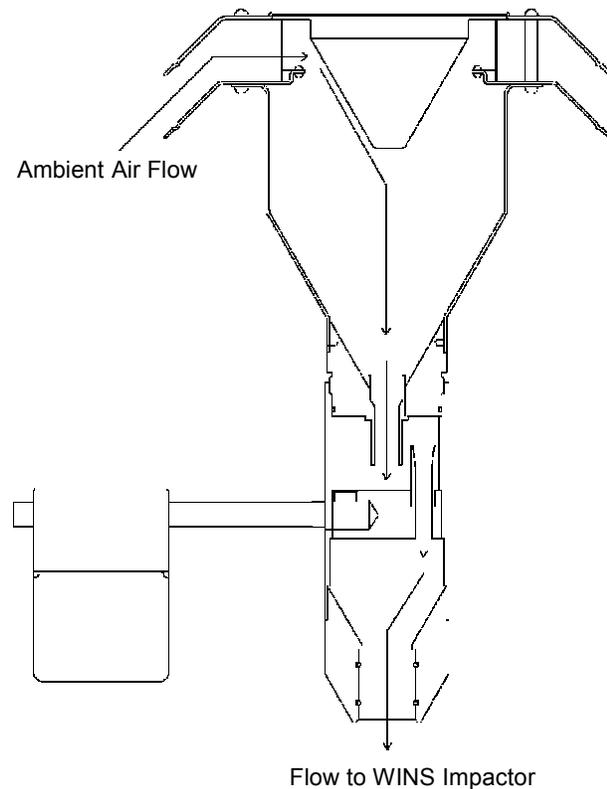


Figure 3.1. Air flow through the  $\text{PM}_{2.5}$  sampler inlet head.

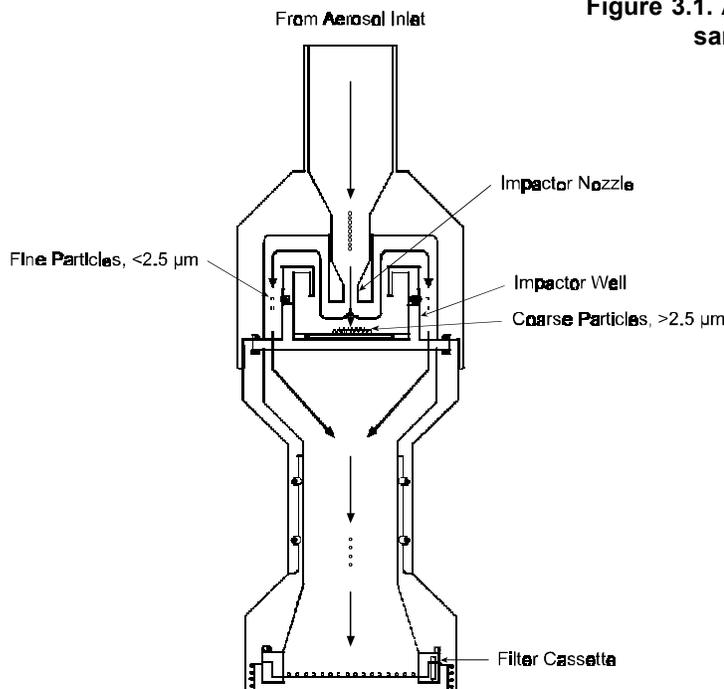


Figure 3-2. Air flow through the  $\text{PM}_{2.5}$  impactor well and filter holder.

(continued)

sampler at the end of a run, an outline or checklist to follow is given in Figure 3-3.

Table 3-1. Field QC/QA Checks

| Requirement <sup>a</sup>             | Frequency                             | Acceptance criteria                    | CFR reference                                  | 2.12 reference        | Information provided                   |
|--------------------------------------|---------------------------------------|--|--|-----------------------|--|
| <b>Calibration standards</b>         |                                       |  |  |                       |  |
| Flow rate (FR) transfer standard     | 1/yr                                  | ±2% of NIST-traceable standard         | Part 50, App. L, Sec 9.1, 9.2<br>Not described | Sec 6.3.3             | Certification of traceability          |
| Field thermometer                    | 1/yr                                  | ±0.1 EC resolution<br>±0.5 EC accuracy | Not described                                  | Sec 4.2.2 and 8.3.2.1 | Certification of traceability          |
| Field barometer                      | 1/yr                                  | ±1 mmHg resolution<br>±5 mmHg accuracy |  | "                     | Certification of traceability          |
| <b>Calibration/verification</b>      |                                       |  |  |                       |  |
| FR calibration                       | If multipoint failure                 | ±2% of transfer std                    | Part 50, App L, Sec 9.2                        | Sec 6.3               | Calibration drift and memory effects   |
| FR multipoint verification           | Per manual, NLT 1/yr                  | ±2% of transfer std                    | Part 50, App L, Sec 9.1, 2                     | Sec 6.3 and 8.3       | Calibration drift and memory effects   |
| <b>One-point FR verification</b>     | 1/4 weeks                             | ±4% of transfer std                    | Part 50, App L, Sec 9.2.5                      |                       |  |
| External leak check                  | Every 5 sampling events               | 80 mL/min                              | Part 50, App L, Sec 7.4                        | "                     | Sampler function                       |
| Internal leak check                  | Every 5 sampling events               | 80 mL/min                              | "  | "                     | Sampler function                       |
| Temp multipoint verification         | On installation, then 1/yr            | ±2 EC                                  | Part 50, App L, Sec 9.3                        | Sec 6.4               | Calibration drift and memory effects   |
| One-point temp. verification         | 1/4 weeks                             | ±4 EC                                  | Part 50, App L, Sec 9.3                        | Sec 6.4 and 8.2       | Calibration drift and memory effects   |
| Pressure calibration                 | On installation, then 1/yr            | ±10 mmHg                               | "  | Sec 6.5               | Calibration drift and memory effects   |
| Pressure verification                | 1/4 weeks                             | ±10 mmHg                               | "  | Sec 8.2               | Calibration drift and memory effects   |
| Clock/timer verification             | 1/4 weeks                             | 1 min/mo                               | Part 50, App L, Sec 7.4                        | Not described         | Verification to ensure proper function |
| <b>Blanks</b>                        |                                       |  |  |                       |  |
| Field blanks                         | approximately 10%                     | ± 30 Fg                                | Part 50, App L, Sec. 8.3.7.1                   | Sec 7.7               | Measurement system contamination       |
| <b>Precision checks</b>              |                                       |  |  |                       |  |
| Collocated samplers                  | Every 6 days                          | CV≤10%                                 | Part 58, App A, Sec 3.5, 5.5                   | Sec 10.2.2            | Measurement system precision           |
| <b>Accuracy</b>                      |                                       |  |  |                       |  |
| FR audit                             | 1/2 wk (automated)<br>1/3 mo (manual) | ±4% of audit standard                  | Part 58, App A, Sec 3.5.1                      | Sec 10.2.3            | Instrument bias/accuracy               |
| External leak check                  | 4/yr                                  | <80 mL/min                             |  | Sec 6.6               | Sampler function                       |
| Internal leak check                  | 4/yr                                  | <80 mL/min                             | Not described                                  | Sec 6.6               | Sampler function                       |
| Temperature check                    | 4/yr                                  | ±2 EC                                  | Not described                                  | Sec 8.3               | Calibration drift and memory effects   |
| Pressure check                       | 4/yr                                  | ±10 mm Hg                              | Not described                                  | Sec 8.3               | Calibration drift and memory effects   |
| <b>Audits (external assessments)</b> |                                       |  |  |                       |  |
| FRM performance eval.                | 25% of sites 4/yr                     | ±10%                                   | Part 58, App A, Sec.3.5.3                      | Sec 10.2.3            | Measurement system bias                |
| External leak check                  | 1/yr or as required                   | <80 mL/min                             | Not described                                  | Sec 6.6.1             | Sampler function                       |
| Internal leak check                  | 1/yr or as required                   | <80 mL/min                             | Not described                                  | Sec 6.6.2             | Sampler function                       |
| Temperature audit                    | 1/yr                                  | ±2 EC                                  | Not described                                  | Sec 10.2.5            | Calibration drift and memory effects   |
| Pressure audit                       | 1/yr                                  | ±10 mmHg                               | Not described                                  | Sec 10.2.5            | Calibration drift and memory effects   |

<sup>a</sup>Requirements in bold/ italics require a change in the CFR that is in process.

Table 3-2. Laboratory QC/QA Checks

| Requirement                     | Frequency                               | Acceptance criteria                    | CFR, 2.12 reference                                  | Information provided   |
|---------------------------------|---|--|--|--|
| <b>Blanks</b>                   |   |  |  |  |
| Lot blanks                      | Once (9 filters/lot;<br>3 boxes, 3/box) | Less than 15 µg/wk<br>difference       | Part 50, App. L, Sec. 8.2<br>2.12, Sec. 7.7          | Filter shipment stability; time to<br>obtain stable weight for the lot |
| Lab blanks                      | Approximately<br>10%/weighing run       | ±15 µg difference                      | Part 50, App. L, Sec. 8.2<br>2.12, Sec. 7.7          | Laboratory contamination   |
| <b>Calibration/verification</b> |   |  |  |  |
| Balance calibration             | 1/yr                                    | Manufacturer's spec.                   | 2.12, Sec. 7.2                                       | Verification of equipment operation                                    |
| Lab temp. calibration           | 3 mo                                    | ±2 EC                                  | Quality assurance project<br>plan (QAPP), Sec. 13/16 | Verification of equipment operation                                    |
| Lab humidity calibration        | 3 mo                                    | ±2%                                    | QAPP, Sec. 13/16                                     | Verification of equipment operation                                    |
| <b>Accuracy</b>                 |   |  |  |  |
| Balance audit                   | 1/yr                                    | ±15 µg for unexposed<br>filters        | 2.12, Sec. 10.2                                      | Laboratory technician operation  |
| Balance check                   | Beginning, every<br>10th sample, end    | #3 µg                                  | 2.12, Sec. 7.14                                      | Balance accuracy/stability   |
| <b>Calibration standards</b>    |   |  |  |  |
| Working mass standards          | 3-6 mo                                  | 25 µg                                  | 2.12, Sec. 4.3 and 7.3                               | Standards verification   |
| Primary mass standards          | 1/yr                                    | 25 µg                                  | "  | Primary standards verification   |
| Temperature standard            | 1/yr                                    | ±0.1EC resolution<br>±0.5EC accuracy   | 2.12, Sec.4.3.7 and 7.6                              | Standard verification<br>( check of accuracy)                          |
| Relative Humidity std           | 1/yr                                    | ±2% RH accuracy,<br>±0.5%RH resolution | 2.12, Sec. 4.3.7 and 7.6                             | Standard verification  |
| <b>Precision</b>                |   |  |  |  |
| Replicate filter weighings      | 1 per weighing<br>session               | ±15 µg difference                      | 2.12, Table 7-1<br>QAPP, Sec. 13/16                  | Weighing repeatability/filter stability                                |

**I. Prepare for Site Visit on Scheduled Date/Time**

Be sure to take the following equipment and supplies to each of the sites:

- # Prew weighed sampling filter(s) in cassette(s), packed in labeled containers. Spares.
- # Prew weighed field blank filters in cassettes, packed in labeled containers, if a field blank study is scheduled.
- # PM<sub>2.5</sub> sampler run data sheet for each sampler. Use of electronic data sheets is encouraged. Add comments to the data sheets. Site notebook and calculator.
- # Laptop computer (or alternative data storage device) and connecting cables to download sampler data.
- # Spare parts and tools including O-rings, silicone grease, laboratory wipes, voltmeter.
- # Operator's manual for the sampler(s) to be serviced.
- # If it is time for the recommended every-fifth-sample-day checks, also take the following:
  - Ready-to-use impactor well assembly or filter/lab wipes/diffusion oil to service the sampler at the site

**II. Install Filter/Cassette and Begin Sampler Operations**

**Note:** If a filter is still in the sampler, follow the instructions for removing the sample/cassette. Refer to Part III. Also, if the schedule calls for any of the following procedures, perform them before installing a new sampling filter/cassette:

- Impactor well replacement or cleaning (after every fifth sampling event)
- Sampler flow verification check (every 4 weeks)
- Sampler ambient pressure and temperature checks (every 4 weeks)
- Sampler verification/calibration (temperature, pressure, flow rate)
- Cleaning of sampler inlet head and downtube (monthly)
- Leak check (after every fifth sampling event)
- Field blanks.

Perform the following steps to install the filter/cassette and begin sampler operations:

- # Be sure the sampler is **not** operating.
- # Fill in initial information on the data sheet.
- # Remove the new filter/cassette from its protective container and visually inspect the filter/cassette for flaws. Verify that this is the correct filter for this sampler, site, and run date.
- # Remove the sampler's filter holder assembly (if required by the manufacturer's instructions). Inspect the O-rings inside the filter holder; service them if necessary.
- # Install the filter/cassette in the filter holder assembly and then install the loaded filter holder assembly in the sampler based on the manufacturer's instructions. If you touch or scratch the filter, void the filter, record this fact, and get another one from the set of extra filters brought to the site.
- # Program the sampler to turn on at the beginning of a sampling period (consult the sampler's instruction manual). The sampling day begins at midnight.
- # Make independent measurements of ambient temperature ( $T_a$ ) and ambient pressure ( $P_a$ ) using transfer standards. Record these values and the  $T_a$  and  $P_a$  values indicated by the sampler on the data sheet.

(continued)

**Figure 3-3. Summary of PM<sub>2.5</sub> sampling procedures.**

**III. Remove Filter/Cassette and End Sampler Operations**

Perform the following steps to remove the filter/cassette and end sampler operations:

- # Arrive at the site as soon as possible after the end of the sampling period. The filter/cassette must be removed within 96 hours after the collection period has ended.
- # Determine  $P_a$  and  $T_a$  using transfer standards. Enter the values on the data sheet.
- # Based on review of the data, record the stop time; total elapsed time; initial and final Q,  $Q_{avg}$ , and  $Q_{cv}$ ; total volume sampled;  $T_a$ ;  $P_a$ ; and so on on the data sheet.
- # After each completed run, download data from the sampler data port to a laptop or alternative data storage device.
- # Open the filter holder assembly (consult the sampler's instruction manual); remove the used filter/cassette; quickly visually inspect the filter for tears, oil, insects, moisture, and so on; and record your observations on the data sheet.
- # Place the filter/cassette inside a properly labeled protective container. Verify the container's label versus the site name, date, and so on.
- # Place the container inside a cooled storage chest. Do not allow the protective container to come into contact with ice or water. Sealed cooling blocks are recommended. Protect the containers from condensed water.
- # Inspect the interior of the filter housing. Note any abnormalities.
- # Inspect the interior of the impactor housing and the exterior of the impactor well. Remove any moisture or dust with a lint-free wipe and make notes on the data sheet.
- # If another sampling run is to begin, insert a new filter/cassette in the filter holder assembly and set up the sampler for the next run.
- # Review the recorded data for start and end times, sample elapsed time, flow rate, filter quality, and temperature to start the process of determining if the sample is valid, questionable, or invalid. Scan through the sampling summary on the sampler display and note flags. Record observations and reasoning for questioning or invalidating a run on the data sheet.
- # Make a final check of the site, and observe and record the presence of any activity that may have affected the particulate loading of the sample.
- # Keep the container holding the filter/cassette at a temperature below 25 EC (preferably cooled to 4 EC), and promptly deliver or ship it and the data sheet (with comments) to the sample custodian or balance operator in the weighing laboratory.

**Figure 3-3 (continued)**

## 4.0 Procurement of Equipment and Supplies

### 4.1 Overview

The establishment of an ambient PM<sub>2.5</sub> air monitoring network requires the procurement of specialized equipment and supplies for field operations and subsequent laboratory determination of particle mass. Information in this section will assist reporting organizations and States in selecting the proper equipment and in acceptance testing that equipment. Section 4.2 describes field operation equipment (excluding sampling filters) and Section 4.3 describes laboratory equipment (including sampling filters). Note that sampling filters are described in Section 4.3 because they are purchased for the laboratory, which must equilibrate and weigh them before field use. In addition to field operations and laboratory equipment, data handling system(s) (including forms, logs, files, and reporting procedures) should be developed and implemented. Information on data handling systems is provided in Section 12.0, "Data and Records Management."

Minimum monitoring equipment requirements and budgetary limits should be established before equipment is purchased. In addition, acceptance criteria for equipment and supplies should be established. Upon receipt of the sampling equipment and supplies, these acceptance checks should be conducted. The results of these checks, including whether the equipment was accepted or rejected, should be recorded in a procurement log. Figure 4-1 is an example of such a log. This log will serve as a permanent record for procurement and provide cost projections for future programs. Table 4-1 lists the major equipment needed, how it should be tested, suggested acceptance limits, and actions to be taken if acceptance limits are not met.

It is impossible to include every item that might be needed by any specific monitoring network. Therefore, each agency should determine the extent of its in-house inventory and the items that should be ordered before sampling can begin. The agency should also be prepared to order any additional equipment required in addition to that outlined in this section.

| Item           | Description                      | Qty | PO #    | Vendor        | Date   |         | Cost  | Initials | Accept/Reject | Comments            |
|----------------|----------------------------------|-----|---------|---------------|--------|---------|-------|----------|---------------|---------------------|
|                |                                  |     |         |               | Ord.   | Rec'd.  |       |          |               |                     |
| 1 case filters | 2 µm pore<br>46.2 mm<br>diameter | 60  | 971-100 | WIZ<br>Supply | 8/1/97 | 8/15/97 | \$100 | ABC      | Accept        | Examined<br>8/20/97 |
|                |                                  |     |         |               |        |         |       |          |               |                     |
|                |                                  |     |         |               |        |         |       |          |               |                     |
|                |                                  |     |         |               |        |         |       |          |               |                     |
|                |                                  |     |         |               |        |         |       |          |               |                     |

Figure 4-1. Example procurement log.

**Table 4-1. Acceptance Checks and Limits for Procurement of Equipment and Supplies**

| Equipment  | Acceptance check  | Acceptance limits   | Action if requirements are not met |
|--|---|---|------------------------------------|
| <b>Field operations</b>  |   |   |                                    |
| Sampler  | Sampler and accessories complete; no evidence of damage. Model designated as reference or equivalent method. Pump and display work. | Specifications outlined in 40 <i>Code of Federal Regulation</i> (CFR) Part 50, Appendix L | Reject sampler                     |
| Calibration quality assurance/quality control (QA/QC) equipment for flow rate, temperature, pressure | Accompanied by certificate. Check values against National Institute of Standards and Technology (NIST)-traceable standards.         | Within accuracy limits described in this document   | Adjust or reject equipment         |
| Audit equipment  | Same as for calibration equipment but must not be the same equipment.   | Within accuracy limits described in this document   | Adjust or reject equipment         |
| <b>Laboratory operations</b>   |   |   |                                    |
| Filters, Teflon®   | Of correct type and undamaged.  | Type as described in 40 CFR Part 50, Appendix L   | Reject filters                     |
| Filter cassettes   | Of correct type and undamaged.  | As specified by sampler manufacturer  | Reject filter cassettes            |
| Filter/cassette protective containers  | Of correct type and undamaged.  | As described in this document   | Reject protective containers       |
| Filter-handling containers   | Of correct type and undamaged.  | As described in this document   | Reject filter-handling containers  |
| Analytical microbalance  | Accompanied by certificate; check values against working standards.   | Readability 1 µg, repeatability 1 µg  | Adjust or reject equipment         |
| Mass reference standards   | Check working standards against NIST-traceable primary standards.   | Tolerance ±25 µg  | Reject standards                   |

## 4.2 Procurement Prerequisites—Field Operations

### 4.2.1 Reference or Equivalent Method Sampler

Each PM<sub>2.5</sub> sampler used for reporting data to determine attainment of the National Ambient Air Quality Standard (NAAQS) for fine (2.5 µm) particulate matter **must** meet U.S. EPA standards and **must** be of a model designated by EPA as a reference or equivalent method. The minimum

sampler requirements are described in the reference method (EPA 1997a) and in 40 CFR Part 53 (EPA 1997b). The PM<sub>2.5</sub> sampler cost will vary with the manufacturer and the sophistication of the sampler. Basic considerations for sampler selection include flow control and measurement systems, maintenance requirements, reliability, ease of operation, and such additional capabilities as sequential sampling and downloading data.

Although a network may decide to use any number of different reference and designated equivalent PM<sub>2.5</sub> monitor models, using a single model of sampler in a network minimizes the variety of spare parts required to keep the network in operation, simplifies training of operators, and adds consistency to the data reduction process. An in-house inventory of general maintenance supplies and replacement parts is recommended. Examples include: various hand tools, laboratory wipes, soft brushes, and cotton swabs. Spare parts for the sampler may be obtained from the manufacturer or may be purchased from other suppliers.

The impactor requires a circular, 35- to 37-mm-diameter filter made of borosilicate glass with no binder. The filter pore size must be 1 to 1.5  $\mu\text{m}$  and the thickness must be 300 to 500  $\mu\text{m}$ . The impactor oil is tetramethyltetraphenyl-trisiloxane, single-compound diffusion oil, with the following specifications at 25 EC: vapor pressure,  $2 \times 10^{-8}$  mmHg; viscosity, 36 to 40 centistokes; and density, 1.06 to 1.07 g/cm<sup>3</sup>.

#### 4.2.2 Calibration Equipment

Specialized equipment is needed to calibrate the sampler for temperature, barometric pressure, and volumetric flow measurements. At a minimum, the following equipment should be procured:

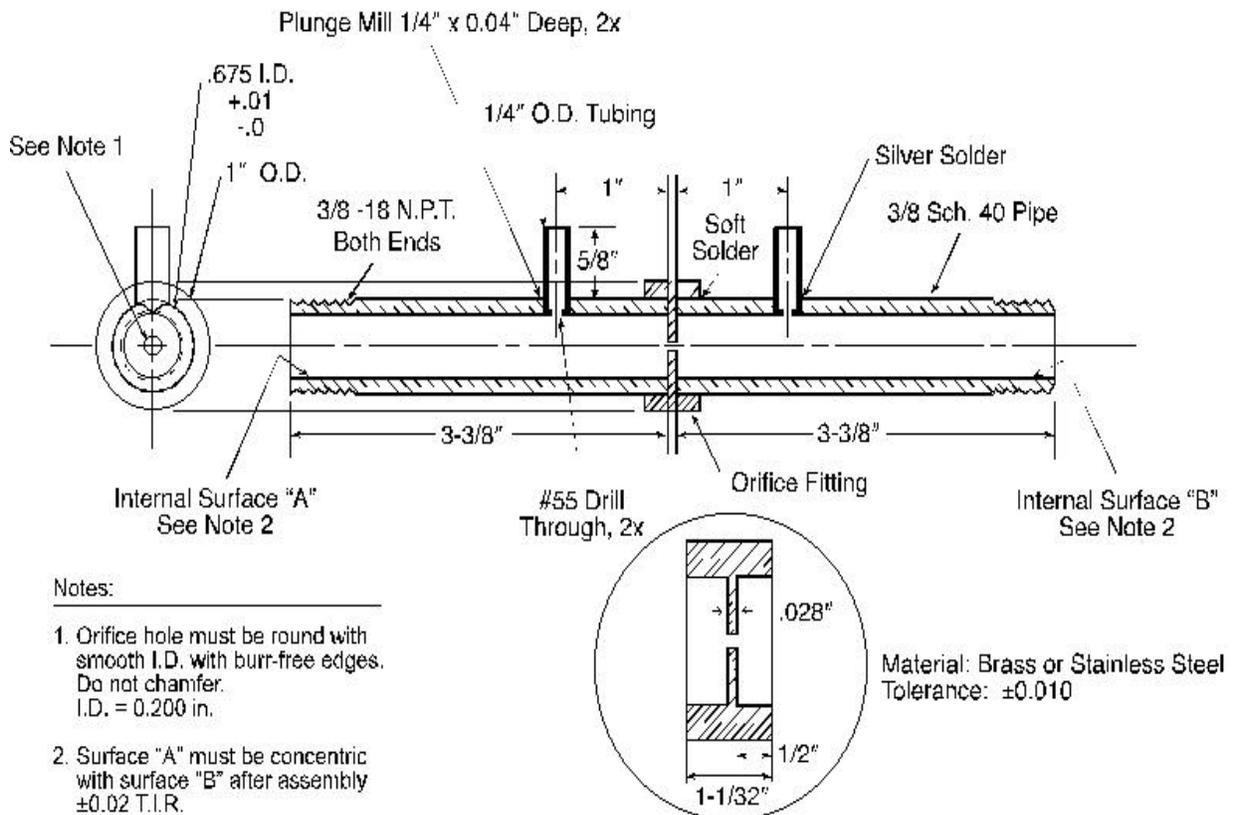
- A thermometer(s) capable of measuring ambient temperatures in a range of -30 to +45 EC, readable to the nearest 0.1 EC. This thermometer should be referenced (ASTM 1992; EPA 1995; NIST 1986, 1988, 1989) to within an accuracy of  $\pm 0.5$  EC to NIST-traceable American Society for Testing and Materials (ASTM) precision thermometers (e.g., ASTM numbers 62C, 63C, and 64C) once a year (ASTM 1995a). Multiple thermometers may be used to cover the temperature range as long as each thermometer meets the accuracy and readability specifications described above. Thermistor or thermocouple thermometers for use in the field should be calibrated against NIST-traceable thermometers. Some flexibility in these recommendations applies at sites where temperatures are extremely cold.
- A barometer capable of measuring barometric pressure (ASTM 1995b) over a range of 600 to 800 mm Hg (80 to 106 kiloPascals [kPa]) and readable to the nearest 1 mmHg. At least once a year, this barometer should be calibrated to within  $\pm 5$  mmHg of a NIST-traceable barometer of known accuracy. A portable, aneroid barometer (e.g., a climber's or engineer's altimeter) is suitable for field use. A Fortin-type, mercury-column barometer may also be useful for laboratory checks of the barometer used for field measurements.
- Flow rate measurement equipment (transfer standards) capable of calibrating or verifying the sampler's flow rate measurement device with an accuracy of  $\pm 2$  percent. This flow rate standard must be a separate, stand-alone device. It must have its own certification and be traceable to a NIST primary standard for volume or flow rate. A variety of flow rate transfer standards, with their optimum flow ranges and their support equipment, are presented in Section 6.3.2.

- A flow rate measurement adapter of the dimensions specified in drawing No. L-30 in Appendix L of 40 CFR Part 50 (EPA 1997a) and described in Section 7.3.6, Appendix L, that will connect the transfer standard outlet to the PM<sub>2.5</sub> sampler flow path entering the impactor assembly and form a leak-free seal. All interconnecting tubing should be flexible and crimp-resistant. This adapter is also used in conducting the required external leak test.

#### 4.2.3 Flow Verification Check Device

As part of the quality control (QC) procedures, a flow verification check device is required to verify that the PM<sub>2.5</sub> sampler is operating at the correct flow rate. Figure 4-2 shows the construction details for an inexpensive and rugged QC flow verification check device that is based on measurement of the pressure drop across an orifice. This device is intended for use with a water manometer or other differential pressure meter that reads in inches of water. **Note:** Proper calibration of the orifice should be conducted in order to accurately predict the device's response to variations in ambient temperature and pressure. Orifices may be calibrated using a NIST-traceable soap-bubble flowmeter of appropriate range (Nelson 1992) or other suitable NIST-traceable volumetric or flow rate standard. Other calibrated volumetric flow rate devices such as the flow rate transfer standards described in Section 6.3.2 may also be used to check flow rates during routine operation.

#### 4.2.4 Audit Equipment



**Figure 4-2. Orifice dimensions to provide approximately 1 in. of water pressure drop at 16.7 L/min. (all dimensions in inches).**

Audit equipment should be similar to the calibration equipment described above. However, all audit equipment must not be the same equipment used for calibration and flow verification checks, although both sets of equipment may be of identical makes and models (see 40 CFR Part 58, Appendix A, Section 3.5.1.2). However, the calibration equipment and the audit equipment must be referenced to the same NIST primary standard for volume or flow rate.

### 4.3 Procurement Prerequisites—Laboratory Operations

#### 4.3.1 Filter Media

In the laboratory, sampler filters are conditioned (while in glass or plastic filter-handling containers such as petri dishes or petri slides), preweighed, and placed first into sample filter cassettes and then into protective containers for transport to the field. The mean relative humidity (RH) in the microbalance environment and/or the conditioning chamber should be held between 30 and 40 percent, with a variability of not more than  $\pm 5$  percent RH over 24 hours. However, where it can be shown that the mean ambient RH during sampling is less than 30 percent, conditioning is permissible at a mean RH within  $\pm 5$  percent RH of the mean ambient RH, but in no case at an RH less than 20 percent. Mean laboratory temperature should be held between 20 and 23 EC, with a variability of not more than  $\pm 2$  EC over 24 hours. The time required for new filter weights to stabilize (see Section 7.6) may be as long as 6 weeks to eliminate initial outgassing. Adequate numbers of filters should be purchased to permit conditioning before use. In addition, it is recommended that a sufficient inventory of more than one production lot of filters be maintained (in case problems are found with a particular lot). In practice, this suggestion means that networks should plan on keeping a minimum filter inventory sufficient for 6 to 12 weeks of operational needs. These inventory levels can be adjusted based on the network's experience with filter weight stabilization times for a given vendor's filters. The EPA filter procurement vendor will address the issue of FRM users need to precondition filters before weighing. This action should should lessen the preuse conditioning time significantly, to approximately the required minimum 24 hours. EPA-purchased Teflon<sup>®</sup> filters will be acceptance-tested prior to distribution to State or local agencies. However, filters should be inspected for obvious flaws (see Section 7.5). Conditioning for outgassing will still be necessary to determine the time, in excess of the 24 hours, needed to obtain a stable weight.

Only sampling filter media that meet the following EPA specifications for use with PM<sub>2.5</sub> samplers should be purchased. The **manufacturer** must certify that these media meet the specifications given in Appendix L of 40 CFR Part 50 (EPA 1997a).

- Size—circular, 46.2-mm diameter  $\pm 0.25$  mm (with support ring)
- Medium—polytetrafluoroethylene (PTFE) Teflon<sup>®</sup> with integral support ring
- Support ring—polymethylpentene (PMP) or equivalent inert material,  $0.38 \pm 0.04$  mm thickness, outer diameter  $46.2 \pm 0.25$  mm, and width of 3.68 mm. The support ring diameter should also be checked for consistency. Variations in diameter will affect the filter exposure area, which, in turn, will affect any operations that involve use of a portion of the filter.
- Pore size— $2 \mu\text{m}$  as measured by ASTM F 316-94
- Thickness—30 to 50  $\mu\text{m}$

- Maximum pressure drop (clean filter)—30 cm H<sub>2</sub>O column at 16.67 L/min clean air flow
- Maximum moisture pickup—No more than 10 µg weight increase after a 24-hour exposure to air at 40 percent RH, relative to the weight after a 24-hour exposure to air at 35 percent RH
- Collection efficiency—greater than 99.7 percent, as measured by the dioctyl phthalate (DOP) test (ASTM 1995c) with 0.3-µm particles at the sampler's operating face velocity
- Filter weight stability (including test for loose, surface-particle contamination and test for temperature stability)—filter weight loss #20 µg in either test, measured as specified in 40 CFR Part 50, Appendix L, Section 6.9
- Alkalinity—less than 25 microequivalents/g of filter, as measured in a procedure based on the one given in Appendix A to this document.

Although not required for determination of PM<sub>2.5</sub> mass concentration under this reference method, additional specifications for the filter should be developed by users who intend to subject archived PM<sub>2.5</sub> filter samples to subsequent chemical analysis. These supplemental specifications include background chemical contamination of the filter and any other filter parameters that may be required by the chemical analysis method. All such supplemental filter specifications should be compatible with and secondary to the primary filter specifications given in Appendix L of 40 CFR Part 50 (EPA 1997a).

#### 4.3.2 Filter Support Cassettes

Most PM<sub>2.5</sub> samplers use filter support cassettes (see Figure 7-3 and 40 CFR Part 50, Appendix L) to hold the sampling filter while in use. Cassette designs may vary slightly for different model samplers and so may not be interchangeable from one sampler model to another. They should be purchased only from the sampler manufacturer or other source approved by the sampler manufacturer. The filters are placed into these cassettes in the laboratory. Filter support cassettes should be uniquely identified. The cassettes, with the filters in place, are then transported to the field in protective containers (see Section 4.3.3). A sufficient number of cassettes should be purchased to allow all placement and removal of filters in cassettes to be performed in the laboratory. Under no circumstances should filters be placed in or removed from the filter support cassettes in the field.

#### 4.3.3 Filter Cassette Protective Containers

Filter cassettes should be transported between the laboratory and the field in protective containers, as illustrated in Figure 7-3. Suitable containers should be available from the manufacturer or from a supplier specified by the manufacturer. Containers should be designed to protect the cassette and prevent it from moving within the container during transport. Containers should be fabricated from materials that have been demonstrated not to cause a static charge buildup. Use of static-proof bags should be investigated. To avoid losses of particulate matter, do not allow the surface of the filter to touch any interior surfaces of the protective container. Containers should be marked for identification purposes. Special, sampler-specific protective containers are needed for the trays or cartridges that hold multiple filters for use in sequential samplers. Consult the sampler or cassette manufacturer for details and availability. If reuse of the

containers is desired, a cleaning procedure that does not cause further contamination or degradation of the container should be developed.

#### 4.3.4 Insulated Shipping Container

An insulated shipping container, capable of maintaining a temperature below 25 EC, is needed to transport the filter cassette containers between the field and laboratory after sampling. Plastic coolers, such as those used by campers and fishers, filled with leak-proof ice substitutes have proven suitable for this use. The sample cassette temperature should be monitored from the time of collection until the sample is equilibrated (i.e., during shipping and handling). Monitoring can be done by placing an inexpensive electronic max-min thermometer in the shipping container for each leg of the trip to the laboratory.

#### 4.3.5 Filter Handling Containers

Filter media (particularly the 46.2-mm-diameter [1.85-in], 2- $\mu$ m pore-size filter specified for use with reference or Class I equivalent samplers) are especially delicate and easily damaged. Post-sampling particle loss and filter damage may occur if proper handling procedures are not followed.

To ensure the integrity of the sample, some type of protective covering is required for filter handling and conditioning within the laboratory. A glass or plastic petri dish or petri slide, designed to hold these filters, is recommended. These are available from some filter manufacturers and laboratory supply houses. The dish should be of suitable size (i.e., large enough to allow easy removal of the filter, yet small enough to prevent excessive movement within the container) and should have a tight-fitting lid to prevent intrusion of dust or loss of particles during handling at the analytical laboratory. A label should be affixed to the container to provide proper documentation. A sufficient number of containers should be available to store filters during laboratory operations.

#### 4.3.6 Analytical Microbalance

An analytical microbalance is required to weigh the sample filters. Its capacity should be adequate to weigh the sample filters (typically 100 to 200 mg). It must have sufficient room to weigh the type and size of filters used (i.e., 46.2-mm diameter). The microbalance must have a minimum readability of  $\pm 1 \mu\text{g}$  and should have a repeatability of 1  $\mu\text{g}$ . Readability is the smallest difference between two measured values that can be displayed by the microbalance. Repeatability is a measure of the ability of a microbalance to display the same result in repetitive measurements of the same weight under the same measurement conditions. **Note:** The precision of mass measurements for unexposed filters based on replicate weighings will be greater than the microbalance's repeatability. The balance must be calibrated at installation and checked immediately before each weighing session. Electrostatic charge buildup may need to be neutralized in the microbalance's weighing chamber and on individual filters. Refer to Section 7.8 and the balance manufacturer's instruction manual and technical notes. Using microbalances with optional data input and output capabilities and automatic tare capabilities is recommended to reduce potential data entry errors and the time required to weigh a given filter.

#### 4.3.7 Calibration Reference Standards

- C Mass Reference Standards - Mass reference standards (or balance check weights) should be used to verify proper microbalance operation as part of normal QC operations. These standards should be ASTM Class 1 or 1.1, traceable to NIST with an individual tolerance of no more than 0.025 mg (ASTM 1993b). The mass reference standards should be selected so as to bracket the maximum and minimum expected filter weights (e.g., 100 to 200 mg, given that the mass range of a typical 46.2-mm filter is from 110 to 160 mg). Also, some microbalance designs only require calibration at their full scale weight; therefore purchase of a mass reference standard at that weight should also be considered to verify full scale calibration. For the same reasons, consult microbalance manufacturer, and if possible, manufacturer's microbalance operating instructions. Mass reference standards should be recertified on a regular basis (e.g., yearly) at a State weight and measures laboratory or at another laboratory that is accredited by the National Voluntary Laboratory Accreditation Program (NVLAP), which is administered by NIST (Harris 1994; White 1997). See Table 4-2 for specifications for a 200-mg mass reference standard.

**Table 4-2. Specifications for 200-mg Mass Reference Standard**

| ASTM Class | Individual weight tolerance (mg) | Approximate cost (\$) <sup>a</sup> |
|------------|----------------------------------|------------------------------------|
| 1          | 0.010                            | 42                                 |
| 1.1        | 0.005                            | —                                  |
| 2          | 0.025                            | 23                                 |

<sup>a</sup> Costs may be \$30 (or more) higher for individual weights with a NVLAP traceable certificate or a NVLAP weight calibration report. Weights that include these certificates are traceable to NIST.

- C Thermometer Standard - Use standard capable of measuring in the range of at least 18 to 25EC, readable to the nearest 0.1EC, and accurate to within 2EC.
- C Relative Humidity Standard- Use standard capable of measuring in the range of 20 to 50% RH, readable to the nearest 0.5% RH, and accurate to within 2% RH.

## 5.0 Installation

### 5.1 Siting Requirements

As with any type of air monitoring study in which the sample data are used to draw conclusions about a geographic area, the validity of those conclusions depends on the representativeness of the sampling data. Therefore, an initial goal of a PM<sub>2.5</sub> monitoring project is to select a safe and secure site where the PM<sub>2.5</sub> measurements will be representative of the monitoring objectives for that site.

#### 5.1.1 Spatial and Temporal Scales of Site

Spatial and temporal scale considerations are important in PM<sub>2.5</sub> sampler siting. Spatial scales may range from a small (0.1- to 0.5-km<sup>2</sup>) area to large regional areas exceeding thousands of square km. Whether the potential impact of particulate pollution is generated by a local or general source category affects the size of the spatial monitoring scale. In addition, the siting of the samplers within a monitoring network should reflect whether the expected impact will be limited to a small area (a few city blocks) or will extend to larger areas (metropolitan or rural). For temporal scale, most interest focuses on either an annual geometric mean concentration or a 24-hour average concentration. Because siting of a PM<sub>2.5</sub> sampler requires considering the prevailing wind direction, a sampler sited for monitoring trends in air quality over a period of a year is not necessarily ideal for measuring 24-hour concentrations. Thus, the choice of temporal scale also affects the sampler location. These spatial and temporal aspects of network design and optimum site exposure are more completely explained in 40 CFR Part 58, Appendix D (EPA 1997a) and in the guidance document for network design and optimum site exposure for PM<sub>2.5</sub> and PM<sub>10</sub> published by the U.S. EPA Office of Air Quality Planning and Standards (OAQPS) (EPA 1997b).

#### 5.1.2 Sampler Location

Although spatial and temporal scales must be considered in site selection, the following sampler location guidelines should be observed regardless of the scale:

- The PM<sub>2.5</sub> sampler must have unobstructed air flow for a minimum of 2 m in all directions. Be sure to provide sufficient area for a collocated Federal Reference Method (FRM) sampler and for installation of a portable FRM performance evaluation (PE) sampler.
- The sampler inlet should be placed at a height of 2 to 15 m above ground level.
- If a PM<sub>2.5</sub> sampler is collocated with any other particulate matter sampler, the spacing between sampler inlets must be \$1 m for other PM<sub>2.5</sub> samplers (or samplers with flow rates <16.67 L/min) and \$2 m for total suspended particulate (TSP) type samplers (or samplers >16.67 L/min). In either case, the spacing between inlets must be no more than 4 m. The heights of the inlets should be within 1 m as measured in the vertical direction.

#### 5.1.3 Safety

A PM<sub>2.5</sub> sampler used for routine sampling must be situated where the operator can reach it safely regardless of weather conditions. If the sampler is located on a rooftop, care should be

taken that the operator's personal safety is not jeopardized by a slippery roof surface during inclement weather. Consideration also should be given to the fact that routine operation (i.e., calibrations, sample filter installation and recovery, flow checks, and audits) involves transporting supplies and equipment to and from the monitoring site.

#### 5.1.4 Electrical Considerations

In 40 CFR Part 50 Appendix L (EPA 1997c), a PM<sub>2.5</sub> sampler is required to operate at 105 to 125 volts alternating current (AC) and a frequency of 59-61 hertz (Hz). The sampler may draw a higher current when the pump starts, possibly necessitating a slow-blow fuse. Although PM<sub>2.5</sub> samplers are required to indicate power interruptions, every effort should be made to provide a stable power source for the monitoring site. The site should be able to provide sufficient power for the primary sampler, a collocated sampler, and an FRM PE sampler.

#### 5.1.5 Security

The security of the sampler itself depends largely on its location. Rooftop sites with locked access and ground-level sites with fences are common. Fences should be of chain-link or similar construction so as not to impede air flow across the site. The inlet of the sampler should extend above the top of the fence. In all cases, the security of the operating personnel as well as the sampler should be considered.

### 5.2 Sampler Installation Procedures

#### 5.2.1 Receipt of Sampler

- Establish an individual logbook for each sampler received.
- On receipt of a PM<sub>2.5</sub> sampler from the manufacturer, visually inspect the sampler to ensure that all components are accounted for.
- Compare the equipment delivered with the items listed on the enclosed packing slip. Notify the manufacturer immediately of any missing or damaged equipment.
- Read the manufacturer's instruction manual and become familiar with the sampler's operating and calibration procedures.
- Assemble the sampler in the laboratory according to the manufacturer's instructions, except do not put oil in the impactor well.

#### 5.2.2 Laboratory Evaluation

The following activities can most conveniently take place in an indoor, laboratory environment. Field measurements personnel, not laboratory personnel, should conduct these tests.

- Energize the sampler. Check to be sure that the pump, mechanical components, and electronic displays are working.
- Perform a leak check according to the manufacturer's instructions. Each sampler's procedure will be somewhat different from the others. The impactor should not have oil in it, and a flow check filter should be placed in the filter cassette for this test.

- Check the flow rate at 16.67 L/min and at  $\pm 10$  percent of this value according to the manufacturer's instructions and Section 6.0, "Calibration Procedures."
- Perform checks of the temperature and pressure sensors.
- Perform a flow verification check according to the manufacturer's instructions and Section 8.3.2.
- For sequential samplers, check that the timing and sequencing functions work properly.
- If this is the first sampler model of its type received, conduct data downloading exercises to become familiar with the process. If it is a sequential sampler, operate it for at least 3 consecutive days to test the system.
- If oil has been added to the impactor well during any of the laboratory tests, remove it before transit to the field site.

### 5.2.3 Setup at Sampling Site

- Carefully transport the sampler to the sampling site.
- Secure the sampler in its predetermined location. The legs of the sampler can be bolted to angle aluminum bars. The bars can be held in the soil with metal stakes or they can be nailed to wooden beams if the sampler is on the surface of a roof. Keep the sampler level. Allow space for installation of a collocated sampler and/or an FRM PE sampler.

### 5.2.4 Field Evaluation

- Check all tubing for cleanliness, crimps, cracks, or breaks.
- Plug the power cord into a line voltage outlet. The use of waterproof interlocking electrical connectors is recommended to ensure operator safety and to avoid shorts and/or power interruptions. Do not allow any electrical connections to become submerged during periods of inclement weather.
- Perform a leak test with a filter in the filter cassette. Do not use the filter for subsequent sampling.
- Allow the system to run and equilibrate to ambient conditions for approximately 15 min.
- Perform checks of the temperature and pressure sensors.
- Perform a flow rate verification check according to the manufacturer's instructions.
- Correct any problems before proceeding. The sampler is now ready for routine use.

## 6.0 Calibration Procedures

### 6.1 Overview

Before a PM<sub>2.5</sub> monitoring program is undertaken, all sampling and analysis equipment must be properly calibrated. Careful, accurate calibrations of sampling instrumentation and associated equipment are fundamental for any monitoring network. The suggestions and requirements specified in this section may serve as initial acceptance checks; further checks may be necessary if acceptance specifications are not met. All data and calculations should be recorded in a calibration logbook or on calibration data sheets. A separate logbook or a separate section of a logbook should be used for each apparatus and sampler in the program. Once the initial calibration of a PM 2.5 sampler's sensors is established, a schedule of field performance checks should be maintained to verify that the sensor calibration has not changed. Note that the calibration verification activity includes first determining if the sensor reading is still within the acceptance limits of the calibration. Only if the sampler sensor reading is not acceptable would a mathematical or instrumental adjustment be made. Whenever field performance checks of the sampler's flow rate, external or internal leakage rate, temperature, or pressure systems show out-of-tolerance conditions, the operator is cautioned to double-check the equipment, the connections, and the sampler before proceeding with recalibration. It is possible that the sampler is not out of calibration and considerable time and effort could be spent unnecessarily.

According to 40 CFR Part 50, Appendix L, Subsection 10 (EPA 1997), the PM<sub>2.5</sub> sampler flow rate measurement system must be calibrated in terms of the actual volumetric flow rate, ( $Q_a$ ), at prevailing ambient conditions of temperature and pressure, as opposed to the standard volumetric flow rate ( $Q_{std}$ ), which is measured or corrected to U.S. EPA-standard conditions of temperature and pressure. The flow rate measurement system consists of a dry gas meter or other flow sensor, a temperature sensor, a pressure sensor, and associated devices which, as needed, make control adjustments to maintain the set point of the volumetric flow rate in response to variations in temperature and pressure. Refer to the operating manual for your PM<sub>2.5</sub> sampler for specific instructions on how to enter calibration data using the keypad and how to make other adjustments. Measured PM<sub>2.5</sub> concentrations are also reported in terms of the actual volume at ambient conditions of temperature and pressure. Therefore, no conversions between  $Q_a$  and  $Q_{std}$  are generally necessary. However, if the calibration for your flow rate standard or transfer standard is expressed in terms of  $Q_{std}$ , conversion to  $Q_a$  is required. The calculations necessary to convert these flow rates are presented in Section 6.2, Equations (6-1) and (6-2).

This section presents the following aspects of calibration procedures:

- A discussion of  $Q_a$  and  $Q_{std}$  flow rate designations and their applicability in a PM<sub>2.5</sub> monitoring program (Section 6.2)
- Basic calibration procedures and associated calculations for calibration of the sampler's flow rate measurement system (Section 6.3)
- Discussion of flow rate transfer standards and calibration equipment (Sections 6.3.2 and 6.3.3)

- Basic calibration procedures for the sampler's temperature and pressure measurement systems (Sections 6.4 and 6.5)
- Procedures for conducting external and internal leak checks (Section 6.6)
- Sampler calibration frequency requirements (Section 6.7).

More detailed information on calibration and operation procedures can be found in each sampler manufacturer's operating instruction manual. These instruction manuals are a required part of the manufacturer's application for designation as an EPA Reference or Equivalent Method and as such have been reviewed and accepted for their designated sampler.

## 6.2 General Aspects of Flow Rate Measurement and PM<sub>2.5</sub> Sampler Calibration

As discussed in Section 3.0, "Summary," a reference or Class I equivalent PM<sub>2.5</sub> sampler includes a specially designed sample air inlet, a size-fractionating impactor, and a sample flow rate control system. The particle-size discrimination characteristics of both the inlet and the impactor are critically dependent on specific internal air velocities; a change in velocity will result in a change in the nominal particle size collected. These velocities are determined by the actual volumetric flow rate (not the mass flow rate) through each device. Therefore, the actual volumetric flow rate through the sampler's inlet and fractionator must be maintained at a constant value that is as close as possible to the design flow rate specified for the sampler. The design flow rate for a given sampler is specified in the sampler's operation or instruction manual. Upper and lower tolerance limits for the sampler's flow rate are specified in 40 CFR Part 50, Appendix L.

As mentioned previously, the calibration of the sampler's flow rate measurement system must be performed in terms of actual volumetric units ( $Q_a$  or  $V_a$ ). Results must be recorded in the same units. However, previous measurements for PM<sub>10</sub> were based on standard units ( $Q_{std}$  or  $V_{std}$ ); thus the certification for some calibration standards may be in terms of standard volumetric flow rate units ( $Q_{std}$ ). Therefore, it is important to understand the definition for each of these two types of volumetric units and the distinction between them. Before calibration procedures are initiated, the operating agency personnel should review the following flow rate definitions:

- $Q_a$ —Actual volumetric air flow rates that are measured and expressed at existing conditions of temperature and pressure are denoted by  $Q_a$  ( $Q_{actual}$ ). Typical units are L/min and m<sup>3</sup>/min. Inlet design flow rates for PM<sub>2.5</sub> samplers are always given in actual volumetric flow units.
- $Q_{std}$ —Air flow rates that have been adjusted to EPA-standard conditions of temperature and pressure (25 EC or 298 K and 760 mmHg or 101 kPa) are denoted by  $Q_{std}$  ( $Q_{standard}$ ). Typical units are L/min and m<sup>3</sup>/min. Standard volume flow rates are often used by engineers and scientists because they are equivalent to mass flow rate units. Prior to 1997, standard volumes (derived from standard volume flow rates and the total time of sampling) were also required in the calculation of mass concentration ( $\mu\text{g}/\text{m}^3$ ) in reporting PM<sub>10</sub> measurements.

These  $Q_a$  and  $Q_{std}$  flow rate units must not be confused or interchanged. If necessary, the flow rate units can be converted, provided the temperature and pressure are known. The following conversion formulas can also be used for average flow rates ( $Q_a$  and  $Q_{std}$ ) over a sampling period by substituting average temperature ( $T_a$ ) and pressure ( $P_a$ ) over the sampling period.

$$Q_{std} = Q_a(P_a/P_{std})(T_{std}/T_a) \quad (6-1)$$

$$Q_a = Q_{std}(P_{std}/P_a)(T_a/T_{std}) \quad (6-2)$$

where

$$\begin{aligned}
 Q_{std} &= \text{standard volume flow rate, standard m}^3/\text{min} \\
 Q_a &= \text{actual volume flow rate, actual m}^3/\text{min} \\
 P_a &= \text{ambient barometric pressure, mmHg (or kPa)} \\
 P_{std} &= \text{EPA standard barometric pressure, 760 mmHg (or 101 kPa)} \\
 T_{std} &= \text{EPA standard temperature, 298 K (25 EC + 273)} \\
 T_a &= \text{ambient temperature, K ( ambient EC + 273).}
 \end{aligned}$$

Any gas flow rate measured or expressed in actual volumetric units ( $Q_a$ ) is always associated with a particular temperature and pressure of the gas. If either the temperature or the pressure changes, the volumetric flow rate will also change, even though the mass flow rate of the gas remains constant. Therefore, when the flow rate is measured at different locations in the sampler, the volumetric flow rate observed will be different if either the temperature or the pressure is different at the various locations. For example, when a flow calibration device is connected to the sampler inlet, the pressure of the air flow measured by the flow calibration standard will be the ambient barometric pressure. However, the pressure of the flowing air measured by the sampler's flow measurement system will be somewhat lower than the ambient barometric pressure because all flow calibration devices cause some pressure drop. If this pressure drop is negligible (<1 percent or <4 in of H<sub>2</sub>O), then there is no problem; the pressure can be considered the same for both measurement systems. But if the pressure drop is significant (>1 percent or >4 in of H<sub>2</sub>O), then the volumetric flow rate measured by the two systems will be different, and this difference should be taken into account when comparing them. **Sampler flow rate measurement systems may or may not automatically correct for this pressure change.** If not, one of the flow rate measurements must be corrected to the same pressure as the other flow rate before they can be compared. This correction is accomplished using the following formula:

$$Q_1 = Q_2 (P_2/P_1)(T_1/T_2) \quad (6-3)$$

where

$$\begin{aligned}
 Q_1 &= \text{actual volume flow rate at the pressure and temperature at the first measurement point, m}^3/\text{min} \\
 Q_2 &= \text{actual volume flow rate at the second measurement point, m}^3/\text{min} \\
 P_1 &= \text{pressure at the first measurement point, mmHg (or kPa)} \\
 P_2 &= \text{pressure at the second measurement point, mmHg (or kPa)} \\
 T_1 &= \text{temperature at the first measurement point, K (EC + 273)} \\
 T_2 &= \text{temperature at the second measurement point, K (EC + 273).}
 \end{aligned}$$

**Note:** In many cases the temperatures will be identical, so that  $T_1=T_2$  and the  $(T_1/T_2)$  term will equal 1.

### 6.3 Calibration of the Sampler Flow Rate Measurement System

A full, detailed, EPA-approved calibration procedure, tailored specifically to each commercially available PM<sub>2.5</sub> sampler, is contained in the operating or instruction manual associated with each sampler designated as a reference or equivalent method under 40 CFR Part 53. Follow that specific procedure carefully and thoroughly to calibrate the sampler.

**CAUTION**

Do not calibrate the flow rate measurement system if there is any doubt that the temperature and pressure measurement systems are not also in calibration. If there is doubt, calibrate the temperature and pressure sensors (refer to Sections 6.4 and 6.5) before calibrating the flow rate measurement system. Also check for internal and external leaks before proceeding (refer to Section 6.6).

This section provides additional precautions, guidance, and information on flow rate standards and calibration that may not be contained in the specific instruction manual procedure. It also presents a generic outline of the general procedure used to calibrate the flow rate measurement systems of commercially available samplers. This information can be used to augment the sampler-specific procedures.

**6.3.1 General Requirements and Guidance**

1. Multipoint calibration and single-point verification of each sampler's flow rate must be performed periodically (see Section 6.7 for frequency) to establish traceability of subsequent flow rate measurements to an authoritative flow rate standard. Calibration with a flow rate standard (or a transfer standard) that is certified against a National Institute of Standards and Technology (NIST)-traceable standard (see Section 6.3.3 regarding NIST traceability) transfers the NIST traceability to the sampler's flow rate measurement system. This NIST traceability is required by Appendix A of 40 CFR Part 58.
2. PM<sub>2.5</sub> samplers may employ various types of flow rate measurement devices. The specific calibration standard and procedure used for calibration or verification of the sampler's flow rate measurement device will vary depending on the type of flow rate measurement system employed (40 CFR Part 50, Appendix L, Sec. 9.2.1). The sampler's operation or instruction manual identifies one or more types of flow rate or volume standards recommended for calibration and provides a detailed calibration procedure.
3. Calibration of the sampler's flow rate measurement system must be in units of the actual ambient volumetric flow rate ( $Q_a$ ) (40 CFR Part 50, Appendix L, Sec. 9.2.1).
4. The sampler flow rate measurement system must be calibrated or verified by installing an unused filter in the filter holder, removing the sampler inlet, and connecting the flow rate adapter and flow rate standard to the sampler, in accordance with the instruction manual, so that the flow rate standard accurately measures the sampler's flow rate. The operator should verify that no leaks exist between the flow rate standard and the sampler (40 CFR Part 50, Appendix L, Sec. 9.2.3).
5. The calibration relationship between the flow rate (in actual L/min) indicated by the flow rate standard and by the sampler's flow rate measurement device must be established or verified in accordance with the sampler's operation or instruction manual. Temperature and pressure corrections may be required for some types of flow rate standards (40 CFR Part 50, Appendix L, Sec. 9.2.4). Consult the operations manual for the particular flow rate

standard for guidance on the nature and calculation of any corrections that may be required for the standard.

6. Monthly verification of the sampler's flow rate shall consist of one flow rate measurement at the sampler's operational flow rate (40 CFR Part 50, Appendix L, Sec. 9.2.4). This one-point verification of the flow rate measurement system may be substituted for a three-point calibration, provided that a full three-point calibration is performed upon initial installation of the sampler and at least once per year thereafter and the flow rate measurement system has met the  $\pm 2$  percent accuracy requirement (40 CFR Part 50, Appendix L, Sec. 9.2.5) in the previous three-point calibration. A full three-point calibration must be carried out whenever a one-point verification indicates that the sampler's flow rate measurement system differs by  $\pm 4$  percent or more from the flow rate measured by the flow rate standard. The one-point verification must be repeated after the three-point calibration as a double-check to ensure the sampler operates properly at the design flow rate of 16.67 L/min following the calibration (40 CFR Part 50, Appendix L, Sec. 9.2.5).
7. Calibration of the sampler's flow rate measurement system must consist of at least three separate flow rate measurements (a multipoint calibration) approximately evenly spaced within the range of -10 to +10 percent of the sampler's operational flow rate (40 CFR Part 50, Appendix L, Sec. 9.2.4). The sampler must have the capability to adjust the flow rate over the -10 to +10 percent range (40 CFR Part 50, Appendix L, Sec. 7.4.2). Calibration procedures for the various samplers call for the results of three different flow rates to be keyed in to establish the calibration. The sampler's instruction manual will provide additional guidance on flow rate adjustment.
8. Following a calibration or verification, the flow rate adapter and flow rate standard are disconnected from the sampler, and the sampler's inlet is carefully reinstalled. The clean filter remains in place. Then the sampler's normal operating flow rate must be determined (in L/min), using the sampler's flow rate measurement system. If the sampler flow rate differs by  $\pm 2$  percent or more from the required operational value of 16.67 L/min, the sampler's flow rate must be adjusted to the specified flow rate (40 CFR Part 50, Appendix L, Sec. 9.2.6). The filter is then removed and the sampler is returned to service.

### 6.3.2 Flow Rate Calibration Standards

Flow rate standards used for calibrating or verifying the sampler's flow rate measurement device must have an accuracy of  $\pm 2$  percent. This flow rate standard must be a separate, stand-alone device designed to connect to the flow rate measurement adapter. The flow rate standard must have its own certification and must be traceable to a NIST primary standard for volume or flow rate (40 CFR Part 50, Appendix L, Sec. 9.1.2). See Section 6.3.3 for more information on NIST traceability. Consult the technical literature and the vendor's descriptions of various flow rate standards and their performance under various weather conditions before making a decision to purchase one.

All flow rate calibration standards must be fully equilibrated to the temperature of the air whose flow rate is to be measured. This temperature equilibration can take up to an hour, depending on the temperature at which the standard was stored prior to moving it to the point of use. During this thermal equilibration period, the standard must be exposed to the prevailing air temperature,

but it also must be protected from precipitation, wind, dust, solar heating, and other conditions that could affect its accuracy.

Various types of flow rate standards that might be considered for sampler calibration follow:

- **Bubble flowmeters**—Although completely manually operated and timed bubble flowmeters are available, a semiautomatic instrument such as the Gilian \* flowmeter is easier to use and is more likely to provide more uniform flow rate measurements. Because the liquid used to form the bubbles is usually water-based, a water-vapor correction may be required to compensate for a volume increase due to evaporation of the water in the instrument. Furthermore, the instrument cannot be used below 0 EC because the soap solution will freeze.
- **Piston flowmeters**—Automatic dry-piston flowmeters such as the BIOS\* flowmeter are relatively easy to use and not subject to the water-vapor correction or the liquid freezing limitation of the bubble flowmeters. However, they may still be subject to a lower operational temperature limit.
- **Mass flowmeters**—Although mass flowmeters may be considered as calibration standards, they have a number of shortcomings in this application. They have a high pressure drop, which could change the sampler flow rate or otherwise affect the operation of the sampler. Mass flowmeters also require conversion of the measured mass flow rate to the actual volumetric flow rate for comparison to the PM<sub>2.5</sub> sampler's displayed value. The flow rate measured by the sampler may also have to be corrected to account for the significant pressure drop introduced by the mass flowmeter itself. Finally, mass flowmeters are subject to zero or span drift, particularly over the wide range of temperatures encountered at field sites, so their accuracy should be reverified frequently over a wide temperature range.
- **Orifice devices**—Orifice devices are simple and reliable, but they too require corrections for temperature and pressure and may have a significant pressure drop. They also require a very accurate and sensitive differential pressure measurement device such as a manometer or an aneroid differential pressure gauge, whose accuracy must be reverified frequently. Electronic micromanometers are convenient to use with an orifice device such as the one illustrated in Figure 4-2. Readings from electronic micromanometers should be cross-checked against a water manometer prior to a round of sampler verifications or calibrations.
- **Laminar flow elements**—Laminar flow elements generally have a lower pressure drop than orifice devices but otherwise have the same requirements and disadvantages.
- **Wet test meters**—Wet test meters are generally not practical for field use but may be used for laboratory calibrations.
- **Dry gas meters**—Dry gas meters may be considered for field calibration. Some meters may have a substantial pressure drop. They should be checked for leaks and mechanical problems and be recertified at least annually.

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\*Mention of commercial products should not be interpreted as endorsement. This product is given as a typical and perhaps well-known example of the general class of instruments; other instruments in the class are available and may be fully acceptable.

### 6.3.3 NIST Traceability and Certification of Flow Rate Standards

The flow rate standard used for flow rate calibration should have its own certification and should be traceable to other standards for volume or flow rate, standards that are themselves NIST-traceable. It is recommended that the “other standards” be either those of a commercial calibration laboratory or those primary standards maintained by the local agency, State, or EPA region. The greater the number of calibration steps needed to link a measurement to a NIST standard, the greater the degradation of quality of this type of traceability. Thus the “other standard” should be at least as accurate (or even more accurate) and stable as the flow rate standards to be made traceable and should be no more than two traceability steps removed from an actual NIST standard. A calibration relationship for the flow rate standard, such as an equation, curve, or family of curves, should be established that is accurate to within 2 percent over the expected range of ambient temperatures and pressures at which the flow rate standard is expected to be used. The flow rate standard must be recalibrated or reverified and recertified at least annually. Appendix 12 of EPA’s *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Part I*, should be consulted for further information on the calibration of primary and secondary standards for flow measurements and the hierarchy of standards (EPA 1998).

The actual frequency with which this recertification process must be completed depends on the type of flow rate standard—some are likely to be much more stable than others. The best way to determine recertification requirements is to maintain a control chart (a running plot of the difference or percent difference between the flow rate standard and the NIST-traceable primary flow rate or volume standard) for all comparisons. In addition to providing excellent documentation of the certification of the standard, a control chart also gives a good indication of the stability of the standard. If the two standard deviation control limits are close together, the chart indicates that the standard is very stable and could be recertified less frequently. (The minimum recertification frequency is 1 year.) On the other hand, if the limits are wide, the chart would indicate a less stable standard that should be recertified more often. A particularly unstable standard may be unusable no matter how often it is recertified.

### 6.3.4 Outline of Generic Flow Rate Calibration Procedure

A fully detailed, EPA-approved flow rate calibration procedure, tailored specifically to each commercially available PM<sub>2.5</sub> sampler, is contained in the operations manual associated with each sampler that is designated as a reference or equivalent method under 40 CFR Part 53. Follow that sampler-specific procedure carefully and thoroughly, in conjunction with the guidance in this section, to calibrate the sampler. The following steps outline a general calibration procedure.

1. Before performing a flow rate calibration, make sure that the sampler temperature and pressure sensors are displaying reasonable readings (that is, they are calibrated).
2. Equilibrate the selected flow rate calibration device to ambient temperature conditions according to guidance provided in Section 6.3.2.
3. Install a filter cassette with an unused 46.2-mm filter in the sampler. This filter should meet all specifications for PM<sub>2.5</sub> sampling, but it does not need to be pre- or postweighed. This filter must not be used for sampling but can be used several more times for calibration

purposes, provided particulate loading on the filter is minimal.

4. Ensure that the sampler is operational and has warmed up. Depending on weather conditions, the sampler should run for a minimum of 10 to 15 min before starting the flow rate verification or calibration process. Check the flow rate display to assess stability.
5. Remove the inlet from the sampler. Place the flow calibration device on the sampler down-tube using a flow adapter device if necessary. Ensure that any valves in the flow adapter are open so that flow through the sampler is unrestricted.
6. Place the sampler in calibration mode according to the instructions in the manufacturer's operating manual.
7. Follow the instructions in the manufacturer's operating manual for performing the multipoint flow calibration.
8. Once calibration is complete, turn off the sampler pump, remove the filter and filter cassette, remove the flow calibration device (and flow adapter device, if applicable), and replace the sampler inlet.
9. The sampler flow rate is now calibrated. Refer to Section 6.3.1 for the procedure for flow rate verification, which will be used until the next flow rate calibration.

#### **6.4 Calibration of Sampler Temperature Sensors**

A fully detailed, EPA-approved calibration procedure for temperature sensors, tailored specifically to each commercially available PM<sub>2.5</sub> sampler, is contained in the operating manual associated with each sampler that is designated as a reference or Class I equivalent method under 40 CFR Part 53. Follow these specific procedure(s) carefully and thoroughly to calibrate the sampler's temperature sensors. It is recommended that a three-point temperature calibration of each temperature sensor be performed each year and that a one-point check of the ambient air temperature sensor be made monthly.

This section provides additional precautions, guidance, and information on temperature standards and calibration that may not be contained in the specific instruction manual procedure. The section also presents a generic outline of the general procedure used for calibration of the temperature measurement systems of commercially available samplers. This information should be used to augment the specific instruction manual procedure.

##### **6.4.1 General Requirements and Guidance**

1. Multipoint (at least three temperature points) calibration followed by single-point verification of each sampler's temperature sensors must be performed annually to establish traceability of subsequent temperature measurements to an authoritative temperature standard. Calibration with a temperature standard that is traceable to a NIST-traceable primary standard (see Section 6.3.3 regarding NIST traceability) transfers the traceability to the sampler's temperature sensors. Because temperature affects the flow rate, NIST traceability of the sampler's temperature measurements is also required for flow rate measurement traceability.

2. PM<sub>2.5</sub> sampler manufacturers use various types of temperature sensors. The specific calibration standard and procedure used for calibration or verification of the sampler's temperature sensor may vary depending on the type of sensors used.
3. Multipoint calibration of the sampler's temperature measurement system must consist of at least three separate comparative temperature measurements approximately evenly spaced over the sampler's expected operational ambient temperature range. Both the ambient air and filter temperatures are monitored in order to assess filter temperature deviation from ambient temperature and to set a flag should the filter temperature rise more than 5 EC above ambient temperatures and stay there for 30 consecutive minutes.
4. Section 4.3.5 of the EPA *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV: Meteorological Measurements* (EPA 1995) contains detailed guidance on calibration of temperature sensors and measurement systems (including a reference to the manufacturer's manual for adjustment of signal conditioning circuits), required calibration equipment, methods for calibrating the temperature, a discussion of  $\Delta T$  calibrations, and an example of a calibration report.
5. Ideally, temperature calibration should occur in the field to assess temperature sensor responses as they occur in normal operation. However, climate conditions may make this difficult and removal of the sampler to an indoor location may be preferable. Usually, the sensor element is removed from the sampler and its connecting wires are left attached to the sampler. The space formerly occupied by the sensor should be plugged with a fitting to prevent any contamination of the sampling system and entry of ambient air.
6. Monthly verification of a sampler's temperature sensors responses should consist of one temperature measurement made at the sampler's current temperature reading. This one-point verification may be substituted for a three-point calibration, provided that a full three-point calibration is performed upon initial installation of the sampler and at least once per year thereafter and the temperature measurement system has met the  $\pm 2$  EC accuracy requirement in the previous three-point calibration. A full three-point calibration must be performed whenever a one-point verification indicates that the sampler's temperature measurement system differs by  $\pm 4$  EC or more from the temperature measured by the temperature standard. A one-point verification of the ambient temperature sensor should be done following the three-point calibration to ensure the sampler has returned to its normal operating conditions.

#### 6.4.2 Temperature Calibration Standards

The operations manual associated with the sampler should identify one or more types of temperature standards recommended for calibration and provide a detailed calibration procedure for each type that is specifically designed for the particular sampler.

The EPA *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV: Meteorological Measurements* (EPA 1995), Section 4.3.5.1, gives information on calibration equipment and methods for assessing response characteristics of temperature sensors. The ambient air and filter temperature sensors of a reference or Class I equivalent PM<sub>2.5</sub> sampler must have a resolution of 0.1 EC and an accuracy of  $\pm 2$  EC over the range of -30 to 45 EC. The

handbook describes how to prepare three stable thermal mass assemblies whose temperatures can be determined to about 0.1 EC. The thermal mass assemblies can be as simple as insulated vacuum bottles (i.e., thermos bottles) containing pure water or ice, or they may be solid cylinders of aluminum metal. A good American Society for Testing and Materials (ASTM)- or NIST-traceable mercury-in-glass thermometer is also needed. The thermometer and the PM<sub>2.5</sub> sampler sensor are both immersed in the thermal mass and allowed to equilibrate; the temperature readings are then compared.

#### 6.4.3 NIST Traceability and Certification of Temperature Standards

The temperature standard used for temperature calibration must have its own certification showing traceability to a NIST primary standard. A calibration relationship to the temperature standard (an equation or a curve) is established that is accurate to within 0.5 EC over the expected range of ambient temperatures at which the temperature standard will be used. The temperature standard must be reverified and recertified at least annually.

The actual frequency of recertification depends on the type of temperature standard; some are much more stable than others. The best way to determine recertification requirements is to maintain a control chart.

Additional reference sources concerning temperature measurements and calibration are: *Liquid-in-Glass Thermometry* (NIST 1976); *Thermometer Calibration: A Model for State Calibration Laboratories* (NIST 1986); *NIST Measurement Services: Liquid-in-Glass Thermometer Calibration Service* (NIST 1988); and *The Calibration of Thermocouples and Thermocouple Materials* (NIST 1989).

#### 6.4.4 Outline of Generic Temperature Calibration Procedure

Both the ambient air and filter temperature sensors should be calibrated once per year. The ambient air sensor is located inside the shielded fixture on the outside of the PM<sub>2.5</sub> sampler and is easy to unfasten and remove for comparison to a transfer standard for temperature. It is possible to conduct the three-point calibration of the ambient sensor at the field site, although it may prove easier to remove the sampler to the laboratory to avoid weather problems and for convenience in preparing the temperature standards. On the other hand, the filter temperature sensor of reference or Class I equivalent PM<sub>2.5</sub> samplers is located in the open space just below the filter cassette. It is threaded through the wall of the filter assembly section of the sampler, and removal of plastic or metal fittings is required to remove the sensor and its associated wiring. It is recommended that this sensor be calibrated in the laboratory. (The temperature sensor housing, the sampler inlet, and the interior of the downtube can also be cleaned in the laboratory.) Be careful when removing the filter temperature sensor—do not gall the fittings because this could start an internal leak after installation. It is suggested that a sampler leak check be performed after reinstallation of the filter temperature sensor.

Several steps to follow in calibrating ambient air temperature sensors are given below. Make frequent reference to the operator's instruction manual for sampler-specific procedures and instructions.

1. Remove the ambient temperature sensor from the aspirated radiation shield so that it can be placed in a constant temperature bath while it is still connected to the sampler's signal

conditioner.

2. Prepare a convenient container (such as an insulated vacuum bottle) for the ambient temperature water bath and the ice slurry bath. See Step 3 below. If complete immersion of the sensor is necessary, wrap it in plastic film so liquid can reach the point where the connecting wire(s) and the sensor interface without wetting them. Use partial immersion when possible, thus keeping the interface dry. If immersion should be avoided altogether, it will be necessary to use thermal masses of metal rather than those based on water. Refer to Section 4.3.5 of Volume IV of the EPA QA handbook (EPA 1995). To further insulate the vacuum bottle, it can be positioned inside a larger 2-gal insulated container that has been modified to allow wires or cables to enter the top. Refer to Figure 4.3.5.3 of Volume IV of the EPA QA handbook.

Keep the temperature changes relatively small and make comparative measurements in this order: AMBIENT6COLD6AMBIENT6HOT6AMBIENT. The range of temperatures need only be as broad as that expected to contain all the ambient temperatures that will be experienced during the upcoming time period, generally a year. The range to be expected is site-specific. HOT may have to be 120 EF for Phoenix, AZ, but around 80 EF for a location such as Barrow, AK.

3. For the ambient bath, use an insulated bottle that was filled with tap or deionized water several hours earlier and allowed to equilibrate to ambient temperature. For the ice slurry, the ice should be made with distilled water and then crushed into pea-sized pieces and mixed with distilled water until an easily penetrable slurry state is reached. As long as ice is present in the slurry and the open end of the bottle is guarded from ambient air temperature fluctuations, the ice slurry temperature will be  $0.0 \pm 0.1$  EC.
4. Wrap the sensor(s) and a thermometer together with a rubber band. The thermometer bulb and the temperature sensor active site should be close together. Immerse the sensor and the attached thermometer in the ambient temperature bath. Use a cork or some other device to cover the open end of the insulated bottle and thus keep ambient air from circulating over the top surface of the water (or ice slurry mass). The bath liquid should be stirred to ensure the temperature is uniform; never use the thermometer or temperature sensor to stir the bath. Gentle stirring should continue, if possible, during the measurement process; however, do not stir while readings are being taken because this may introduce noise into the readings. Wait for the ambient thermal mass and the sensor/thermometer temperatures to equilibrate. Be sure successive temperature readings are stable (indicating equilibration with the ice slurry) before taking comparative readings.
5. For each thermal mass, in the order indicated in Step 2 above, make a series of five measurements, taken about a minute apart. Accurately read the meniscus of the thermometer. Use magnification if necessary to see the meniscus; avoid parallax errors. If the measurements made support the assumption of equilibrium, then average the five readings and record the result as the sensor temperature relative to the thermometer for hot, ambient, and cold, 0.0 EC relative to the ice slurry. Record all readings in the sampler notebook.

## 6.5 Calibration of Sampler Pressure Sensor

Each reference or Class I equivalent PM<sub>2.5</sub> sampler has a built-in atmospheric pressure sensor whose output is processed to allow control of the actual sampling flow rate to the design value of 16.67 L/min. This section gives information concerning the care of barometers, their principles of operation, and how an aneroid barometer can be made traceable to a mercury-column Fortin barometer and then used to field-check the readings provided by the sampler's pressure sensor. The manufacturer's operating or instruction manual must be consulted for sampler-specific information on how to make adjustments to calibrate the pressure sensor.

### 6.5.1 General Requirements

1. As required in 40 CFR Part 50, Appendix L, the sampler must have the capability to measure the barometric pressure of the air surrounding the sampler over a range of 600 to 800 mmHg. This measurement shall have a resolution of 5 mmHg and a NIST-traceable accuracy of  $\pm 10$  mmHg.
2. According to ASTM standard D 3631 (ASTM 1977), a barometer can be calibrated by comparing it with a secondary standard traceable to a NIST primary standard.
3. The Fortin mercurial type of barometer works on fundamental principles of length and mass and is therefore more accurate but also more difficult to read and correct than other types. By comparison, the precision aneroid barometer is an evacuated capsule with a flexible bellows coupled through mechanical, electrical, or optical linkage to an indicator. It is potentially less accurate than the Fortin type but can be transported with less risk to the reliability of its measurements and it presents no hazard to personnel from mercury spills. The Fortin type of barometer is best employed as a higher quality laboratory standard that is used to adjust and certify an aneroid barometer in the laboratory. The certified aneroid barometer can then be taken to the field and used to verify readouts from the transducer-type pressure sensor in the sampler. The sampler sensor can be left in the sampler during the comparison because atmospheric pressures are equivalent within a 100-m horizontal distance and a 0.5-m vertical distance. If there is a discrepancy, the aneroid barometer should be recompared to the Fortin-type barometer or other standard upon return to the laboratory. A discrepancy with the sampler sensor could indicate an offset in the sampler's pressure sensor adjustment or the need for replacement of the unit.
4. Protect all barometers from violent mechanical shock and explosively sudden changes in pressure. A barometer subjected to either of these events must be recalibrated. Minimize the vertical and horizontal temperature gradients across the instruments. Locate the instrument so as to avoid direct sunlight, drafts, and vibrations.

### 6.5.2 Calibration Procedures

#### 6.5.2.1 Fortin -Type Barometer Readings

1. Read the temperature from the thermometer attached to the barrel to the nearest 0.1 EC.
2. Lower the mercury level in the cistern until it clears the index pointer. Raise the level slowly until a barely discernible dimple appears on the surface of the mercury.

3. Tap the barrel near the top of the mercury column.
4. Set the vernier so that the base just cuts off light at the highest point of the meniscus; carefully avoid parallax error.
5. Read the height of the mercury column from the barometer in the manner appropriate to the vernier scale used to the equivalent of the nearest 0.1 mmHg. Apply appropriate corrections for temperature and gravity as described in the barometer's instruction booklet.

#### **6.5.2.2 Aneroid Type Barometer**

1. Always use and read an aneroid barometer when it is in the same position (vertical or horizontal) as it was when calibrated. Locate the portable aneroid barometer next to the laboratory's primary standard to begin the calibration process.
2. Immediately before reading the scale of an aneroid barometer with mechanical linkages, tap its case lightly to overcome bearing drag.
3. Read the aneroid barometer to the nearest 1 mmHg. If the aneroid barometer is to be taken to the field, compare its readings to the laboratory's primary pressure standard. If possible, adjust the portable barometer to match the reading of the primary standard. If the portable barometer reading cannot be adjusted, note the offset and correct field readings accordingly.

### **6.6 Leak Checks**

Two types of leak checks are specified for use with  $PM_{2.5}$  samplers—an external leak check and an internal filter bypass leak check. The results for both of these procedures should be recorded. Figure 6-1 is an example of such a form. Each leak check procedure is described below.

#### **6.6.1 External Leak Check**

The sampler components to be subjected to this leak test include all components and their interconnections in which external air leakage would or could cause an error in the sampler's measurement of the total volume of sample air that passes through the sample filter.

All  $PM_{2.5}$  samplers must include external air leak test components, accessory hardware, operator interface controls, a written procedure, and all other items necessary to carry out a leak test of the sampler at a field monitoring site without additional equipment.

**PM<sub>2.5</sub> Sampler Sheet**  
**General Information, and Leak Check, and Time Check**

Site Location: \_\_\_\_\_ Site Designation: \_\_\_\_\_

Name and Affiliation: \_\_\_\_\_

Observer Name(s) and Affiliation(s): \_\_\_\_\_

---

Sampler Model: \_\_\_\_\_ Sampler ID: \_\_\_\_\_

**Leak Checks:** \_\_\_\_\_ Date of Test: \_\_\_\_\_

| External |                          |  | Internal |                          |
|----------|--------------------------|--|----------|--------------------------|
| Time     | Indicated Pressure, mmHg |  | Time     | Indicated Pressure, mmHg |
|          |                          |  |          |                          |
|          |                          |  |          |                          |
|          |                          |  |          |                          |
|          |                          |  |          |                          |
|          |                          |  |          |                          |
|          |                          |  |          |                          |
|          |                          |  |          |                          |
|          |                          |  |          |                          |

Note: Repeat test or make repairs if necessary to achieve minimal leakage before proceeding with flow rate calibration.

Time Check:      Sampler Time \_\_\_\_\_ Reference Time \_\_\_\_\_

Comments:

**Figure 6-1. Example leak-check information sheet.**

Follow these general steps to perform the external leak check procedure:

1. Remove the sampler inlet and install the flow rate measurement adapter supplied with the sampler (Figure 6-2). Install a leak check filter in the sampler filter holder.
2. Close the valve on the flow rate measurement adapter and use the sampler air pump to draw a partial vacuum into the sampler, including (at least) the impactor, the filter holder assembly (filter in place), the flow measurement device, and the interconnections between these devices, of at least 55 mmHg (75-cm water column), measured at a location downstream of the filter holder assembly.
3. Plug the flow system downstream of these components to isolate the components under vacuum from the pump, such as with a built-in valve.
4. Stop the pump.
5. Measure the trapped vacuum in the sampler with its built-in pressure-measuring device.
6. Measure the vacuum in the sampler with the built-in pressure-measuring device again at least 10 min after the first measurement or at the elapsed time specified in the sampler's operations manual.

### CAUTION

Following completion of the leak test, the adapter valve should be opened slowly to limit the flow rate of air into the sampler. An excessive air flow rate may blow oil out of the impactor well and contaminate surfaces.

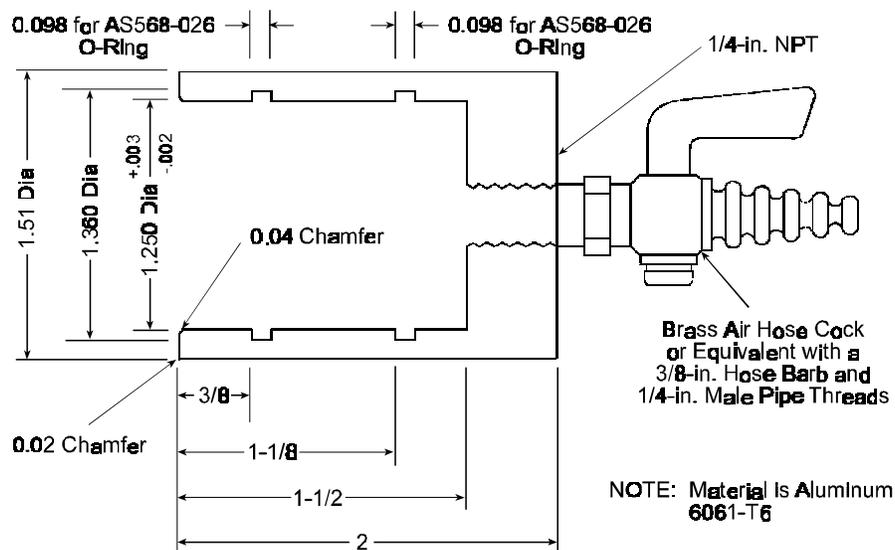


Figure 6-2. Flow adapter (all dimensions are in inches).

7. Upon completion of the leak test, slowly open the adapter valve, remove the adapter and plugs, and restore the sampler to the normal operating configuration.

To pass the external leakage test, the difference between the two pressure measurements should not be greater than the number of mmHg specified for the sampler by the manufacturer, based on the actual interior volume of the sampler, that indicates a leak of less than 80 mL/min.

Variations of the suggested technique or an alternative external leak test technique may be required for samplers whose design or configuration would make the suggested technique impossible or impractical. Therefore, a manufacturer may propose an alternative procedure to EPA under the reference or equivalency application under 40 CFR Part 53. In such cases, the manufacturer's EPA-approved leak test procedure should be followed.

### 6.6.2 Internal Filter Bypass Leak Check

This test determines if any portion of the sample flow rate that leaks past the sample filter without passing through the filter is significant relative to the design flow rate for the sampler. The suggested technique for implementing this leak test follows:

1. Perform an external leak test as described above.
2. Install a **flow-impervious** membrane material in the filter cassette, either with or without a filter, as appropriate, which effectively prevents air flow through the filter.
3. Use the sampler air pump to draw a partial vacuum within the sampler, downstream of the filter holder assembly, of at least 55 mmHg (75-cm water column).
4. Plug the flow system downstream of the filter holder to isolate the components under vacuum from the pump, such as with a built-in valve or an automatically activated solenoid valve.
5. Stop the pump.
6. Measure the trapped vacuum in the sampler with its built-in pressure-measuring device.
7. Measure the vacuum in the sampler with the built-in pressure-measuring device again several minutes after the first pressure measurement.
8. Remove the flow plug and membrane and restore the sampler to the normal operating configuration.

For successful passage of this test, the difference between the two pressure measurements should not be greater than the number of mmHg specified for the sampler by the manufacturer, based on the actual internal volume of the portion of the sampler under vacuum, that indicates a leak of less than 80 mL/min. As with the external leak check procedure described above, variations of the suggested technique may be used, provided that they were approved as part of the manufacturer's reference or equivalency application for that sampler.

If the leak rate for either of the above procedures is excessive, note the problem on the leak-check information sheet (Figure 6-1). Check the O-rings on the flow rate adapter for nicks, wear, and excessive flattening. Also check the outside of the intake tube (the downtube) where the device seats for deep scratches or dirt that could allow leakage. Check that the filter holder is

well-seated. Rectify any problems and repeat the leak check(s).

### 6.7 Verification/Calibration Frequency

Checking to verify that a sampler's flow rate measurement system still reads within the acceptance limit of the previous calibration should be done according to the sampler manufacturer's operating instructions, should take place at the field site, and should be done at least annually or

- Following major electrical or mechanical maintenance, such as replacement of a circuit board or rebuilding of the sampling pump assembly
- When a routine flow rate verification indicates a variation from the set point (16.67 L/min) of more than 4 percent.

If the annual verification fails, then recalibration and/or maintenance is required. Before beginning a flow rate calibration, the operator should repeat the flow rate verification to be certain there were no leaks in the connections between the flow rate verification device and the sampler inlet and that the flow rate data were reduced and interpreted correctly. In addition, the operator should ensure that the collector's ambient temperature and pressure measurement systems are responding accurately; if not, these systems must be recalibrated (or replaced and calibrated) before performing a flow rate calibration.

The recommended intervals for calibration and verification of flow rate, temperature, and barometric pressure are summarized in Table 6-1.

**Table 6-1. Calibration and Verification Check Intervals**

| Parameter   | Recommended minimum interval                                 |
|---|--|
| Flow rate calibration (multipoint)  | Upon failure of flow rate multipoint verification            |
| Flow rate multipoint verification   | On installation, then annually or when out of specifications |
| Flow rate verification (single-point)   | Every 4 weeks  |
| Temperature calibration (Multi point; both ambient air inlet and filter temperature sensors)                        | Upon failure of multipoint verification                      |
| Temperature multipoint verification   | On installation, then annually or when out of specifications |
| Temperature verification (single-point; ambient air inlet sensor, filter temperature sensor if convenient to do so) | Every 4 weeks  |
| Pressure calibration (multipoint)   | On installation, then annually or when out of specifications |
| Pressure verification (single-point)  | Every 4 weeks  |

## 7.0 Filter Preparation and Analysis

### 7.1 Overview

The quality of data from the PM<sub>2.5</sub> sampling program depends on several factors. A primary consideration is the analytical laboratory staff's attention to detail and their microbalance operation technique. This section offers guidelines to enhance the data quality of the laboratory operation and, hence, the PM<sub>2.5</sub> mass concentration and any additional chemical qualitative and quantitative determinations.

Microbalance resolution and repeatability requirements and filter media requirements are given in Section 4.3. Laboratory activities are summarized in Table 7-1.

### 7.2 Microbalance Environment

Gravimetric analysis of the filters is performed with a microbalance with a readability of 0.001 mg (1 µg) and a repeatability of 1 µg as listed in the microbalance's performance specifications. **Note:** The precision of mass measurements for unexposed filters based on replicate weighings will be greater than the microbalance's repeatability. Because of the greater sensitivity needed for measuring microgram-range weights or weight differences, microbalances are vulnerable to relatively small changes in physical environmental conditions, such as vibration, electrostatic charge buildup, temperature, or relative humidity (RH). Features to offset the effect of these variables on the measurements may be integrated into the design of the microbalances, or they may be offered as options.

Each microbalance used in the weighing procedures should be identified by a balance number. Make sure that the microbalance has been calibrated (at least annually) and maintained according to the manufacturer's recommendations. If it is out of calibration, the microbalance should be calibrated by a microbalance service technician according to the manufacturer's directions. The analyst should not attempt to repair the microbalance.

The microbalance must be located in the same controlled environment in which the filters are conditioned. Locate the microbalance in a climate-controlled, draft-free room or chamber (preferably dedicated to the use of balances and employing, if necessary, a high-efficiency particulate air [HEPA]-filtered air supply system on its inlet air system) to minimize human traffic effects and to stabilize the conditions of the weighing environment. Interior rooms without windows are preferred. The microbalance environment should maintain a slightly positive pressure. Ingress to and egress from the environment should be minimized. Dust contamination can be minimized by taking clean room measures such as cleaning the weighing area daily, installing sticky floor covering on the entrance(s) to the weighing area, and wearing clean laboratory clothing over anything exposed to uncontrolled environments. Ideally, a small chamber should be used because it is easier and cheaper to control the environment in a smaller space. Ideally, the microbalance should be situated within a laminar flow fume hood to minimize contamination by the analyst.

The following general guidelines should be followed to control environmental factors that may affect microbalance performance:

- Select a room that is not subject to large temperature or humidity variations and that has its

heating and air conditioning maintained 24 hours a day, including weekends.

- C Locate the microbalance away from potential sources of drafts such as doors, windows, aisles with frequent traffic, ventilation ducts, and equipment with fans or moving parts.
- Locate the microbalance out of direct sunlight and away from local heating or cooling sources such as open flames, hot plates, water baths, ventilation ducts, windows, and heat-producing lamps.
  - Locate the microbalance on a sturdy, vibration-free base (ideally, a stone weighing table) and away from any equipment that produces vibrations. If these arrangements are not possible, isolate the microbalance from such equipment by placing a stabilizing slab under the microbalance and/or by placing composite vibration damping pads at three points under the microbalance's legs or under the stabilizing slab. Placing the pads at only three points eliminates any possible rocking associated with pads placed at four points.
  - Ensure that the microbalance's base is sufficiently level to permit leveling of the microbalance according to the manufacturer's instructions.

### 7.3 Mass Reference Standards

Mass reference standards should be in the range of 100 to 200 mg, given that the mass range of a typical 46.2-mm diameter filter is from 110 to 160 mg. They should be certified as being traceable to NIST mass standards (see ASTM 1993b; Harris 1993; Kupper 1990). Additionally, they should have an individual tolerance of no more than 0.025 mg. Examples of mass reference standards that meet these specifications are American National Standards Institute/American Society for Testing and Materials (ANSI/ASTM) Classes 1 and 1.1. The mass reference standards should be recalibrated on a regular basis (e.g., yearly) at a State weights and measures laboratory holding a NIST certificate of traceability or at a calibration laboratory accredited by the National Voluntary Laboratory Accreditation Program (NVLAP), which is administered by NIST (Harris 1994; White 1997). The recalibration frequency should be determined from records of previous recalibrations of these standards.

**Note:** The microbalance's resolution and repeatability are smaller than the tolerance of the most accurate classes of mass reference standards. However, the reported uncertainty of a mass reference standard recalibrated at NIST-traceable or NVLAP-accredited laboratories will be smaller than the individual tolerance of the standard. The accuracy of the gravimetric analysis may be limited by the uncertainty of the standards rather than by the microbalance's characteristics. Because the loading mass is determined by difference, the loading error associated with the calibration standard's tolerance can be estimated as

$$\text{loading error in } \mu\text{g} = (\text{standard uncertainty in mg/standard mass in mg}) \times (\text{loading mass in } \mu\text{g}).$$

Two separate sets of mass reference standards are recommended. Working calibration standards should be used for routine filter weighing and kept next to the microbalance in a protective container. Laboratory primary standards should be handled very carefully and should be kept in a locked compartment. The working standards' masses should be verified against the laboratory primary standards every 3 to 6 months to check for mass shifts associated with handling or contamination. The verified values of the working standards as measured relative to the laboratory primary standards should be recorded in a laboratory QC notebook and used to check

**Table 7-1. Filter Preparation and Analysis Checks**

| <b>Activity</b>                 | <b>Method and frequency</b>  | <b>Requirements</b>   | <b>Action if the requirements are not met</b>      |
|---------------------------------|--|---|--|
| Microbalance                    |  | Resolution of 1 µg, repeatability of 1 µg.  | Obtain proper microbalance.                        |
| Microbalance environment        |  | Climate-controlled, draft-free room or chamber or equivalent; clean area around microbalance.   | Modify the environment.                            |
| Mass reference standards        | Working standards verified every 3 to 6 months against National Institute of Standards and Technology (NIST)-traceable laboratory primary standards.                                     | Standards bracket weight of filter; individual standard's tolerance less than 25 µg; handle with smooth, nonmetallic, clean forceps.  | Obtain proper standards or forceps.                |
| Filter handling                 | Observe handling procedure.  | Use powder-free gloves and smooth, clean forceps. Replace polonium-210( <sup>210</sup> Po) antistatic strips every 6 months.  | Discard mishandled filter or old antistatic strip. |
| Filter integrity check          | Visually inspect each filter.  | No pinholes, separation, chaff, loose material, discoloration, or filter non-uniformity.  | Discard defective filter.                          |
| Filter identification           | Write filter number on filter handling container and on laboratory data form in permanent ink.   | Make sure the numbers are written legibly.  | Replace label or correct form.                     |
| Presampling filter conditioning | Determine the correct conditioning period (at least 24 hours) for each new lot of filters. Observe and record the conditioning chamber's RH and temperature; enter laboratory data form. | Check for stability of lot blank filter weights. Weight changes should be <15 µg per week before and after equilibration. Mean RH between 30 and 40 percent, with a variability of not more than ±5 percent over 24 hours. Mean temperature should be held between 20 and 23 EC, with a variability of not more than ±2 EC over 24 hours. | Revise conditioning period; repeat conditioning.   |

(continued)

Table 7-1. (continued)

| Activity   | Method and frequency  | Requirements  | Action if the requirements are not met   |
|--|---|---|--|
| Pre- and post-sampling filter weighing                   | Observe all weighing procedures. Perform all quality control (QC) checks.   | Neutralize the electrostatic charge on filters. Wait until balance indicates a stable reading to record value.  | Repeat weighing. Many laboratories routinely repeat the weighing.  |
| Internal QC  | After approximately every 10th filter, rezero the microbalance and reweigh at least one working standard. Weigh approx. 10 percent laboratory blanks/weighing session. Reweigh one replicate filter at the end of the weighing session. Weigh approx. 10% field blanks. | The working standard measurements should agree within 3 $\mu\text{g}$ of the verified values. Laboratory blank and replicate measurements should agree within 15 $\mu\text{g}$ . Field blank measurements should agree within 30 $\mu\text{g}$ .                                | Troubleshoot and take appropriate corrective action as specified in the quality assurance project plan (QAPP) to attain acceptable levels. Do not correct or invalidate $\text{PM}_{2.5}$ measurements based on high blank levels. |
| Postsampling filter storage                              | Monitor the time between sampling and weighing.   | Weighing should be completed within 240 hours (10 days) after the end of sampling, unless the filter is maintained at 4 EC or below during the entire time between retrieval from the sampler and start of the conditioning, in which case the period shall not exceed 30 days. | Troubleshoot and take appropriate corrective action.   |
| Postsampling inspection, documentation, and verification | Examine the filter and field data sheet for correct and complete entries. If sample was shipped in a cooled container, verify that low temperature was maintained.  | No damage to filter. Field data sheet complete. Sampler worked OK.  | Save filter for inspection. Flag sample. Troubleshoot and take appropriate corrective action.  |
| Postsampling filter equilibration                        | Equilibrate filters for at least 24 hours. Observe and record the equilibration chamber's RH and temperature; enter on laboratory data sheet.   | Mean RH between 30 and 40 percent, with a variability of not more than $\pm 5$ percent over 24 hours. Mean temperature should be held between 20 and 23 EC, with a variability of not more than $\pm 2$ EC over 24 hours.   | Repeat equilibration.  |

the calibration of the microbalance. If multiple microbalances are being used, all working standards should be verified at the same time to ensure that all gravimetric measurements are intercomparable.

Always use smooth, nonmetallic forceps for handling mass reference standards. The standards are handled only with these forceps, which are not used for any other purpose. Mark these forceps to distinguish them from the forceps used to handle filters. Forceps should be cleaned with alcohol and lint-free wipes before handling standards and then should be allowed to air-dry. Handle the standards carefully to avoid damage that may alter their masses.

#### **7.4 Filter Handling**

Careful handling of the filter during sampling, conditioning, and weighing is necessary to avoid measurement errors due to damaged filters or a gain or loss of collected particles on the filters. Whenever filters are handled, the analyst should wear antistatic, powder-free gloves; these gloves act as an effective contamination barrier. Gloves that are packed in a box can carry an electrostatic charge. It is a good practice to discharge them by touching a good electrical ground after putting them on. The filters should be handled carefully by the support ring, rather than by the filter material, with smooth, nonserrated forceps that are used only for that purpose. Mark these forceps to distinguish them from the forceps used to handle mass reference standards. Forceps should be cleaned with alcohol and lint-free wipes before handling filters and then allowed to air-dry. These precautions reduce the potential effect from body moisture or oils contacting the filters and subsequently affecting the measured weights.

If filter loadings will be speciated chemically, the possibility of contamination from gloves should be considered. Some gloves have been found to be contaminated with sulfate. If antistatic gloves are used, ensure that ammonium chloride is not a component of the antistatic reagent. Powder-free gloves that are certified to be free of chloride, nitrate, and sulfate are available.

In the laboratory, each filter should be transferred from its sealed manufacturer's packaging to a clean filter-handling container, such as a glass or plastic petri dish or petri slide, to reduce the risk of contamination. The filter should remain in this container, except for weighing, until it is loaded into a filter cassette prior to sampling. Each filter should have a unique identification number. If such a number is not provided by the filter manufacturer, a label that lists the filter number can be attached to the filter-handling container. It is recommended that each microbalance be assigned a block of filter numbers to be processed and used sequentially. Take care to avoid mistakenly assigning the same number twice or omitting a number. Color-coded labels may be helpful for separating groups of filters when more than one microbalance is being used.

If sufficient resources are available, bar coding for filters can be introduced. Bar code readers and printers for microbalances are already generally available and can significantly improve the efficiency of filter inventory tracking and processing.

#### **7.5 Filter Integrity Check**

All filters should be visually inspected for defects before the initial weighing. If any defects are found, discard the filter. Return any lot of filters containing a high number of defects to the supplier. Specific filter defects to look for are the following:

- C Pinhole—A small hole appearing (a) as a distinct and obvious bright point of light when examined over a light table or screen or (b) as a dark spot when viewed over a dark surface.
- C Separation of ring—Any separation or lack of seal between the filter and the filter border reinforcing the ring.
- C Chaff or flashing—Any extra material on the reinforcing, polyolefin ring or on the heat seal area that would prevent an airtight seal during sampling.
- C Loose material—Any extra loose material or dirt particles on the filter.
- C Discoloration—Any obvious discoloration that might be evidence of contamination.
- C Filter nonuniformity—Any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity or density across the face of the filter.
- C Other—A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

## 7.6 Filter Conditioning

New filters should be placed in the conditioning environment immediately upon arrival and stored there until the presampling weighing. Filters must be conditioned immediately before both the pre- and postsampling weighings. Filters must be conditioned for at least 24 hours (see Section 7.7) to allow their weights to stabilize before being weighed.

Filters must be conditioned at the same conditions (humidity within  $\pm 5$  percent RH) before the pre- and postsampling weighings. Mean RH must be held between 30 and 40 percent, with a variability of not more than  $\pm 5$  percent over 24 hours. However, where it can be shown that the mean ambient RH during sampling is less than 30 percent, conditioning is permissible at a mean RH within  $\pm 5$  percent RH of the mean ambient RH, but in no case less than 20 percent RH. Mean temperature should be held between 20 and 23 EC, with a variability of not more than  $\pm 2$  EC over 24 hours. RH and temperature should be measured and recorded on a continuous basis during filter conditioning (either by a recording hygrothermograph or by electronic instruments).

If spikes of temperature or, especially, RH occur during the conditioning period, the appropriate local decision maker should evaluate all relevant data and decide if the spikes are significant enough to compromise the conditioning period. The evaluation of the T and RH data should include blank performance in relation to the spikes. Experience with this comparison should eventually lead to the ability to estimate which spikes are too big. The evaluation should also include the significance of the timing of the spike in relation to the time of weighing, the closer to the weighing, the more significant the effect on the weight.

If the data recording device requires selection of an interval, a minimum of 5 minute averages might be used initially. In this situation, the data evaluator may use a rolling ( accumulative) average or standard deviation of of the averages for the 24 hours; these methods of comparing variability to the control limits ( $\pm 2$ EC or  $\pm 5\%$ RH) will appropriately minimize the effect of a spike at the beginning of the 24 hours; they will also inappropriately minimize the effect of a spike at the end of the 24 hour period (such as upon entering the weighing room prior to

weighing). Use of standard deviation of averages during the period will be more statistically representative of the conditions and will probably minimize spikes more than the rolling average method. Another consideration, for local decision makers, is whether a 95 or 99 % confidence interval is more acceptable.

An air-conditioned room may be used as a conditioning chamber, if RH and temperature can be maintained within the above specifications while filters are being conditioned. If the room's heating and ventilation system cannot maintain RH within these specifications, passive chemical or active electromechanical methods should be used to control RH. See ASTM (1993a) for information about maintaining constant RH by means of saturated saltwater (e.g., magnesium chloride) solutions in an airtight chamber. Chambers with active humidity and temperature controllers are available from laboratory supply firms. Conditioning chamber malfunctions, discrepancies, and maintenance activities should be recorded in a logbook or a laboratory notebook.

Within the conditioning chamber, the filters should be placed on a covered rack or an open-sided cabinet that will allow air circulation over the filters while reducing the chance that airborne material inside the chamber will settle onto the filters.

Care should be taken to avoid contaminating PM<sub>2.5</sub> filters inside the conditioning chamber with particulates released by other filter media (e.g., quartz and glass) that are also being conditioned in the chamber. Laboratory blanks (see Section 7.7) should be used to check for potential cross-contamination from airborne particulates inside the conditioning chamber. If there is evidence of such cross-contamination, corrective actions should be taken. One possible solution for a cross-contamination problem is maintaining separate conditioning chambers for PM<sub>2.5</sub> filters and for other filter media.

Filters should be conditioned in their filter-handling containers. Label, if possible, both the container's lid and bottom half. During conditioning, the lid should be placed either beneath the open container or partially covering the container. Care should be taken to avoid accidentally mixing up the filter identification numbers during conditioning. To improve filter inventory control, place the filters in the chamber in numerical order so that the analyst can more easily weigh the filters in numerical order.

Researchers in the arid western and southeastern portions of the United States have found that some Teflon<sup>®</sup> filters exhibit a loss of weight for a period of time after they are removed from their original shipping containers. The magnitude of weight loss varies from lot to lot and may be due to loss of volatile components from the polyolefin support ring or the glue used with some filters to adhere the Teflon<sup>®</sup> material to the support ring. In the arid West, weight loss of up to 150 µg has been observed (Desert Research Institute 1994). Some filters require at least 6 weeks to equilibrate.

In the Southeast, filter weight stability experiments were done as part of U.S. EPA's research to develop the volatility test now included in 40 CFR Part 53.66 of the revised requirements for designation of reference and equivalent methods for PM<sub>2.5</sub> (Eisner 1997). Small but still relatively significant (i.e., from 0 to 45 µg) weight losses were observed. These experiments showed that the problem could be addressed by active conditioning (e.g., passing HEPA-filtered air across the filters for a 1-hour duration) instead of passive conditioning. Active conditioning was conducted

with each filter sitting in the bottom of an open petri dish. Consecutive 4-hour periods of active conditioning of filters did not change the weight by more than  $\pm 5 \mu\text{g}$ .

**Note:** Typically, filters come packed together in large groups or in a container with separators. This package is usually contained inside another clear, reclosable plastic package, which may, in turn, be placed inside a box used in shipping. The more time that each filter is exposed to the conditioning environment, the more likely that its weight will be stable by the end of a conditioning period.

New filters should be removed individually from their sealed packages, placed in their own filter-handling containers (e.g., an uncovered petri dish), and conditioned for a sufficient time (see Section 7.7) to allow their weight to stabilize before use.

### 7.7 Lot Blanks, Laboratory Blanks, and Field Blanks

Three types of blank filters should be used. Lot blanks are unsampled filters used to determine filter weight stability over long periods of time (e.g., 1-6 weeks) due to the volatilization of material from the filter or to the absorption of gaseous material into the filter from the atmosphere. Laboratory blanks are conditioned, unsampled filters used to determine any weight change between pre- and postsampling weighings due to contamination in the microbalance environment. Field blanks are conditioned, unsampled filters used to determine whether similar contamination occurs during sampling.

The weight stability of filters can be determined by assigning three unsampled filters from each new filter exposure lot as exposure lot blanks. A filter lot is defined as a single shipment of filters from a filter manufacturer or from another source. A filter exposure lot is defined as a subsample of filters from the filter lot to be conditioned within a specific time period. After an initial 24-hour conditioning, these three exposure lot blanks are reweighed periodically (e.g., weekly/daily) and stored in the conditioning chamber (with the other filters) between weighings. These measurements should be recorded in the QC notebook (see Figure 7-1) or an equivalent database. These weighings should continue until the 24-hour weight change is less than  $15 \mu\text{g}$  (i.e., three times the precision for weighing unexposed filters). This filter weight stability experiment determines the period that the entire filter lot should be conditioned before it can be used for routine sampling. This experiment need not be continued during routine sampling but should be repeated when a new lot of filters is received.

Laboratory blanks should be kept inside the conditioning chamber except during weighing sessions. Weigh enough laboratory blanks during a presampling weighing session to provide at least one single-use laboratory blank during each subsequent postsampling weighing session. The pre- and postsampling weights should be recorded in the QC notebook and the laboratory data form (see Figures 7-1 and 7-2) or an equivalent database. If the weight change exceeds  $15 \mu\text{g}$ , contamination in the conditioning chamber may be occurring. Take appropriate troubleshooting and corrective actions.

Field blanks for manual (i.e., single-filter) samplers should be transported to the sampling site, momentarily installed in the sampler, removed, and stored in their protective containers, inside the sampler's case at the sampling site until the exposed filters are retrieved for postsampling weighing. In the case of sequential (i.e., multiple-filter) samplers, field blanks should be placed in

unused sampler slots, kept there during the sampling interval, and retrieved with the exposed filters. This approach presumes that unused slots are available and that the sampler can be programmed to not sample the field blank. If this cannot be done, follow the procedure used for the manual sampler. As a recommended best-case practice, site operators are encouraged to occasionally install field blanks in a nonactive manual sampler's filter holder over a 24-hour period during which the sampler is not scheduled to be sampling. This practice may be a useful troubleshooting technique.

Field blanks should be implemented at approximately 10% of a monitor's sampling frequency. Therefore, a monitor operating on a 1 in 6 day schedule would be expected to have approximately 6 blanks in a year while a monitor operating every day would be expected to have approximately 36 blanks. The pre- and postsampling weights should be recorded in the QC notebook and the laboratory data form or an equivalent database. If the weight change exceeds 30 µg, contamination during transportation or at the sampling site may be occurring. Take appropriate troubleshooting and corrective actions. Note: Since field blanks reflect the effect of factors occurring at **all** steps of the data collection process, evaluation of the effects of field factors requires removing the lab blank variability from the total field variability values and then looking at the remainder to see the variability due to factors having an effect in the cassette assembly, transport to the field, sampling, transport back to the laboratory, and recovery of the filters from the cassette. This consideration does not mean that the field blank acceptance criteria might be changed from 30 Fg or the laboratory blank criteria from 15 Fg by the field variability.

## 7.8 Electrostatic Charge Neutralization

Electrostatic charge buildup will prevent a microbalance from operating properly. Static charge is the accumulation of electrical charges on the surface of a nonconductive material. Common symptoms of this problem include noisy readout, drift, and sudden readout shifts. To reduce static charge within the balance, it may be necessary to place a radioactive antistatic strip containing a very small amount (i.e., 500 picocuries) of  $^{210}\text{Po}$  in the weighing chamber. It may also be necessary to pass each filter near, but not touching, an antistatic strip before it is weighed. See Engelbrecht et al. (1980), Hawley and Williams (1974), and Weil (1991) for more information about electrostatic charge and how to minimize its effects.

$^{210}\text{Po}$  antistatic strips are used to reduce electrostatic buildup in the microbalance's weighing chamber and on individual filters by charge neutralization. They neutralize electrostatic charges on items brought within an inch of them. These antistatic strips are safe, commonly available, and inexpensive.  $^{210}\text{Po}$  has a half-life of 138 days. Change the antistatic strips every 6 months and dispose of the old strips according to the manufacturer's recommendations. Antistatic solutions are available for coating (and, at appropriate and relatively infrequent intervals, recoating) the interior and exterior nonmetallic surfaces of the weighing chamber. This coating facilitates the draining of electrostatic charges away from these surfaces (by making them conductive) to a common ground to which the metallic conductive surfaces are connected. Earth-grounded conductive mats may also be placed on the weighing table surface and beneath the analyst's shoe surfaces to reduce electrostatic charge buildup.

Do not

assume that grounding eliminates all electrostatic buildup because the electrical ground may not be perfect. Even though a filter weight might stabilize within 30 to 60 sec and no weight drift is

observed during that period, the microbalance may still be influenced by some electrostatic buildup. It may still be necessary to repeat the neutralization procedure and to use antistatic strips inside the weighing chamber.

| Filter Lot Number C20102   |  | Analyst J. Armstrong                 |  |                                      |
|----------------------------|--|--------------------------------------|--|--------------------------------------|
| Balance Number A44603      |  | QC Supervisor R. Vanderpool          |  |                                      |
| Analysis Date              | Initial Working Standard 1 Weight (mg) | Final Working Standard 1 Weight (mg) | Initial Working Standard 2 Weight (mg) | Final Working Standard 2 Weight (mg) |
| 6/30/97                    | 100.000                                | 100.002                              | 199.999                                | 199.998                              |
| 8/13/97                    | 100.001                                | 100.001                              | 200.001                                | 200.000                              |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
| Filter Number <sup>a</sup> | Analysis Date                          | Presampling Mass (mg)                | Analysis Date                          | Postsampling Mass (mg)               |
| D-110 (LB)                 | 6/30/97                                | 136.546                              | 8/13/97                                | 136.550                              |
| D-111 (LB)                 | 6/30/97                                | 129.999                              | 8/13/97                                | 130.006                              |
| D-112 (LB)                 | 6/30/97                                | 130.633                              | 8/13/97                                | 130.645                              |
| R-700 (FB)                 | 6/30/97                                | 130.896                              | 8/13/97                                | 130.904                              |
| R-701 (FB)                 | 6/30/97                                | 128.339                              | 8/13/97                                | 128.345                              |
| R-702 (FB)                 | 6/30/97                                | 130.929                              | 8/13/97                                | 130.936                              |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |
|                            |  |                                      |  |                                      |

<sup>a</sup> Indicate blank measurements here (LB = lab blank, FB = field blank).

Figure 7-1. Example laboratory internal QC log.

| Filter Lot Number            | C20102                | Analyst                | J. Armstrong                 |
|------------------------------|-----------------------|------------------------|------------------------------|
| Balance Number               | A44603                | QC Supervisor          | R. Vanderpool                |
| Presampling Filter Weighing  | Date 6/30/97          | RH 33                  | Temp 22                      |
| Postsampling Filter Weighing | Date 8/13/97          | RH 38                  | Temp 21                      |
| Filter Number <sup>a</sup>   | Presampling Mass (mg) | Postsampling Mass (mg) | Net Mass Filter Loading (mg) |
| 100 mg (WS)                  | 100.000               | 100.001                | 0.001                        |
| 200 mg (WS)                  | 199.999               | 200.001                | 0.002                        |
| D-110 (LB)                   | 136.546               | 136.550                | 0.004                        |
| D-111 (LB)                   | 129.999               | 130.006                | 0.007                        |
| D-112 (LB)                   | 130.633               | 130.645                | 0.012                        |
| R-700 (FB)                   | 130.896               | 130.904                | 0.008                        |
| R-701 (FB)                   | 128.339               | 128.345                | 0.006                        |
| R-702 (FB)                   | 130.929               | 130.936                | 0.007                        |
| R-691                        | 139.293               | 139.727                | 0.434                        |
| R-692                        | 136.020               | 136.455                | 0.435                        |
| R-693                        | 135.818               | 136.260                | 0.442                        |
| R-694                        | 131.456               | 131.905                | 0.449                        |
| R-695                        | 137.508               | 137.973                | 0.465                        |
| R-696                        | 136.098               | 135.554                | 0.456                        |
| R-697                        | 131.029               | 131.483                | 0.454                        |
| R-698                        | 125.175               | 125.641                | 0.466                        |
| R-699                        | 131.165               | 131.633                | 0.468                        |
| R-691 (R)                    | 139.293               | 139.730                | 0.437                        |
| 100 mg (WS)                  | 100.002               | 100.001                | 0.001                        |
| 200 mg (WS)                  | 199.998               | 200.000                | 0.002                        |
|                              |                       |                        |                              |
|                              |                       |                        |                              |
|                              |                       |                        |                              |
|                              |                       |                        |                              |

<sup>a</sup>Indicate working standard (WS), lab blank (LB), field blank (FB), or replicate (R) measurement here.

Figure 7-2. Example laboratory data form.

Charge neutralization times may need to be longer than 60 sec for sampling situations in which (1) a high amount of charge has developed on collected particles due to their origin or (2) the particle loading on a filter is large. Electrostatic charge buildup becomes greater as the air becomes drier. A 60-sec charge neutralization may be sufficient in ambient indoor air conditioned to 37 percent RH and 23 EC but not in 20 percent RH and 23 EC in arid environments. This latter environment may require that the filter sit for more time on the antistatic strip. The longer neutralization period may have to be performed inside the weighing chamber or in a second small chamber used only for charge neutralization.

### 7.9 Presampling Filter Weighing (Tare Weight)

The reference method (EPA 1997) requires that the presample filter weighing be conducted within 30 days of the sampling period. The microbalance must be located in the same controlled environment in which the filters are conditioned. The filters must be weighed without intermediate or transient exposure to other conditions or environments.

This section presents procedures specific to a common commercially available microbalance. Calibration, QC checks (and acceptance tolerances), and operational procedures may have to be adapted for use with other microbalance models.

Researchers have found that the precision of mass measurements for unexposed filters based on replicate weighings is typically 3 to 5  $\mu\text{g}/\text{filter}$  (Desert Research Institute 1994). The precision on exposed filters is typically 5 to 8  $\mu\text{g}/\text{filter}$ . Precision for exposed filters with loadings heavier than 1  $\text{mg}/\text{cm}^2$  of filter surface area may approach  $\pm 2$  percent of the loading. The precision for net mass filter loadings (typically 6 to 9  $\mu\text{g}/\text{filter}$ ) is defined as the square root of the sum of the squares of the pre- and postsampling precision.

Follow these steps during the presampling filter weighing:

1. Record the RH and temperature of the conditioning chamber on the laboratory data form and in the laboratory QC notebook or database.
2. Clean the microbalance's weighing chamber with a fine brush, if necessary. Antistatic brushes for cleaning the microbalance and the surrounding area are available. Avoid using pressurized gas, which may blow damaging debris and oils into the microbalance's mechanism. Recoat the interior and external nonmetallic surfaces of the chamber with an antistatic solution, if necessary. Clean the surfaces near the microbalance with antistatic solution- or methyl alcohol-moistened disposable laboratory wipes. Clean the standard forceps with a lint-free cloth and the filter forceps with the moistened wipes. Allow the forceps to air-dry. Make sure the forceps are thoroughly dry before use. Even a small amount of moisture can cause a significant measurement bias.
3. To ensure maximum stability, it is recommended that the microbalance be turned on at all times. This procedure enables the microbalance to be operational at any time and eliminates the need for a warmup period before analyses are performed. Newer microbalances are always turned on (except for their displays) when they are plugged in.
4. Zero (i.e., tare) and calibrate the microbalance according to the manufacturer's directions. Many newer microbalances calibrate themselves automatically when the operator presses a

key.

- Using smooth, nonserrated, nonmetallic forceps, weigh two working mass reference standards (for example, a 100-mg standard and a 200-mg standard) as a QC check. Handle the working standards carefully to avoid damage that may alter their masses. Verify the working standards' masses every 3 to 6 months or after any incident of rough handling against the laboratory's primary standard weights (ASTM Class 1 or 1.1 standards). The working standards should bracket the mass of a blank or a loaded filter. Wait until the microbalance's display indicates that a stable reading has been obtained. Consult the microbalance's operating manual for more information about obtaining stable readings. Record the certified and measured values of these standards on the laboratory data form and in the laboratory QC notebook or database.

If the verified and measured values of a working standard disagree by more than 3  $\mu\text{g}$  (i.e., three times the microbalance's repeatability), reweigh the working standard. If the two values still disagree, troubleshoot and take appropriate corrective action, which may include (1) recertifying the working standards against the laboratory primary standards and/or (2) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

Weigh enough laboratory blanks during a presampling weighing session to provide at least 10 percent or one single-use laboratory blank during each subsequent postsampling weighing session. Weigh enough field blanks during a presampling weighing session to provide at least 10 percent or one single-use field blank during each subsequent postsampling weighing session. Record the presampling weights on the laboratory data form and in the laboratory QC notebook or database.

- Weigh the filters. Operate the balance according to the manufacturer's directions. Newer microbalances are so easily rezeroed that analysts should rezero the balance before each weighing. Take the filter from its filter-handling container (petri dish or equivalent) by gently slipping the filter-handling forceps under the outer polyolefin support ring. Hold the filter only by the ring, not by the filter material. Pass the filter, support ring side up, near a  $^{210}\text{Po}$  antistatic strip for 30 to 60 sec immediately prior to weighing. The antistatic strip should be inside the weighing chamber or as close to the chamber door as is practical. Immediately transfer the filter to the microbalance's pan and close the weighing chamber door. After the microbalance's display indicates that a stable reading has been obtained, record the balance number, the filter number, the filter lot number, and the filter's tare weight (presampling mass) on the laboratory data form.
- After approximately every 10th filter weighing, the analyst should reweigh at least one of the working standards. Record the measurement on the laboratory data form and in the laboratory QC notebook or database. If this measurement disagrees from the verified value by more than 3  $\mu\text{g}$  (i.e., three times the microbalance's reproducibility), reweigh the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

- At the end of the weighing session, reweigh both working standards. Record the measurements on the laboratory data form and in the laboratory QC notebook or database.
8. Any unused filter whose weight is outside the normal range (i.e., 110 to 160 mg) should be investigated. If there is a consistent negative replication ( $>15 \mu\text{g}$ ) for laboratory blank filters, it is usually a sign that the filters have not equilibrated long enough. In this case, troubleshoot and take appropriate corrective action.
  9. One routine filter should be reweighed at the end of the weighing session. Record the replicate measurement on the laboratory data form. If the replicate measurement disagrees from the original measurement by more than  $15 \mu\text{g}$ , reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.
  10. Return the filter to the filter-handling container, replace the lid to cover the filter, and return the container to the conditioning chamber to protect it from contamination prior to sampling.
  11. Check the filter cassettes and the backing screens for fractures, cracks, evidence of wear, or contamination. Clean or replace as necessary. The cassettes can be washed in a dishwasher and then rinsed with deionized water.
  12. When the time comes for the filters to be used at the sites (use must occur within 30 days of the initial weighing), install each filter in a filter cassette, and put the filter/cassette assembly into a protective container for transport to the sampler (see Figure 7-3). Attach a label with the filter identification number to the outside of the protective container. Double-check the entries in the laboratory data form (Figure 7-1). Prepare several extra filters in case a filter is invalidated during the installation process.
  13. If the filters will be mailed, the field operator should be supplied with reinforcing envelopes or some other means (in addition to the protective container) to protect exposed filters during their shipment back to the analytical laboratory.

### 7.10 Postsampling Documentation and Inspection

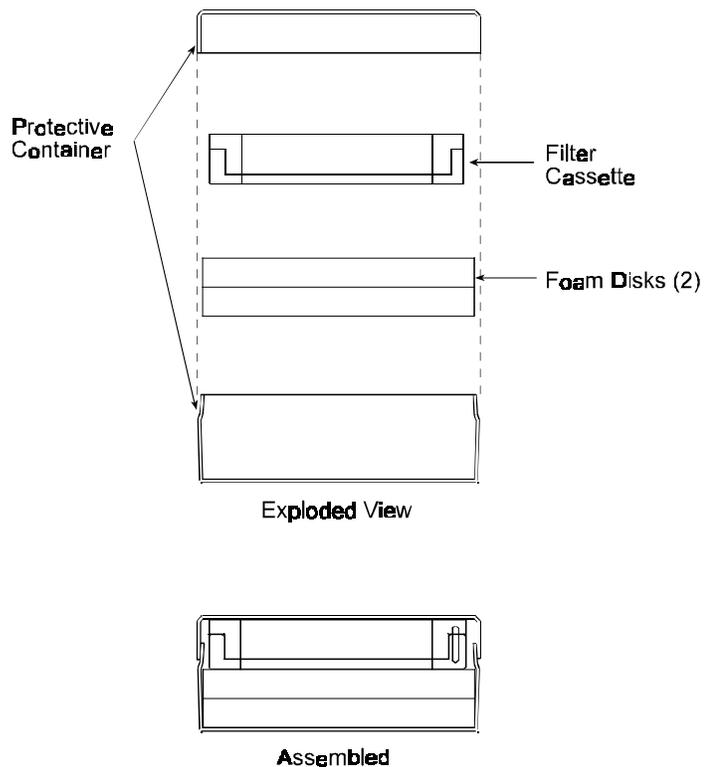
Upon receipt of the sample from the field, the analyst should follow these steps:

1. Examine the field data sheet. Determine whether all data needed to verify sample validity and to calculate mass concentration (e.g., average flow rate, ambient temperature and barometric pressure, and elapsed time) are provided. If data are missing or unobtainable from a field operator or if a sampler malfunction is evident, save the filter for inspection and record on the laboratory data form that the sample has been voided and the reason. Notify the appropriate personnel.

2. If the shipment was to be kept cold, for storage at 4 EC verify that the temperature of the cooler's interior was maintained at the desired point. This can be done by quickly reading the min/max thermometer that was placed in the container at the time of shipment or by inspecting the cool packs to determine if they are still frozen or cold. If the temperature is between 4 and 25EC, but is below the maximum temperature that was recorded during the filter sampling period, assess the temperature and note other relevant information and alert the QC decision maker to possible need to determine a shorter analysis deadline for the sample. All sample shipments that were intended to be shipped at 4EC and in fact were received cold should be refrigerated at approximately 4EC until several hours before the start of the filter conditioning period. If the protective container is cold, allow it to warm to the filter conditioning environment's temperature before opening to preclude water condensation on a cold filter. Remove the filter cassette from its protective container and examine the container. If particulate matter or debris is found in the protective container after the filter has been removed, record that the sample has been flagged as questionable and state the reason as a remark at the bottom of the laboratory data form. Save the filter for inspection. Notify the appropriate personnel.

3. Match the filter identification number with the correct laboratory data form on which the original microbalance number, filter number, presampling filter weight, and other information were inscribed. Group filters according to the microbalance used to determine their initial tare weights. Initial separation of filters in this way eliminates the risk of a measurement error that could result from the use of different microbalances for pre- and postsampling weighings.

4. Remove the filter from both the protective container and the filter cassette. Some cassettes may require special tools to disassemble them. **Note:** The stainless steel screen is sharp. Be careful! Be very careful when removing the filter from the cassette. Be careful not to touch or otherwise disturb the filter and its contents. Transfer the filter to a filter-handling container labeled with the corresponding filter number. Place the used filter in the container "dirty-side" up. Keep the particles from contact with the walls of the container. The



**Figure 7-3. PM<sub>2.5</sub> filter cassette and protective container.**

filter should be handled with clean, smooth forceps and should not be touched by hands. Inspect the filter for any damage that may have occurred during sampling. If any damage is found, note that the sample has been flagged as questionable and state the reason as a remark at the bottom of the laboratory data form. Save the filter for inspection. Notify the appropriate personnel.

5. Transfer the filter in its filter-handling container to the conditioning chamber.
6. Allow the filter to condition for at least 24 hours.
7. Remove the cassette and stainless steel backing screen to another area for cleaning.

### 7.11 Postsampling Filter Weighing (Gross Weight)

Both the pre- and postsampling filter weighings should be performed on the same analytical balance. Different analysts can perform the pre- and postsampling filter weighings as long as the appropriate standard operating procedures (SOPs) have been followed and the working standard and replicate measurements are within specifications. Use an effective technique to neutralize static charges on the filter. The postsampling conditioning and weighing shall be completed within 240 hours (10 days) after the end of the sampling period, unless the filter is maintained at 4 EC or below during the entire time between retrieval from the sampler and start of the conditioning, in which case the period shall not exceed 30 days.

Follow these steps during postsampling filter weighing:

1. Group filters in numerical order according to the microbalance used for preweighing and by their filter numbers. Reweigh each filter on the same microbalance on which its presampling weight was obtained.
2. Repeat Steps 1 through 11 in Section 7.9.
3. At least one laboratory blank and any available field blank (or approximately 10 percent of the weighed filters, if the number of filters being weighed is larger than 10) should be weighed. If the pre- and postsampling weights for the laboratory blanks disagree by more than 15  $\mu\text{g}$ , repeat the measurements. If the pre- and postsampling weights for the field blanks disagree by more than 30  $\mu\text{g}$ , repeat the measurements. If the two measurements still disagree, troubleshoot and take appropriate corrective action as specified in the reporting organization's QAPP. Measurements for sampled filters should not be corrected to account for blank measurements. High blank values should not cause the automatic invalidation of sampled filters that were measured during the same weighing session. Instead, high blank values should trigger troubleshooting and corrective action to reduce blank values to acceptable levels.
4. One routine filter should be reweighed at the end of the weighing session. Record the replicate measurement on the laboratory data form. If the replicate measurement disagrees from the original measurement by more than 15  $\mu\text{g}$ , reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

5. If the filter will receive further analysis, return it to the filter-handling container and note on the container and the laboratory data form that additional analyses are required. Transfer the filter to the laboratory responsible for performing the additional analyses.

### 7.12 Calculation of Net Mass Filter Loading

A filter's postsampling mass minus its presampling mass is the net mass loading for that filter. Record this value on the laboratory data form. Refer to Section 11.0, "Calculations, Validations, and Reporting of PM<sub>2.5</sub> Monitoring Data" for the calculations required to compute and report ambient PM<sub>2.5</sub> concentrations in µg/m<sup>3</sup>. Measurements for sampled filters should not be corrected to account for blank measurements.

### 7.13 Additional PM<sub>2.5</sub> Analysis Requirements

Section 2.8.1.5 of 40 CFR Part, Part 58, Appendix D states:

- (a) Within 1 year after September 16, 1997, chemical speciation will be required at approximately 25 PM<sub>2.5</sub> core sites collocated at Photochemical Assessment Monitoring Station (PAMS) sites (one type 2 site per PAMS area) and at approximately 25 other core sites for a total of approximately 50 sites. The selection of these sites will be performed by the Administrator in consultation with the Regional Administrator and the States. Chemical speciation is encouraged at additional sites. At a minimum, chemical speciation to be conducted will include analysis for elements, selected anions and cations, and carbon. Samples for required speciation will be collected using appropriate methods and sampling schedule in accordance with procedures approved by the Administrator.
- (b) Air pollution control agencies shall archive PM<sub>2.5</sub> filters for all other SLAMS sites for a minimum of 1 year after collection. These filters shall be made available for supplemental analyses at the request of EPA or to provide information to State and local agencies on the composition for PM<sub>2.5</sub>. The filters shall be archived in accordance with procedures approved by the Administrator. Storage conditions will likely involve temperature control (1 to 4EC), and protection from light, vibrations, and dust sources.

### 7.14 Internal Quality Control

Keep a laboratory QC notebook or database (with disk backups), which should contain QC data, including the microbalance calibration and maintenance information, routine internal QC checks of mass reference standards and laboratory and field filter blanks, and external quality assurance (QA) audits. These data will duplicate data that are already recorded on laboratory data forms but will consolidate them so that long-term trends can be identified. It is recommended that QC charts be maintained on each microbalance and included in this notebook. These charts may allow the discovery of excess drift that could signal an instrument malfunction.

At the beginning of each weighing session, after the analyst has completed calibrating the microbalance and measuring the two working standards, weigh the laboratory and field blanks. Weigh enough laboratory blanks during a presampling weighing session to provide at least one laboratory blank during each subsequent postsampling weighing session. Weigh enough field blanks during a presampling weighing session to provide approximately 10% field blanks.

Reweigh at least one of the routinely exposed, sampled filters and the two working standards at the end of the weighing session.

Record the working standard, blank, and replicate measurements on the laboratory data form and in the laboratory QC notebook or database. If the working standard measurements differ from the verified values or the presampling values by more than 3  $\mu\text{g}$  (i.e., three times the microbalance's repeatability), repeat the working standard measurements. If the laboratory blank or replicate measurements differ from the presampling values or previous postsampling values by more than 15  $\mu\text{g}$  (i.e., three times the precision for unexposed filters), repeat the blank or replicate measurements. If the pre- and postsampling weights for the field blanks disagree by more than 30  $\mu\text{g}$ , repeat the measurements. If the two measurements still disagree, troubleshoot and take appropriate corrective action as specified in the reporting organization's QAPP. Measurements for sampled filters should not be corrected to account for blank measurements. High blank values should not cause the automatic invalidation of sampled filters that were measured during the same weighing session. Instead, high blank values should trigger troubleshooting and corrective action to reduce blank values to acceptable levels.

If more than one microbalance is used, the pre- and postsampling measurements of the filter should be made on the same microbalance.

The QC or laboratory supervisor should certify on the laboratory data forms the acceptability of filter weighings and QC checks and the completeness of the data. The QC or laboratory supervisor should sign or initial each completed form. When bound together, these forms can serve as a laboratory data notebook.

## 8.0 Field Operation

### 8.1 Overview

Sampling operations provided in this section are independent of any one type of commercially available PM<sub>2.5</sub> sampler. Because operational procedures may vary among sampler models, consult the manufacturer's operations manual before the sampler is put into service. Sampling procedure checks are summarized in Table 8-1.

### 8.2 Activities to Perform Each Site Visit

This section reviews the steps taken during a site visit to prepare for and complete a sample run, sample validation criteria, and proper handling of the filter/cassette assembly. Also consult the sampling procedures checklist in Figure 3-3.

#### 8.2.1 Beginning a Run

Care must be taken to ensure that the filter is clean and undamaged before it is installed in the sampler. The filter/cassette assembly should be kept in its protective container until installation; damaged filters must be placed in the protective container and returned to the weighing laboratory, where they will be examined and then discarded.

1. Annotate the following on a data sheet (or in a computer file) such as the Single-Filter PM<sub>2.5</sub> Sampler Run Data Sheet shown in Figure 8-1:

**Note:** Figure 8-1 is merely an example data sheet for a single-filter sampler and assumes the data sheet will accompany the sample to the laboratory. Because sampler data are downloaded and archived, table entry of data relative to run start and end operating conditions may not need to be made.

- Date and time of sampler setup visit
  - Site identification and location
  - Sampler model, unique sample ID number (this may be the cassette and/or filter ID number; or some other tracking number)
  - Sample start date and time
  - Unusual conditions that may affect samples (e.g., subjective evaluation of pollution on that day, construction activity, weather conditions)
  - Setup operator's signature or initials.
2. Ensure the sampler is not operating. If the sampler is set to automatically begin operation, ensure that enough time is available to complete these setup procedures before it starts.
  3. Open the filter holder assembly according to the manufacturer's instructions. Visually inspect the O-rings inside the filter holder to ascertain that they are present and secure. Do not sample without these O-rings installed because the system will no longer be leak-free. Install the uniquely identified filter cassette containing the preweighed filter. Never remove

the filter from the cassette. This is done only at the filter weighing facility. Reinstall the filter cassette holder and ensure that the fittings around the impactor housing and the filter assembly are secure.

4. The sampler is now ready to sample. If the sampler is not already set to turn on automatically for the next sampling period (generally at midnight), program the controls to do so.
5. Visually inspect the monitoring site and its equipment to ensure that all sampling components are ready for the next run day(s). Note any changes in the site surroundings, especially dust-producing activities.
6. Visually inspect the records of the sampler. If it is time for the monthly/every 4 weeks check, measure and record independent measurements of ambient temperature and pressure, and ensure that the ambient temperature (i.e., inlet temperature) and pressure readings taken by the sampler are within 4.0 EC and 10 mmHg of the independent readings, respectively. Be certain that the independent temperature sensor (thermometer or thermistor probe) is located side-by-side with the sampler's ambient temperature sensor. This will require that the sensor be placed in the louvers of the sampler's temperature screen and kept out of direct sunlight. Also check the sampler's display for the filter temperature and ensure this value is reasonable compared to the ambient temperature display.

### 8.2.2 Ending a Run

1. Visually inspect the sampler readouts to ensure that the sampler is operating properly. Sequential samplers require manipulation of a number of display screens to retrieve all data. Consult the operating manual. Also check the sampler for any other obvious problems, such as a full water collection jar. If problems are identified, describe them on the sample run data sheet and take corrective actions before starting another run. If the weather is bad, provide a temporary shelter to facilitate data transfer and to protect exposed parts of the sampler. A small work table may be useful.
2. Record the following information on the sampler run data sheet (or save it to a data file):
  - Date and time of postsampling site visit; then display the sampler readout or downloaded computer file and record
  - Stop time and total elapsed time of the sample run
  - Final flow rate, average flow rate, coefficient of variation of the flow rate, and total volume sampled
  - Sampler's indicated ambient temperature and barometric pressure at the end of the run
  - Also record, if your schedule calls for it, the current temperature, pressure, and flow rate of the sampler
  - Conditions at the site or of the collector that may have affected the sample
  - Any flags triggered by the sampler (e.g., power outage, flow rate variation)

Table 8-1. Sampling Procedure Checks

| Procedure   | Frequency and method   | Requirements  | Action if requirements not met   |
|---|--|---|--|
| Filter installation<br><i>(Never remove filter from cassette for inspection!)</i> | Visually check filter. Install filter in cassette, and securely close filter holder.   | Filters must be uniquely identified, tare-weighed, undamaged, and in cassette.  | Void the filter, and install substitute filter/cassette.   |
| Sample validation and documentation   | Visually check each sample and the keypad display or downloaded sampler data for completeness.   | Record sampling date, filter and sampler ID, station location, flow rates, sample time, and unusual conditions on data sheet or computer screen.  | Complete or correct the documentation. Cross out invalid information with a single line. Initial and date changes. |
| Postsample inspection   | Visually check filters while in cassettes for tears, missing pieces, or leakage. Review sampler operation.   | There should be no evidence of filter damage or sampler malfunction.  | Flag the sample as questionable; correct the cause of malfunction.   |
| Leak checks   | Check for leaks when suspected or after every-fifth-sample-day service of WINS impactor.   | Leak-check results must be within parameters specified by manufacturer.   | Determine cause of leak and correct. Validate and/or calibrate the sampler flow rate.                              |
| Flow rate checks  | Check flow rate at least every 4 weeks or once per month at each collector in the network.   | Indicated sampler flow rate must be within $\pm 4$ percent of the measured flow rate.   | Correct problems. Recalibrate the sampler if needed.   |
| Field blank check   | At least one field blank available per weighing session. Install, then immediately remove filter from sampler; store in protective container inside sampler case. Or preferably, install in idle single-filter sampler for 24 hours, then remove and process. For sequential samplers, install in unused holder. | Rotate from sampler to sampler so all are checked. Special requirements for sequential samplers: should have one or more field blanks with each batch of filters to be weighed. Refer to Section 7.0. | Reassess filter-handling techniques and storage conditions.  |

| <b>Single-Filter PM<sub>2.5</sub> Sampler Run Data Sheet</b>                                   |  |   |   |
|--|--|---|---|
| Date/Time of Setup Visit <u>8/7/97; 15:30</u>  |  | Date/Time of Postsampling Visit <u>8/11/97; 10:15</u> |   |
| Site Identification <u>NC-723</u>  |  | Site Name <u>Research Triangle Park</u>               |   |
| Sampler Model <u>O-A-2013</u>  |  | Sampler ID <u>A1</u>                                  | Filter ID <u>R691</u>                     |
| Start Date <u>8/8/97</u>   | Stop Date <u>8/8/97</u>  | Well No. and Installation Date <u>#4; 8/7/97</u>      |   |
| Start Time <u>00:00 (midnight)</u>   | Stop Time <u>24:00 (midnight)</u>                                    | Elapsed Time <u>24.0</u> hr                           |   |
| <b>FIELD</b>   |  |   |   |
| Conditions at Time of Initial Site Visit <u>8/7/97; 15:30</u>                                  |  |   |   |
| Sampler:   |  | Indicated P <sub>a</sub> : <u>752.2</u> mmHg          | Indicated T <sub>a</sub> : <u>24.0</u> EC |
|  |  | Indicated Q: <u>N/A</u> L/min                         |   |
| Check Data (if scheduled):   |  | P <sub>a</sub> <u>754</u> mmHg                        | Measured using: <u>Field barometer #4</u> |
|  |  | T <sub>a</sub> <u>24</u> EC                           | Measured using: <u>ASTM thermometer</u>   |
| Initial and Final Conditions (as retrieved from sampler screen or data storage, if available): |  |   |   |
|  |  | <b>Run Start</b>                                      | <b>Run End</b>                            |
| Indicated P <sub>a</sub>   | <u>754.0</u>   | <u>758.4</u>  | mmHg                                      |
| Indicated Q  | <u>16.64</u>   | <u>16.60</u>  | L/min                                     |
| Indicated T <sub>a</sub>   | <u>22.4</u>  | <u>23.5</u>   | EC  |
| Indicated Q <sub>avg</sub>   | <u>—</u>   | <u>16.67</u>  | L/min                                     |
| Indicated Q <sub>cv</sub>  | <u>—</u>   | <u>0.3</u>  |   |
| Volume sampled   | <u>—</u>   | <u>24.03</u>  | m <sup>3</sup>                            |
| Sample Removal Date/Time <u>8/11/97 10:30</u>  |  | Sample Ship Date <u>8/11/97</u>                       |   |
|  |  | No  | Yes                                       |
| Was Sample Shipped Cold?   |  | <input checked="" type="checkbox"/>                   | <input type="checkbox"/>                  |
|  |  | No  | Yes                                       |
| Questionable Sample?   |  | <input checked="" type="checkbox"/>                   | <input type="checkbox"/>                  |
| (If yes, explain in notes)   |  |   |   |
| <b>LABORATORY</b>  |  |   |   |
| Filter Information:  |  | Sample Receipt Date/Time <u>8/12/97 15:00</u>         |   |
| Filter ID: <u>R691</u>   | Initial Weight: <u>139.293</u> mg                                    | Final Weight: <u>139.727</u> mg                       |   |
| Calculated Concentration: <u>18.06</u> µg/m <sup>3</sup>                                       | Interior Temp. of Filter Transit Container Upon Opening <u>22</u> EC |   |   |
| Notes: _____   |  |   |   |
| Setup Operator: <u>J. Jones</u> <u>8/7/97</u>  |  | End Operator: <u>A. Smith</u> <u>8/11/97</u>          |   |
| Reviewed by: <u>A. Boss</u> <u>8/15/97</u>   |  |   |   |

Figure 8-1. Example PM<sub>2.5</sub> sampler run data sheet.

- C Explanations for questionable or voided samples
- End operator's signature or initials.
3. Download the runtime data for the completed run using a laptop computer or other data transfer device that may be specified in the particular sampler's operating manual.
  4. Carefully open the sampler's filter holder assembly according to the manufacturer's instructions. If the filter cassette comes apart or sticks to the upper housing during this process, close and gently reopen the assembly. Do not allow the filter to be shaken, dropped, or touched by any foreign object (fingers, rain, and so on). Visually examine the filter and cassette for damage or unusual appearance. Make notes and then immediately place the filter cassette inside an appropriately marked protective container for storage and later transport to the weighing laboratory.
  5. Inspect the interior of the filter housing and the sampler itself. Note any abnormalities on the sampler run data sheet.
  6. Conduct any scheduled maintenance activities. Refer to Section 9.2.2 for details on impactor well inspection, cleaning, and/or replacement.
  7. If another sampling run will be performed, install a filter cassette according to the instructions in Section 8.2.1, above, and the manufacturer's instructions.
  8. Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the run data sheet. Retrieve equipment and documentation. Secure the site.

### 8.2.3 Sample Validation

Observations made by the site operator concerning the sampler's operation are most important in deciding if a sample is valid. Never discard a filter/cassette. Operators are encouraged to use data qualifiers or flags and to record free-form notes on the data sheet, in a site logbook, or on computer data entry screens. The final decision for invalidation will be made after the information provided by the operator and the filter/cassette itself are examined at the office or laboratory. Tight control of sampler operation is important because too many invalid samples could cause an entire quarter's data set to be lost. The following criteria have been established to assist the operator in determining whether or not a sample is valid:

#### 1. Timing

All samplers must operate for at least 23 but not more than 25 hours (1,380 to 1,500 min). Samples taken for less than 23 hours are invalid for the daily average but can be used to determine exceedances of the PM<sub>2.5</sub> standard. Samplers are programmed to stop whenever the filter becomes so heavily loaded that proper flow rate cannot be maintained.

#### 2. Flow Rates

The average flow rate for a 24-hour period must be within 5 percent of 16.67 L/min at actual conditions. If this limit is exceeded, investigate the cause. Use the following criteria as a basis for sample invalidation:

- Deviations in flow rate during sampling (due to mechanical failure) of more than 5 percent from the design value require a flow rate verification check. Refer to Section 8.3.2.3 for information. Remember to use a flow check filter, not one used for sampling. If the sampler's flow rate verification check indicates that the comparison did not meet the  $\pm 4$  percent acceptance criteria, the sample may be questionable and should be flagged for potential invalidation.
- If the sampler flow rate decreased because of heavy particulate loading on the filter, the sample should not be invalidated because the heavy loading may indicate an episodic situation that deserves study.
- Changes in flow rate calibration of more than 4 percent, as determined by a monthly field flow rate verification check, may cause invalidation of all samples collected since the last acceptable flow rate check. Make corrections or repairs, verify the flow rate, and recalibrate the sampler if required.

### 3. Filter Quality

A sample collected on any filter that has obviously been damaged (i.e., torn, frayed, or has pinholes) during the collection process should be invalidated. Return such a filter/cassette assembly to the office or laboratory with notes from its inspector.

### 4. Filter Temperature

Based on the data downloaded from the sampler for the run, the filter temperature must be no greater than 5 EC above the ambient temperature for more than 30 consecutive min, based on 5-min averages. Take corrective action if it is.

## 8.2.4 Sample Handling

### 8.2.4.1 Handling a Valid Sample

1. The sample must be removed from the collector within 96 hours (4 days) of the end of the run. Promptly deliver the filter cassette in its protective container, accompanied by the completed run data sheet, to the analytical laboratory. Depending on whether the sample is shipped or transported at near-ambient temperatures or at 4 EC, the laboratory has from 10 to 30 days from the end of the run to determine the sample weight. Package and handle the samples to avoid vibrations that may dislodge particles. Do not allow the filter surfaces to touch any of the interior surfaces of the protective container. The container must be transported in an insulated container capable of maintaining a temperature of less than 25 EC (77 EF) unless the transit time to an air-conditioned building or refrigerator is only a few hours. If desired, the sample may be cooled to 4 EC by placing leak-proof ice substitutes inside the insulated container. Do not allow the protective container to become wet. Place a min/max thermometer or a liquid crystal temperature sensor strip inside the shipping container to verify that the sample(s) remained cool during transit to the laboratory. Handle field blank filters in the same manner.

### 8.2.4.2 Handling a Questionable Sample

1. Complete as much of the run data sheet as possible and explain any omissions.
2. Mark “questionable” on the run data sheet and record comments on the data sheet and/or in the site logbook.
3. Do not discard the filter.
4. Deliver the filter cassette in its container (as if it were a valid sample) and the run data sheet to the analytical laboratory, where a final decision on sample validity will be made. If your quality assurance (QA) program calls for it, notify the appropriate person of the potentially invalid sample.

## 8.3 Activities to Perform Every Five Operating Days

### 8.3.1 Impactor Well Cleaning

Cleaning of the impactor well is strongly recommended after every 5 days of operation. Also empty any water from the water trap, clean the sampler interior, inspect the seals, and reinstall the trap. Cleaning the well is necessary to prevent the sampler from operating with an overloaded  $PM_{2.5}$  impactor. When operating in an area with higher  $PM_{10}$  concentrations, more frequent cleaning of the well may be necessary. Refer to Section 9.2.2 for impactor maintenance guidance.

Impactor wells may be cleaned at the field site. Take care to thoroughly clean the well and not to introduce contaminants. Others may prefer to have spare wells that can be cleaned in a laboratory or other indoor location. The well should be transported to the sampling site in an upright position. During transport, protect the well from excessive vibration, precipitation, direct sunlight, and other harmful environments. Likewise, the used well should be protected in transit back to the laboratory. It is recommended that wells be numbered or otherwise identified and records kept of their use.

In the case of sequential samplers in use every day, sampling may be interrupted for up to 1 hour without invalidating the day's sample. Exercise care not to contaminate the filter during the well cleaning or replacement process.

## 8.4 Activities to Perform Every Four Weeks

### 8.4.1 Flow Rate Verification Check

A flow rate verification check of the sampler flow rate is recommended every month. Control charts presenting flow rate verification check data (indicated vs. measured) should be maintained. These charts provide a reference of instrument flow rate drift patterns and indicate when flow limits ( $\pm 4$  percent) have been exceeded. The field check is made by installing a measuring device (which is traceable to National Institute of Standards and Technology (NIST) and calibrated within the range of the flow rate) on the inlet of the sampler. Calibration procedures for the measurement device are referenced in Section 6.3.4.

Calibration checks of the sampler flow rate require the instrument to be running. The following flow rate verification procedures are independent of any device. A variety of transfer standards

may be used with this procedure; however, the necessary apparatus and subsequent calculations to determine the sampler's flow rate will vary.

#### 8.4.2. Field Check Apparatus

The following equipment is required for field calibration checks of temperature, pressure, and flow rate:

- A thermometer readable to the nearest 0.1 EC, capable of accurately measuring temperature to the nearest  $\pm 1$  EC, and referenced to a NIST standard or an American Society for Testing and Materials (ASTM) thermometer within  $\pm 0.5$  EC at least annually
- A barometer capable of accurately measuring ambient barometric pressure to the nearest  $\pm 1$  mmHg and referenced to a NIST or ASTM barometer within  $\pm 5$  mmHg at least annually
- An orifice device (or other acceptable flow measurement device) and calibration relationship
- The sampler's calibration information (an equation)
- A clean flow check filter, installed in a cassette
- A flow check data sheet (such as the example given in Figure 8-2) or the sampler's logbook.

#### 8.4.3 Leak Check

Before conducting quality control (QC) field calibration checks of temperature, pressure, and flow rate, perform a sampler leak check as follows:

1. Insert the clean filter (designated the "leak check filter") into the sampler filter holder as described in the operating procedure in Section 8.1. Leak check filters should never be used for subsequent sampling. The same filter may be used for the leak check that was used for the flow rate verification check.
2. Remove the inlet and install the flow rate adapter on the top of the downtube (see Figure 6-2). Close the adapter valve to prevent air flow.
3. Perform the leak check procedure according to the specific instructions in the manufacturer's operating manual. Also refer to Section 6.6 of this document.

#### CAUTION

Open the flow rate adapter's valve slowly to prevent a sudden rush of air into the system, which could cause oil to splash from the impactor well onto the filter and other components.

4. If the sampler leak check is within acceptable limits as detailed in the operating manual, the sampler is operating properly and you may proceed with the temperature, pressure, and flow rate checks. If the limit is exceeded, investigate and correct any malfunction. If necessary, recalibrate the sampler before sampling is resumed.
5. Turn off the sampler, remove the flow audit device, and replace the inlet.
6. Remove the leak check filter from the filter holder. This filter is never reused for sampling

but, if carefully handled, may be used several times as a leak check filter.

### PM<sub>2.5</sub> Sampler Flow Check Data Sheet

Date of Flow Check \_\_\_\_\_

Time of Flow Check \_\_\_\_\_

Site Identification \_\_\_\_\_ Site Name \_\_\_\_\_

Sampler Model \_\_\_\_\_ Sampler ID \_\_\_\_\_ Filter ID \_\_\_\_\_

Sampler Indicated P<sub>a</sub> \_\_\_\_\_ mmHg T<sub>a</sub> \_\_\_\_\_ EC \_\_\_\_\_ Other P<sub>a</sub> \_\_\_\_\_ mmHg T<sub>a</sub> \_\_\_\_\_ EC \_\_\_\_\_

Unusual Conditions: \_\_\_\_\_

---

Flow Check Device \_\_\_\_\_ S/N \_\_\_\_\_ Calibration Date \_\_\_\_\_

Flow Check Device Calibration Relationship: m: \_\_\_\_\_ b: \_\_\_\_\_ r: \_\_\_\_\_

| Measurement No. | Indicated Flow, L/min<br>(Q <sub>ind</sub> ) | Measured Flow, L/min<br>(Q <sub>ref</sub> ) |
|-----------------|--|---|
| 1               |  |   |
| 2               |  |   |
| 3               |  |   |
| 4               |  |   |
| 5               |  |   |
| Average         |  |   |

Percent Difference from Reference Measured Flow Rate (a) \_\_\_\_\_

(a) Percent Difference = [(Average Q<sub>ind</sub> - Average Q<sub>ref</sub>)/Average Q<sub>ref</sub>] × 100

Percent Difference from Design Flow Rate (b) \_\_\_\_\_

(b) Percent Difference = [(Average Q<sub>ind</sub> - 16.67)/16.67] × 100

Operator's Signature/Date \_\_\_\_\_

Reviewer's Signature/Date \_\_\_\_\_

Figure 8-2. Example PM<sub>2.5</sub> sampler flow check data sheet.

#### 8.4.4 Procedure for Field Flow Rate Verification Check

1. Insert the clean filter (designated the “flow check filter”) into the sampler filter holder as described in the operating procedure in Section 8.1. Flow check filters should never be used for subsequent sampling but, if carefully handled, may be used several times as a flow check filter.
2. Turn on the sampler and allow it to warm up to operating temperature (approximately 15 min).
3. Read and record the following information on a sampler flow check data sheet such as the example given in Figure 8-2.
  - Ambient temperature ( $T_a$ ), EC (as indicated by the sampler’s display)
  - Ambient temperature ( $T_a$ ), EC (as indicated by the field check thermometer or thermistor)
  - Ambient barometric pressure ( $P_a$ ), mmHg (as indicated by the sampler’s display)
  - Ambient barometric pressure ( $P_a$ ), mmHg (as indicated by the field check barometer)
  - Sampler ID number and model
  - Flow verification check device serial number and calibration relationships (if appropriate)
  - Date, location, and operator’s signature.
4. Remove the inlet from the sampler downtube and replace it with the flow verification check device. The installation of an orifice/electronic manometer flow verification check assembly is shown in Figure 8-3. Allow time for the flow verification check device to reach ambient temperature

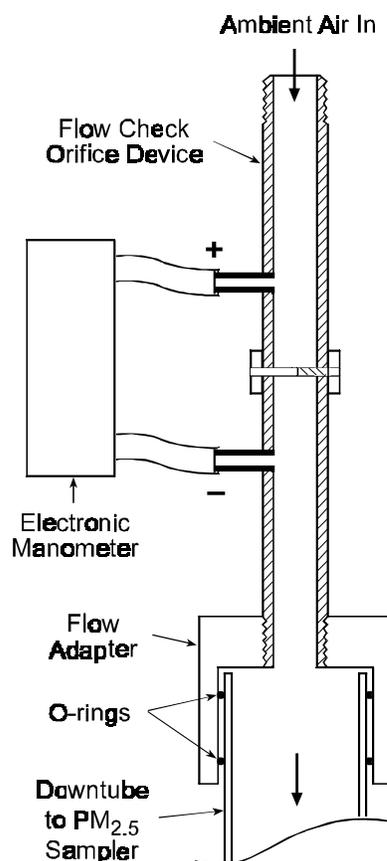


Figure 8-3. Example of installation of sampler flow verification check assembly.

before use.

5. Record the flow rate measured by the flow verification check device and the actual flow rate indicated by the sampler. This process be repeated for a total of at least five measurements if climate conditions permit. Take the average of the recorded measured flow rates and the average of the indicated flow rates.
6. Using the above information, calculate the percentage difference as:

$$\% \text{ difference} = [(indicated_{avg} - measured_{avg}) / measured_{avg}] \cdot 100\% .$$

7. If the sampler flow rate is within 96 to 104 percent of the measured flow rate (at actual conditions), **and** if the sampler flow rate is within 95 to 105 percent of the design flow rate of 16.67 L/min, the sampler is operating properly. If either limit is exceeded, repeat the leak check procedure, as stated in Section 8.3.2.2. After doing so, investigate and correct any malfunction. If necessary, troubleshoot the sampler further, recheck the flow, and, if necessary, recalibrate before sampling is resumed.
8. Turn off the sampler, remove the flow verification check device, and replace the inlet.
9. Remove the flow verification check filter from the filter holder.
10. Set up the sampler for the next sampling period according to the operating procedure in Section 8.1.
11. Have a supervisor or QC reviewer sign and date the sampler flow check data sheet.

## 9.0 Sampler Maintenance

### 9.1 Overview

Preventive maintenance is defined as a program of planned actions aimed at preventing failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and to provide for more complete data acquisition. This section addresses maintenance associated with reference or Class I equivalent PM<sub>2.5</sub> monitoring methods only. Maintenance of the laboratory microbalance and support equipment is not covered. Laboratories often employ a balance service representative who services balances on a 6-month or annual basis.

Some sampler maintenance can be performed at the field site. Major maintenance of the sampler or components, such as the pump, can be performed more conveniently when the equipment is brought to a laboratory or maintenance facility, provided time and labor are available to move the equipment.

A maintenance schedule should be established for each sampler and systematic records should be kept as scheduled and unscheduled maintenance occurs. Files should reflect the history of maintenance, including all replacement parts, suppliers, and cost expenditures and should include an inventory of on-hand spare equipment for each sampler.

Recommended supplies for all maintenance activities include an alcohol-based general-purpose cleaner that leaves no residue, cotton swabs, a small soft-bristle brush, paper towels, distilled water, and miscellaneous hand tools. Additional supplies may be necessary for specific procedures, as indicated below. Specifications for many of the supplies are given in Section 4.0, "Procurement of Equipment and Supplies." of this document. A compressed-air source is also recommended but not required.

All activities recommended below are in addition to those specified in the operating manual specific to the sampler and are not intended to replace those activities. Maintenance activities are summarized in Table 9-1.

**Note:** Always remove the impactor and filter cassette prior to removing the inlet downtube or cleaning any of the instrument parts upstream of these items. Such activities could dislodge dirt, oil, grease, or other materials that could deposit into the impactor well or onto the sampling filter. Temporarily store the impactor and filter cassette in a clean, dry location, away from contaminating materials (dust, dirt, rain, and so on) and direct sunlight.

### 9.2 Five-Day Maintenance Procedures

#### 9.2.1 Water Collector

At least every 5 sampling days, inspect the water collector bottle. Remove accumulated water, clean the interior of the bottle, inspect the seals, and replace the bottle in the holder.

**Table 9-1. Summary of PM<sub>2.5</sub> Sampler Maintenance Items**

| Frequency <sup>a</sup>     | Maintenance item <sup>b</sup>  |
|----------------------------|--|
| Every 5 sampling days      | <ol style="list-style-type: none"> <li>1. Service water collector bottle.</li> <li>2. Clean or change-out impactor well.</li> </ol>  |
| Monthly                    | <ol style="list-style-type: none"> <li>1. Clean sampler inlet surfaces.</li> <li>2. Clean impactor housing and impactor jet surfaces. Examine O-rings.</li> <li>3. Clean interior of sampler case.</li> <li>4. Check sampler clock accuracy.</li> <li>5. Check condition of sample transport containers.</li> </ol>  |
| Quarterly (every 3 months) | <ol style="list-style-type: none"> <li>1. Inspect O-rings of inlet. Remove and lightly coat them with vacuum grease.</li> <li>2. Clean sampler downtube.</li> <li>3. Inspect and service water seal gasket where downtube enters sampler case.</li> <li>4. Inspect and service O-rings of impactor assembly.</li> <li>5. Inspect and service vacuum tubing, tube fittings, and other connections to pump and electrical components.</li> <li>6. Inspect and service cooling air intake filter and fans.</li> </ol> |
| Other periodic maintenance | <ol style="list-style-type: none"> <li>1. As recommended by the manufacturer's sampler manual.</li> </ol>  |

<sup>a</sup>Frequency may vary depending on climate, amount of particulate matter in the air, weather, and so on.

<sup>b</sup>Remove impactor and filter cassette before servicing any upstream sampler components.

### 9.2.2 Impactor Well

At least every 5 sampling days, disassemble and clean the impactor well (refer to Figure 9-1). If spare wells are available, cleaning can be done in the laboratory. Separate the upper and lower portions of the well, remove the used filter from the well, wipe the two halves of the well clean with lint-free laboratory tissues or cloth, put a new borosilicate glass filter in the well's bottom, and add  $1 \pm 0.1$  mL of impactor oil. Check to be sure the oil covers the filter uniformly and there are no air bubbles beneath the filter. Reassemble the well and place it in the impactor. To reduce preparation time, several spare impactor wells should be prepared at one time and stored in a clean, dust-free container until needed.

## 9.3 Monthly Maintenance Procedures

### 9.3.1 Inlet

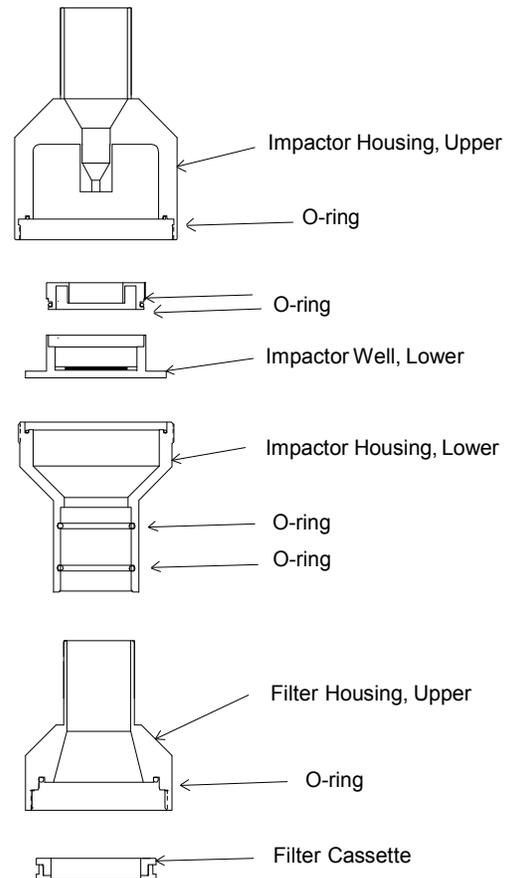
Figure 9-2 shows a disassembled sampling inlet. The inlet seals to the downtube with two O-rings.

If the water collector attached to the side of the inlet is glass, care should be taken during handling not to crack or break it because the sampler will not maintain an adequate vacuum

during operation then. The glass water collector may be either replaced with a plastic jar or wrapped with insulating tape to minimize the chance of accidental breakage.

To dismantle and clean the sampler inlet

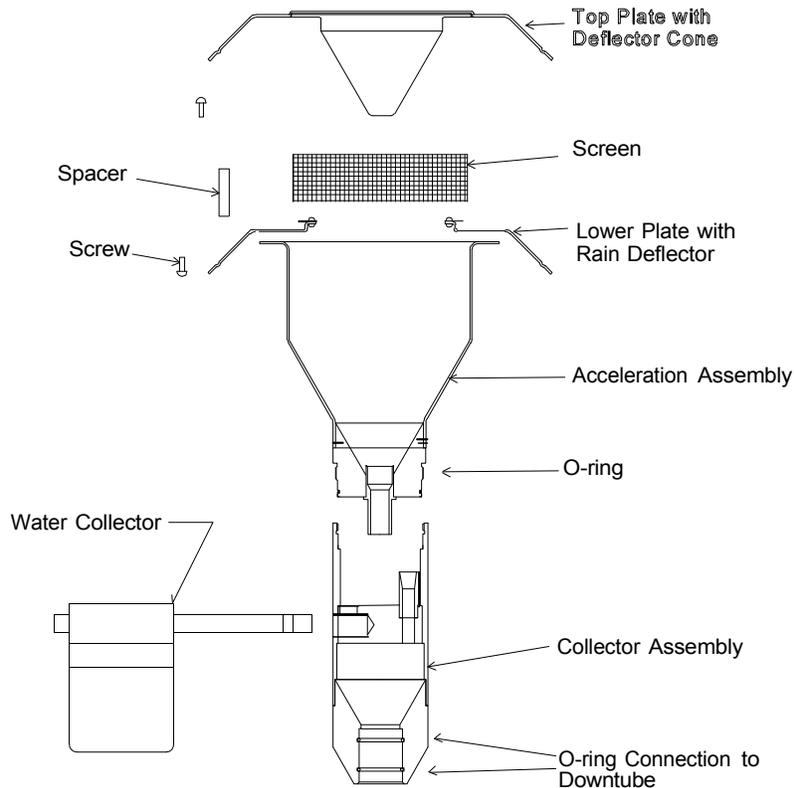
- Mark each assembly point of the sampler inlet with a pen or pencil to provide “match marks” during reassembly. Critical assembly points are already “keyed.”
- Disassemble the sample inlet unit according to Figure 9-2, taking care to retain all the parts. **Note:** If the assembly screws appear frozen, the application of penetrating oil or commercial lubricant will make removal easier.
- Using a soft brush, cloth, and cotton swabs, lightly scrub all interior surfaces and the bug screen with distilled water and/or the general-purpose cleaner. Pay particular attention to small openings and crevices. Cotton swabs and/or a small soft brush are most helpful in these areas. Using laboratory tissue and cotton swabs moistened with distilled water, wipe all surfaces to remove any remaining deposits. Completely dry all components.
- Check all the O-rings for distortion, cracks, fraying, lack of lubricating grease, or other problems. Apply a thin coating of vacuum grease or replace the O-rings as necessary.
- Reassemble the unit in accordance with the previously scribed match marks. Take particular care to ensure that all O-ring seals are properly sealed and that all screws are uniformly tightened.



**Figure 9-1. Exploded cross-sectional view of design of a  $PM_{2.5}$  impactor well and filter holder.**

### 9.3.2. Impactor Housing and Well

Figure 9-1 shows a typical disassembled impactor unit. With the impactor assembly open, inspect the interior of the impactor housing, both above and below the impactor well. These areas should



**Figure 9-2. Exploded cross-sectional view of a  $PM_{10}$  sampler inlet head.**

be clean and dry. If necessary, clean the areas with a lint-free wipe. On a monthly basis, clean the interior of the impactor jet using a lint-free pipe cleaner or similar tool.

Check the O-rings for distortion, cracks, fraying, lack of a light coating of vacuum grease, or other problems and replace as necessary.

### 9.3.3 Other

Wipe down the interior of the sampler's case to remove bugs, dirt, and/or water deposits that may have collected inside the case. This procedure may be required more frequently during summer months. Inspect the cooling air intake filter and clean it if necessary.

Check the sampler's internal clock against a timepiece known to be accurate within 1 min/month.

Record the value and note whether the sampler's clock has gained or lost time since the previous month's time check.

Check the filter cassettes and the backing screens for fractures, cracks, buckling, evidence of wear, or contamination. Clean or replace as necessary.

Conduct leak test after reassembly.

#### **9.4 Quarterly Maintenance**

The following activities should be performed once every 3 months at the time of the regular monthly maintenance.

##### **9.4.1 Inlet and Downtube**

Inspect the O-rings in the aerosol inlet and condition them with a very light coating of vacuum grease. This will inhibit breakdown and fraying of the O-rings caused by friction on the inlet tube.

Inspect the outer and inner surfaces of the tip (closest to the sampler inlet) of the downtube, and remove any particulate deposits using isopropyl rubbing alcohol or water and a soft bristle brush or wads of lint-free laboratory wipes or cloth. Dry the downtube completely before reinstallation.

Inspect the rubber water seal gasket (located at the point where the downtube enters the sampling case) for cracks or other evidence of leakage. Lubricate or replace the gasket as necessary, according to the sampler's operating manual.

##### **9.4.2 Impactor**

Remove the O-rings in the impactor assembly and condition them with a light coating of vacuum grease.

##### **9.4.3 Other**

Examine the vacuum tubing for crimps, cracks, breaks, water, or dirt and replace as necessary.

Examine the tubing fittings to ensure proper seating and tightness.

Clean or replace the cooling air intake filter and fans as required.

#### **9.5 Other Periodic Maintenance**

As recommended by the manufacturer's sampler operating instruction manual.

#### **9.6 Refurbishing PM<sub>2.5</sub> Samplers**

PM<sub>2.5</sub> samplers that have been operated in the field for extended periods may require major repairs or complete refurbishment. In these cases, the manufacturer's manual must be referred to before work is undertaken. A sampler that has been subject to major repairs or refurbishment must be leak-checked and calibrated prior to sample collection. Such a sampler should be treated as if it were newly received.

## 10.0 Performance Evaluation Procedures

### 10.1 Overview

This section summarizes the performance evaluation (PE) procedures for assessing measurement uncertainty, precision, and bias for reference or class I Equivalent methods for PM<sub>2.5</sub> sampling at State and local air monitoring stations (SLAMS) sites. These requirements are specified in 40 *Code of Federal Regulations* (CFR) Part 58, Appendix A (EPA 1997). This section also gives suggestions for components of performance and systems audits of samplers, balances, and operational procedures that reporting organizations may wish to conduct in addition to those to be provided by the U.S. Environmental Protection Agency (EPA) Regional Office.

Table 10-1 consolidates the required and recommended PE components and lists the personnel

**Table 10-1. Systems and Performance Evaluation Components for Manual Method PM<sub>2.5</sub> Samplers**

| Component   | Conducted by                                   | Frequency                  | Performance goals   |
|---|--|----------------------------|---|
| 1. Sampler flow rate audit. (a) Sampler display versus audit device reading. (b) Audit device reading versus designated flow rate (required). | Reporting organization                         | Quarterly for each sampler | (a) ±4 percent<br>(b) ±5 percent                                |
| 2. Precision via permanently collocated sampler at 25 percent of sampling sites (required).   | Reporting organization                         | Every 6 days               | 10 percent coefficient of variance (CV) for total precision     |
| 3. Federal Reference Method (FRM) PE via 1 to 2 days collocation of an FRM sampler at 25 percent of sampling sites each year (required).      | Reporting organization or EPA Regional Offices | Four times per year        | ±10 percent for total bias                                      |
| 4. Balance systems and performance audits (recommended).  | Reporting organization                         | Annually                   | Meet QAPP goals; agreement within 50 µg for 200-mg audit weight |
| 5. Sampler performance audits (temperature, pressure, flow rate) (recommended, based on quality assurance project plan [QAPP]).               | Reporting organization                         | Per QAPP                   | Per QAPP  |
| 6. Systems audit (required).  | EPA Regional Offices                           | Once every 3 years         | Meet CFR and QAPP goals   |
| 7. Systems audit (recommended).   | Reporting organization or State                | Soon after network startup | Meet CFR and QAPP goals   |

responsible for conducting and reporting the results, the frequency at which the audits or checks are to be performed, and the performance goal.

For the PE components listed in Table 10-1 that are conducted by the State agency or reporting organization, it is expected that the agency or organization will include details in its QAPP and standard operating procedures (SOPs) that describe

- Conduct and reporting of the required quarterly sampler flow rate audits
- Operation and reporting of data from the required collocated samplers to estimate precision
- Any additional systems or performance evaluations it wishes to conduct.

In general, any PE or audit, whether conducted by personnel outside or within the monitoring network, should be conducted under the following guidelines:

- Without special preparation or adjustment of the system to be evaluated.
- By an individual with a thorough knowledge of the instrument or process being evaluated but not by the routine operator.
- With accurate, calibrated, National Institute of Standards and Technology (NIST)-traceable transfer standards that are completely independent of those used for routine calibration and QC flow checks (although both calibration standards and audit standards may be referenced to the same primary standard for flow rate, volume, temperature, pressure, or mass).
- With complete documentation of audit information. This documentation includes but is not limited to the types of instruments evaluated, the audit transfer standards and transfer standard traceability, the instrument model and serial numbers, the calibration information, and the collected audit data.

Overviews of the procedures for conducting performance and systems evaluations of the PM<sub>2.5</sub> collection and measurement processes are provided in these sections:

- Section 10.2, "Performance Evaluations" (sample flow rate audit, precision assessment, FRM PE, balance accuracy, and sampler operation audits)
- Section 10.3, "Systems Audits" (State or reporting organization audits, EPA Regional Office audits).

## 10.2 Performance Evaluations

This section presents performance audit procedures specific to reference or Class I equivalent PM<sub>2.5</sub> samplers that operate on the inertial impaction principle at a sample flow rate of 16.67 L/min. Procedures for auditing samplers that operate according to other principles (e.g., open-path optical monitors) are not discussed here. 40 CFR Part 58, Appendix A (EPA 1997) requires that reporting organizations assess, on a calendar quarterly basis, the flow rate accuracy of each primary (data-reporting) PM<sub>2.5</sub> sampler used in their SLAMS monitoring networks by conducting an audit of each sampler's operational flow rate.

Appendix A of 40 CFR Part 58 also requires reporting organizations to permanently collocate samplers at 25 percent of their sites to assess measurement system precision. Furthermore, the reporting organizations are required to assess total measurement system bias by making quarterly

side-by-side comparisons between the site's primary sampler  $PM_{2.5}$  concentration results and those of an FRM PE sampler that is temporarily installed adjacent to the site sampler. This operation may be implemented by the EPA Regional Office.

This section also offers guidance to States or reporting organizations that may wish to incorporate internal audits into their  $PM_{2.5}$  monitoring program. Internal performance audits are recommended for sampler flow rate, temperature, and barometric pressure. Balance audits are also suggested. Reporting organizations may elect to include an internal audit of the operational aspects of  $PM_{2.5}$  samplers as part of their overall QAPP. The person conducting these audits should be someone other than the regular site operator. It is also important that the audit devices for flow rate, temperature, and barometric pressure not be the ones used for regular site checks and calibrations. However, the audit transfer standards may be traceable to the same primary standards as the working standards.

### 10.2.1 Sampler Flow Rate Audit

The calibration relationship between the flow rate (in actual L/min) indicated by the flow rate standard and the sampler's flow rate measurement device should be established or verified in accordance with the sampler's operation or instruction manual prior to the audit; however, no adjustments may be made to the sampler's flow rate sensors or controls before the audit is performed. A suggested audit data recording form is shown in Figure 10-1. For automated data acquisition, a printout may be attached to this and subsequent audit data sheets. The example audit data sheet shown in Figure 1-1 should be styled to include additional information as necessary. In conducting the sampler flow rate audit (or any audit for that matter) the auditor should consult the manufacturer's operations manual for sampler-specific procedures, suggestions, and precautions.

The audit of the sampler's flow rate consists of one measurement made at the sampler's operational flow rate. The flow rate transfer standard used for the audit must not be the same flow rate standard used to verify or calibrate the sampler but may have been made traceable to the same primary standard for flow rate or volume. The sampler's operation or instruction manual should provide specific instructions for using the instrument's flow rate readout or display. The audit is performed by the operator or by personnel from the quality assurance (QA) unit of the reporting organization as follows:

1. Install a flow rate audit filter/cassette in the filter holder and energize the sampler. Filters used for flow rate audits should not be used further for sampling. If the network's sampling schedule is on a daily basis or if loss of a sampling day cannot be accommodated, a flow rate audit may be conducted during a sampling event provided that
  - The active sampling filter is removed and protected from contamination and damage by storing it in a clean container between uses
  - A flow rate audit filter is used for the audit
  - The active sampling period is not interrupted for more than 1 hour in order to achieve a minimum overall sampling period of 23 hours.
2. Allow the sampler to warm up for a minimum of 15 min to allow the sampler's electronics

and vacuum pump to stabilize.

3. Record the sampler's flow rate as the "sampler flow rate before audit" on an audit data sheet, such as the example given in Figure 10-1.
4. Complete the required information at the top of the sampler audit data sheet (Figure 10-1).
5. Remove the sampler inlet and install the flow rate adapter device (refer to Figure 6-2).
6. Connect the adapter to the flow rate transfer standard outlet with flexible tubing, being careful not to crimp the tubing. Refer to Figure 8-3 for an example of the setup. If the transfer standard is electronic (such as an electronic micromanometer coupled to an orifice device), allow sufficient warmup time, based on one's prior experience with the device, for readings to stabilize.
7. Recheck the flow rate indicated by the PM<sub>2.5</sub> sampler's data display or other readout device. Record this value and the corresponding flow rate indicated by the audit device.
8. Record at least three sampler and audit flow rate readings of the output data directly on the audit data sheet (Figure 10-1). If electronic recording devices are used for one or more of the parameters, be sure that the clocks are synchronized. If units other than actual flow are displayed by the audit device, the ambient temperature and barometric pressure will have to be independently determined and calculations will have to be made to express results at ambient conditions. See Section 10.2.1.1 for calculation methods. Record all results on the data sheet and average them. Also compare the audit flow rate with the designated flow rate, 16.67 L/min.
9. Calculate the average difference in flow rate in L/min and as a percentage of the audit flow rate. Record all data for eventual submission to the EPA Aerometric Information Retrieval System (AIRS) database.
10. Following the measurement with the flow rate standard, remove the adapter and reinstall the sampler inlet. From the sampler's display, read the sampler's operating flow rate again (in L/min) with the flow rate filter in place. Record this as the "sampler flow rate after audit" on the audit data sheet. Remove the flow rate audit filter. Do not use it for further sampling; it may be used several more times for flow rate audits of the same model sampler, provided it is carefully stored between uses to prevent contamination or damage. Stop using this filter when a buildup of particles on the filter surface is visible.
11. Replace the sample filter if it was removed in the midst of a sampling event. Otherwise, return the sampler to its pre-audit condition.

#### 10.2.1.1 Flow Rate Audit Data Calculations

PM<sub>2.5</sub> flow rate audit results should be reported at ambient conditions and should not be corrected to standard conditions (e.g., 25 EC, 760 mmHg). However, for some types of audit devices, it may be necessary to correct the indicated audit flow rates to ambient conditions. Equation (10-1) may be used to make this correction. The following correction should be applied only if the audit device reports flow rate already corrected to standard conditions (e.g., mass flowmeters). Be sure to verify the standard conditions of temperature and pressure ( $T_{std}$  and  $P_{std}$ , respectively) applicable to the audit device.

**PM<sub>2.5</sub> Sampler Audit Data Sheet**  
**Flow Rate, Temperature, and Pressure Audit**

Site Name. \_\_\_\_\_ Site Location. \_\_\_\_\_  
 Auditor's Name. \_\_\_\_\_ Observer's Name. \_\_\_\_\_  
 Sampler IO-2001 Sampler ID: R-102  
 Model: \_\_\_\_\_  
 Flow Rate Audit Device Manufacturer, Model, Serial Number: \_\_\_\_\_ Orifice device, S/N 104,  
 coupled to electronic digital micromanometer  
 Temperature Audit Device Manufacturer, Model, Serial Number: \_\_\_\_\_ Thermistor  
 temperature probe, S/N T01562-53  
 Pressure Audit Device Manufacturer, Model, Serial Number: \_\_\_\_\_ Hand-held aneroid barometer,  
 S/N 420292

---

**Flow Rate Audit:**

Date of Test: 8/13/97

Sampler flow rate before audit 16.65 L/min Sampler flow rate after audit 16.72 L/min

| Sampler's Indicated Flow Rate, L/min | Audit Flow Rate, L/min | Difference (Sampler - Audit), L/min | Difference ÷ Audit Flow, % |
|--------------------------------------|------------------------|-------------------------------------|----------------------------|
| 16.72                                | 16.59                  |                                     |                            |
| 16.65                                | 16.51                  |                                     |                            |
| 16.74                                | 16.61                  |                                     |                            |
| 16.70 Avg.                           | 16.57 Avg.             | 0.13                                | 0.8%                       |

---

**Temperature Audit:**

Date of Test: 8/13/97

| Sampler's Indicated T <sub>a</sub> , EC |           | Audit T <sub>a</sub> , EC |           | Difference (Sampler - Audit), EC |        |
|---|-----------|---------------------------|-----------|----------------------------------|--------|
| Inlet                                   | Filter    | Inlet                     | Filter    | Inlet                            | Filter |
| 33.7                                    | 34.5      | 33.1                      | 34.7      |                                  |        |
| 33.8                                    | 34.6      | 33.2                      | 34.8      |                                  |        |
| 33.7                                    | 34.5      | 33.0                      | 34.7      |                                  |        |
| 33.7 Avg.                               | 34.5 Avg. | 33.1 Avg.                 | 34.7 Avg. | 0.6                              | -0.2   |

---

**Pressure Audit:**

Date of Test: 8/13/97

| Sampler's Indicated P <sub>a</sub> , mmHg | Audit P <sub>a</sub> , mmHg | Difference (Sampler - Audit), mmHg |
|---|-----------------------------|------------------------------------|
| 759                                       | 760                         |                                    |
| 761                                       | 760                         |                                    |
| 758                                       | 759                         |                                    |
| 759 Avg.                                  | 760 Avg.                    | -1                                 |

Notes: Attach additional sheets, if necessary.  
 Show calculations on attached sheets.

Figure 10-1. Example PM<sub>2.5</sub> Flow rate, temperature, and pressure audit form.

$$Q_a = Q_{std} (T_a/P_a) (P_{std}/T_{std}) \quad (10-1)$$

where

- $Q_a$  = flow rate at actual conditions, L/min  
 $Q_{std}$  = flow rate reported at standard temperature and pressure (e.g., 25 EC, 298 K; 760 mmHg or 101 kPa), L/min  
 $T_a$  = ambient temperature, K  
 $P_a$  = ambient barometric pressure, mmHg or kPa  
 $P_{std}$ ,  $T_{std}$  = standard barometric pressure and temperature, respectively.

Determine the percentage difference between the sampler-indicated flow rates and the audit-measured flow rates using Equation (10-2). Record the result on the audit data sheet. Any deviation greater than  $\pm 2$  percent may require recalibration.

$$\% \text{ difference} = 100 \times (Q_a(\text{sampler}) - Q_a(\text{audit})) / Q_a(\text{audit}) \quad (10-2)$$

### 10.2.2 Temperature Audit Procedures

Ambient temperature, filter temperature, and ambient pressure sensors on the PM<sub>2.5</sub> sampler may be audited in conjunction with the flow rate audit. Make no adjustments to the sampler prior to recording all audit results. Record the audit data on a form similar to the one shown in Figure 10-1.

The recommended procedure for auditing temperature probes is to use a bath of water, oil, or other suitable liquid to provide a stable temperature. A container such as a thermos bottle or Dewar flask should be used to insulate the bath, minimizing heat transfer to and from the fluid in the flask. Temperature sensors and thermometers should never be used to stir the bath. If water is used for the bath, be sure that evaporative cooling does not cause the temperature in the bath to fall during the audit process. A minimum of three temperatures should be used for the audit. Details of temperature calibration procedures are found in Section 6.0, "Calibration Procedures," of this document and can be adapted for audit purposes. Record the stabilized temperature reading and report the results on a data sheet such as the one shown in Figure 10-1. Any deviation greater than 2 EC should be reported for corrective action.

### 10.2.3 Barometric Pressure Audit Procedures

Ambient pressure is audited using a suitably calibrated sensor at ambient conditions. Refer to Section 6.0 of this document for information on establishing traceability and the use of portable barometers. Record all findings on an audit data form or in a bound notebook. Any deviation greater than 10 mmHg should be reported for corrective action.

### 10.2.4 Assessment of Precision Using Collocated Samplers

Collocated sampler results, where the duplicate sampler is not necessarily a reference method sampler but a sampler of the same designated method as the primary sampler, are used to assess measurement system precision according to the schedule and procedure specified in 40 CFR Part 58, Appendix A, Section 3.5.2. The regulations require that 25 percent of a reporting organization's primary samplers have another sampler (termed the duplicate sampler) permanently collocated at the same site and collect a sample on at least an every sixth day schedule so that the

primary and duplicate samplers capture samples from all days of the week throughout the year. The site locations for the duplicate samplers are decided by choosing those sites that are most likely to have higher concentrations of  $PM_{2.5}$  or that have been found to be in violation of the National Ambient Air Quality Standards (NAAQS) during initial studies. Refer to 40 CFR Part 58, Appendix A, Section 3.5.2 for details. The collocated sampler measurements will be used by EPA to calculate quarterly and annual precision estimates for each primary sampler and for each designated method employed by each reporting organization. A data quality objective (DQO) of 10 percent CV or better has been established for the operational precision of  $PM_{2.5}$  monitoring data.

#### 10.2.4.1 Procedure for Collocated Measurements

The collocated sampler must be kept clean, serviced, and inspected frequently for proper operation, and the calibration of its flow rate, temperature, and pressure measurement systems must be checked and verified at the same intervals as the primary sampler.

The station's primary site collector and the duplicate sampler must be located at least 1 m but no more than 4 m apart, and their inlets should be at the same height above ground and within 1 m of each other as measured in the vertical direction. Space either sampler at least 2 m away from a high-volume sampler. The samplers must be calibrated and operated according to their operations manuals, and the samples must be stored, shipped, and analyzed identically, as described in the sampler's operation or instruction manual and in general accordance with the guidance in this handbook and in the reporting organization's QAPP and standards of procedure. The primary sampler must be operated during the comparison period as closely as possible to the way it operates during normal sampling or monitoring. Collocated sampling should be conducted during a regularly scheduled sampling period whenever possible.

The concentrations determined from the site's primary sampler and from the collocated, duplicate sampler are to be reported to EPA as directed in 40 CFR Part 58, Appendix A, Section 4. **Note:** All collocated measurements must be reported, even those that might be considered invalid because of identified malfunctions or other problems that occurred during or following the sample collection period. Although all measurements must be reported, only those sample pairs having  $PM_{2.5}$  concentrations above  $6 \mu\text{g}/\text{m}^3$  will be selected for use in precision calculations because at low concentrations agreement between the measurements of collocated samplers may be relatively poor.

#### 10.2.5 Federal Reference Method Performance Evaluation

Because the size and volatility of the particles making up ambient particulate matter vary over a wide range and the mass concentration of particles varies with particle size, it is difficult to define the accuracy of  $PM_{2.5}$  measurements in an absolute sense. The accuracy of  $PM_{2.5}$  measurements is therefore defined in a relative sense, referenced to measurements provided by the FRM. Accuracy is defined as the degree of agreement between a subject field  $PM_{2.5}$  sampler (the primary sampler) and a collocated FRM PE sampler operating simultaneously at the monitoring site and totally independent of the site operations with respect to operator, sampler, sample handling, and weight determination. Audit results include both random (precision) and systematic (bias) errors.

The EPA Regional Offices are responsible for conducting the FRM PEs to assess total measurement system bias. The reporting organization is responsible for assisting FRM PE personnel by coordinating schedules and assisting in the setup of the FRM sampler, which will be on site for approximately 2 days.

Twenty-five percent of SLAMS PM<sub>2.5</sub> primary samplers within each reporting organization will be assessed with an FRM PE every calendar year so that in a 4-year span all FRM or Federal Equivalent Method (FEM) samplers will have been audited. Further, every designated FRM or FEM within a reporting organization must

- Have at least 25 percent of each method designation audited, including those at collocated sites (even those collocated with FRM samplers used in the precision assessment program)
- Have at least one sampler audited
- Be audited at a frequency of four audits per year.

Details of the schedule for performing collocated measurements with the FRM sampler and guidelines for determining the initial deployment of FRM PE samplers based on sites likely to be in violation of the NAAQS are provided in 40 CFR Part 58, Appendix A, Section 3.5.3. The results from the primary sampler and the duplicate FRM sampler are used by EPA to calculate the accuracy of the primary sampler on a quarterly basis, the bias of the primary sampler on an annual basis, and the bias of a single reporting organization on an annual basis.

#### **10.2.6 Balance Accuracy Assessment**

Each State or reporting organization should conduct an internal accuracy assessment of each microbalance on an annual basis. A performance audit of the microbalances used to weigh PM<sub>2.5</sub> filters requires the use of an independent set of American Society for Testing and Materials (ASTM) Class I standard weights traceable to NIST with a tolerance of no more than 0.010 mg. Weights of 100 and 200 mg are suggested. These weights must not be the same ones used as working standards for the day-to-day operation of the microbalance but may be traceable to the same primary standard used to trace the working standards. Because microbalances are extremely delicate instruments and should not be operated by inexperienced personnel, it is recommended that the PE of the filter-weighing process be done in cooperation with the laboratory personnel. The person normally performing the weighings for PM<sub>2.5</sub> monitoring should assist the auditor by preparing the balance as if a series of filter weighings were to be done. Record all data on data sheets or in the laboratory's balance notebook. The balance display should agree with the designated value of the audit weight to within  $\pm 0.020$  mg (twice the tolerance for an ASTM Class 1 weight).

Many laboratories maintain an agreement with a service representative to conduct regular servicing of the balances. It may be instructive to conduct a performance audit prior to the periodic servicing and again immediately after the servicing.

### **10.3 Systems Audits**

Periodic systems audits of PM<sub>2.5</sub> programs are strongly recommended. Such audits may be particularly important at the startup of a new monitoring effort and can help recognize and

pinpoint problems before significant parts of data are affected. Loss of data could cause a reporting organization's airshed to fall into a nonattainment classification. The EPA Regional Offices are charged with conducting a systems audit once every 3 years. These audits are designed to assess the entire measurement system and data management activities with regard to PM<sub>2.5</sub> monitoring networks. These activities would include the following:

- Initial equilibration, weighing, and transportation of the filters to the sampler
- Site selection criteria assessment
- Equipment installation
- Site security
- Equipment maintenance
- Calibration procedures
- Handling and placement of the filters
- Operation of the sampler and sample collection
- Removal, handling, and transportation of the filter from the sampler to the laboratory
- Weighing, storage, and archival of the sampled filter
- Data analysis and reporting.

EPA specifies that QA and QC programs follow the requirements for QAPPs contained in the EPA document *Requirements for Quality Assurance Project Plans for Environmental Data Operations*, EPA QA/R-5 (EPA 1994). Auditors should develop a checklist based on these guidelines and on the specific information regarding PM<sub>2.5</sub> monitoring in this QA handbook.

Effective system auditing requires the auditor to observe the auditee's entire sample acquisition and analysis system and the associated data processing operations. These location include the sampling site, laboratory, the shipping and receiving area where field samples are received and logged in, the sample archival area, and the data processing areas.

Data about specific samples obtained during on-site performance audits may be useful during the systems audit. Tracking specific samples and the associated data completely through the system (sample acquisition, shipping, receiving, logging in, weighing, archiving, data processing, and reporting) ensures that every significant operation is examined. This approach also allows the auditor to look at the "interfaces" between different operations—the interactions between field and laboratory tasks—and not just the individual tasks in isolation. Detailed and summary reports and data submitted to EPA databases (e.g., AIRS) for the audited period should be reviewed as a follow-up to the audit, if the timing of these reports permits.

Effective preparation for a systems audit requires developing a checklist similar to the one shown in Figure 10-2. Auditors are encouraged to develop their own checklists based on their experience and as dictated by the individual audit situation.

| <b>Checklist for PM<sub>2.5</sub> Auditing</b>  |   |                        |     |         |
|---|---|------------------------|-----|---------|
| Site or Laboratory Location _____   |   | Site Designation _____ |     |         |
| Auditor Name and Affiliation _____  |   |                        |     |         |
| Observer(s) Name and Affiliation _____  |   |                        |     |         |
| Audit Question  | Y | N                      | N/A | Comment |
| <b>On Site:</b>   |   |                        |     |         |
| • Are logbooks and required data sheets filled in promptly, clearly, and completely?  |   |                        |     |         |
| • Are filters handled with the necessary care and finesse to avoid contamination and/or loss of material? Does the operator keep the filter-handling area neat and clean?   |   |                        |     |         |
| • Is the equipment sited properly? Are there any changes at the site that might compromise original siting criteria (e.g., fast-growing trees or shrubs, new construction)? |   |                        |     |         |
| • Does the equipment appear to be well maintained and free of dirt and debris, bird/animal/insect nests, excessive rust and corrosion, etc.?                                |   |                        |     |         |
| • Are the walkways to the station and equipment kept free of tall grass, weeds, and debris?   |   |                        |     |         |
| • Is the station shelter (if any) clean and in good repair?   |   |                        |     |         |
| <b>Sampler Calibration and Maintenance:</b>   |   |                        |     |         |
| • Does the flow rate standard used for routine flow rate calibration have its own certification traceable to a NIST primary standard for volume of flow rate?               |   |                        |     |         |

(continued)

Figure 10-2. Example systems audit questionnaire.

| Audit Question  | Y   | N   | N/A | Comment |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
|---|---|-----|-----|---------|-------------------|-------|-----|----------------------|----------------------|--|-------------------|---|--|-------------------|------------------------------|--|--------------------|-------------------------|--|
| <b>Sampler Calibration and Maintenance (continued):</b>   |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Is the calibration relationship for the flow rate standard (e.g., an equation, curve, or family of curves relating actual flow rate [Q<sub>a</sub>] to the flow rate indicator reading) accurate to within 2 percent over the expected range of ambient temperatures and pressures at which the flow rate standard may be used? Verify by inspecting the original calibration data and calculating the error in the calibration relationship for each calibration point used to generate the relationship.</li> </ul>  |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Is the flow rate standard used for routine sampler calibration/verification recalibrated or reverified against a NIST-traceable standard at least annually?</li> </ul>   |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <b>Balance Maintenance and Weighing Procedures:</b>   |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Is the filter preparation and weighing area neat and clean?</li> </ul>   |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Are logbooks kept up to date and properly filled in?</li> </ul>  |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Is the balance on a service agreement for regular professional maintenance? If not, is there someone in the organization with the appropriate training to service the balance?</li> </ul>  |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Does the analytical balance used to weight filters have a readability of ±1 µg?</li> </ul>   |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Is there a formal logbook or file for balance maintenance? Are entries current?</li> </ul>   |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Are regular (e.g., daily, when in use) calibration checks made and recorded?</li> </ul>  |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Are filters conditioned immediately before both the pre- and postsampling weighings? The following conditions are specified in 40 CFR Part 58, Appendix L:</li> </ul>  |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <table border="0"> <tr> <td>Mean temperature:</td> <td>20–23</td> <td>EC.</td> </tr> <tr> <td>Temperature control:</td> <td colspan="2">±2 EC over 24 hours.</td> </tr> <tr> <td>Average humidity:</td> <td colspan="2">30 to 40 percent relative humidity (RH); however, where it can be shown that the mean ambient RH during sampling is between 20 and 30 percent, conditioning is permissible at a mean RH within ±5 percent of the mean ambient RH during sampling.</td> </tr> <tr> <td>Humidity control:</td> <td colspan="2">±5 percent RH over 24 hours.</td> </tr> <tr> <td>Conditioning time:</td> <td colspan="2">Not less than 24 hours.</td> </tr> </table> |   |     |     |         | Mean temperature: | 20–23 | EC. | Temperature control: | ±2 EC over 24 hours. |  | Average humidity: | 30 to 40 percent relative humidity (RH); however, where it can be shown that the mean ambient RH during sampling is between 20 and 30 percent, conditioning is permissible at a mean RH within ±5 percent of the mean ambient RH during sampling. |  | Humidity control: | ±5 percent RH over 24 hours. |  | Conditioning time: | Not less than 24 hours. |  |
| Mean temperature:   | 20–23   | EC. |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| Temperature control:  | ±2 EC over 24 hours.  |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| Average humidity:   | 30 to 40 percent relative humidity (RH); however, where it can be shown that the mean ambient RH during sampling is between 20 and 30 percent, conditioning is permissible at a mean RH within ±5 percent of the mean ambient RH during sampling. |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| Humidity control:   | ±5 percent RH over 24 hours.  |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| Conditioning time:  | Not less than 24 hours.   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |
| <ul style="list-style-type: none"> <li>Are new filters placed in the conditioning environment immediately upon arrival and stored there until the presampling weighing?</li> </ul>  |   |     |     |         |                   |       |     |                      |                      |  |                   |   |  |                   |                              |  |                    |                         |  |

(continued)

Figure 10-2. (continued)

| Audit Question  | Y | N | N/A | Comment |
|---|---|---|-----|---------|
| <b>Balance Maintenance and Weighing Procedures (continued):</b>   |   |   |     |         |
| • Is the analytical balance located in the same controlled environment in which the filters are conditioned?  |   |   |     |         |
| • Are the filters weighed immediately following the conditioning period without intermediate or transient exposure to other conditions or environments?   |   |   |     |         |
| • Are filters conditioned at the same environmental conditions before both the pre- and postsampling weighings?   |   |   |     |         |
| • Are both the pre- and postsampling weighings performed on the same analytical balance, using an effective technique to neutralize static charges on the filter?   |   |   |     |         |
| • Are both weighings performed by the same analyst? (If not, have results of the different analysts been compared statistically?)   |   |   |     |         |
| • Are presampling weighings done within 30 days of the sampling periods in which filters are to be exposed? (Review logbook to look for evidence of filters that exceed their time span.)   |   |   |     |         |
| • If filters are stored at ambient temperature, are the post-sampling conditions and weighings completed within 240 hours (10 days) after the end of the sample period?   |   |   |     |         |
| • If filters are stored at 4 EC or below during the entire time between retrieval from the sampler and start of conditioning, are the postsampling conditioning and weighings completed within 30 days after the end of the sample period?  |   |   |     |         |
| • Are new field blank filters weighed along with the pre-sampling (tare) weighing of each lot of PM <sub>2.5</sub> filters?   |   |   |     |         |
| • Are QC field blank filters routinely used, observing the following handling steps: transport to the sampling site, installation in the sampler, retrieval from the sampler (without sampling), and reweighing?  |   |   |     |         |
| • Are QC laboratory blank filters weighed along with the pre-sampling (tare) weighing of each set of PM <sub>2.5</sub> filters and reweighed when the exposed filters are received from the field? (These laboratory blank filters should remain in the laboratory in protective containers during the field sampling and should be reweighed as a QC check.) |   |   |     |         |
| • Was the balance calibrated as specified by the manufacturer at installation and recalibrated immediately prior to each weighing session?  |   |   |     |         |

(continued)

Figure 10-2. (continued)

| Audit Question   | Y | N | N/A | Comment |
|--|---|---|-----|---------|
| <b>Recordkeeping and Calculations:</b>   |   |   |     |         |
| • Are logs and/or charts of the balance room temperature and humidity on file?                           |   |   |     |         |
| • Are records of shipments (incoming and outgoing) maintained?   |   |   |     |         |
| • Are records of sample filter condition upon arrival at laboratory kept (i.e., temperature)?            |   |   |     |         |
| • Are data management files in order?  |   |   |     |         |
| • Is there evidence that data validation, internal QA review, and complete data reporting have occurred? |   |   |     |         |
| • Is the personnel management structure sound?   |   |   |     |         |

Figure 10-2. (continued)

## 11.0 Calculations, Validations, and Reporting of PM<sub>2.5</sub> Monitoring Data

### 11.1 Overview

This section presents the calculations required to compute and report ambient PM<sub>2.5</sub> concentrations. A summary of all calculation formulas is given in Table 11-1. The end result of these calculations is the integrated (average) PM<sub>2.5</sub> mass concentration during the sampling period, expressed in units of µg/m<sup>3</sup>. To obtain this concentration, it is necessary to determine the mass of particles collected by the filter and the total volume of air that was sampled. Unlike many other pollutant measurements, the volumes used to calculate the PM<sub>2.5</sub> concentration **must** always be expressed at the **actual** ambient temperature and pressure used to acquire the sample (40 CFR Part 50.3, as amended 6/28/97). This process represents a change from the convention used to report PM measurements prior to June 1997, in which volumes were adjusted to “U.S. EPA-standard” conditions, defined as 25 EC (298 K) and 760 mmHg (101 kPa).

Accurate measurement and control of the sampler’s flow rate are needed to determine the total sampled volume and to ensure that the particles trapped by the filter are in the correct particle size range. Reference samplers and Class I equivalent samplers separate PM<sub>2.5</sub> particles by inertia. Obtaining the correct particle size range by inertial separation requires maintaining the correct air velocity in the sampler’s size separator system (impactor). This velocity is correct when the specified design flow rate is maintained.

### 11.2 Calculations

#### 11.2.1 Sample Volume Calculations

Both reference and equivalent method samplers are required to provide measurements of the total volume of air sampled ( $V_a$ ), in m<sup>3</sup> at the actual ambient temperatures and pressures during sampling (40 CFR Part 50, Appendix L, paragraph 7.4.5.2). If the sampler’s flow measurement system is properly calibrated,  $V_a$  should be accurate, and no further sample volume calculations are required.

**Table 11-1. Formulas for Calculations Associated with PM<sub>2.5</sub> Monitoring**

| Calculation   | Formula                              | Equation No. | Section |
|---|--------------------------------------|--------------|---------|
| Alternative calculation of total sample volume, used only if $V_a$ is not available directly from the sampler | $V_a = Q_{avg} \times t \times 10^3$ | 11-1         | 11.2.1  |
| Determination of PM <sub>2.5</sub> mass collected on filter (net weight)                                      | $M_{2.5} = (M_f - M_i) \times 10^3$  | 11-2         | 11.2.2  |
| Determination of PM <sub>2.5</sub> mass concentration   | $PM_{2.5} = M_{2.5}/V_a$             | 11-3         | 11.2.3  |

**Note:** In the event the total sample volume measurement from the sampler is not available, the total sample volume may be calculated by multiplying the average flow rate, in actual m<sup>3</sup>/min, by the elapsed sample collection time in minutes. Both of these measurements are required to be provided by reference and equivalent method samplers. Use the following formula only if  $V_a$  is not available directly from the sampler:

$$V_a = Q_{avg} \times t \times 10^{13} \quad (11-1)$$

where

$V_a$  = total sample volume, actual m<sup>3</sup>  
 $Q_{avg}$  = average sample flow rate over the sample collection period, L/min  
 $t$  = total elapsed sample collection time, min  
 $10^{13}$  = units conversion (m<sup>3</sup>/L).

For example, a sampler with an average flow rate of 16.7 L/min ( $Q_{avg}$ ) for a 1,410-min (23.5-hour) sampling period ( $t$ ) would have a total sample volume ( $V_a$ ) of 23.5 m<sup>3</sup>.

### 11.2.2 Net PM<sub>2.5</sub> Mass Calculation

The mass of particulate matter collected on the filter during the sampling period is determined by subtracting the initial (tare) mass of each filter from the final mass of the filter, as

$$M_{2.5} = (M_f - M_i) \times 10^3 \quad (11-2)$$

where

$M_{2.5}$  = total mass of PM<sub>2.5</sub> collected during the sampling period, μg  
 $M_f$  = final mass of the equilibrated filter after sample collection, mg  
 $M_i$  = initial (tare) mass of the equilibrated filter before sample collection, mg  
 $10^3$  = units conversion (μg/mg).

For example, a filter that weighed 139.293 mg before sampling ( $M_i$ ) and 139.727 mg after sampling ( $M_f$ ) would have a PM<sub>2.5</sub> mass ( $M_{2.5}$ ) of 434 μg.

### 11.2.3 PM<sub>2.5</sub> Concentration Calculation

Each PM<sub>2.5</sub> mass concentration measurement is calculated by dividing the total mass of PM<sub>2.5</sub> (Equation 11-2) collected during the sampling period ( $M_{2.5}$ ) by the total volume of air sampled ( $V_a$ ) (taken directly from the sampler readout display or calculated from Equation 11-1), as

$$PM_{2.5} = M_{2.5} / V_a \quad (11-3)$$

For example, a sample with a mass ( $M_{2.5}$ ) of 434 μg collected from a total sample volume ( $V_a$ ) of 23.5 m<sup>3</sup> corresponds to a PM<sub>2.5</sub> concentration ( $PM_{2.5}$ ) of 18.5 μg/m<sup>3</sup>.

## 11.3 Verification of Manual Calculations and Data Entry

This section applies to calculations performed using manually recorded or transcribed data and when hand calculators, computerized spreadsheets, or other calculation aids are used by field

operators or weighing technicians. This section also describes verification procedures for data keyed from handwritten forms into a computer.

### 11.3.1 Verification of Manual Calculations

Calculations should be verified when new sites are set up, when new personnel are trained, and when calculation aids such as spreadsheets are changed or updated. These calculations should be reverified periodically as part of the regular audit program. The basic procedure for verifying manual calculations is as follows:

- Gather the raw data sources to be checked. These might include the analyst's notebook or the field operator's report sheets on which the data were originally recorded.
- Obtain a copy of the resulting data report. The report should represent the data after they have been entered into the monitoring agency's electronic data system. This will ensure that all sources of human error, including keying errors, are included in the check.
- Independently verify the results based on the raw data. If calculation aids were used, do not use the same spreadsheet or calculator program that was originally used; the verification calculations should be done as independently as possible.
- Verify that the correct formulas, conversion constants, and reporting units were used.

The amount of data to be recalculated and verified depends on several factors. If the original calculations are suspect (for example, where mistakes have been found in a certain individual's work), all the questioned results should be recalculated. When there is no reason to suspect widespread errors, as with recalculations done as part of a routine audit, only a percentage of the data set need be checked initially. A commonly used audit guideline is to check 7 percent of manual calculations, provided that at least one example of each type of calculation is checked. This is a flexible guideline that depends on the amount of data to be checked and other factors. A cross-section of data should be selected for checking (e.g., the first and last values in a data set, extreme values, atypical values). The monitoring agency's Quality Assurance Project Plan (QAPP) should describe the frequency of these verifications, procedures to be followed, acceptance criteria, and corrective actions.

### 11.3.2 Verification of Manual Data Entry

Two methods are typically used for keystroke data entry quality control (QC): duplicate keying and proofing. Duplicate keying is defined as data entry by two different operators, followed by resolution of all discrepancies by referring back to the original forms. Proofing refers to manual (visual) comparison of data entered by a single operator against the original forms. The person who proofs the data should not be the person who entered it originally. Duplicate keying is far more likely to eliminate simple keystroke mistakes than proofing. It is also more cost-effective for large data sets but requires somewhat more work to set up initially.

If a data set has not been entered by the duplicate keying technique, a certain percentage of residual errors is to be expected. Keystroke data entry error rates can be estimated by comparing the original report pages or logbooks to the data reported out of the data system. The end use of the data should be considered in determining whether the error rate is unacceptably high. For

example, if the data will be used for purposes of compliance or litigation, any avoidable errors may be considered unacceptable. In such a case, the data should be rekeyed as described above.

If a data set has been entered by an effective duplicate keying procedure, simple keystroke errors are expected to be virtually nonexistent (less than 1 in 1,000,000, even with key operators of modest ability). If a check of duplicate-keyed data turns up a significant error rate, the integrity of the duplicate keying procedure should be investigated by examining the Standard Operating Procedure (SOP) for deficiencies. If no procedural problems are found, noncompliance with the SOP or hardware problems should be investigated.

In addition to typographical errors, other potential sources of data entry errors should also be considered while performing an audit of data entry. These include poor handwriting on the original data forms and smears or water damage that renders the writing difficult to read. Hardcopy records must be maintained for a period of time so that audits and verification checks can be performed. The monitoring agency's QAPP should describe the policies regarding verification of manual data entry, audit frequency, and retention policies for original records needed to verify the electronic data.

## 11.4 Validation

### 11.4.1 Data Validation

Validation of monitoring data is an extensive topic that can only be touched on in this section. The general principles are presented in Volume I of this series. Validation data screening is done for two primary reasons:

- To verify that the data have been recorded, entered, and calculated correctly—Validation screening can sometimes be used to identify problems such as failing equipment, siting problems, or operator errors.
- To screen for potential outliers (data points that are exceptional in concentration value or in some other way)—Data points identified as potential outliers are not necessarily invalid; validation screening simply identifies data points for further investigation.

**For example, if the field sampled filter transport temperature is between 4 and 25 EC, but is below the maximum temperature that was recorded during the filter sampling period, consider that this condition was never intended, by the authors of the method, to, necessarily, and by itself, to be a cause for invalidating the resulting PM<sub>2.5</sub> concentrations.** The appropriate decision maker should take into the decision all other QC and QA data relevant to the decision.

Data validation can be done in several different ways. A combination of techniques is often the most effective. The exact criteria to be applied and corrective actions taken should be described in the agency's QAPP. Some techniques that may be applicable to PM<sub>2.5</sub> data validation are the following:

- Graphing and visually examining time series of operating parameter data such as flow checks (e.g., control charting)
- Graphing and visually examining scatter plots of data (e.g., duplicate sample results)

- Range checking (does the data fall within an expected range of values?)
- Statistical checking (flagging data points outside 3-sigma or other statistically derived limits)
- Evaluation of goodness of fit and linearity (applicable to linear regression data such as calibrations)
- Regular review of operators' notes and communication with operators to identify problems
- Review of audit results to identify data potentially impacted by audit findings.

Being flagged as a potential outlier by the methods listed above is **not** *prima facie* evidence that a data point is invalid. Outliers must be thoroughly investigated before they can be excluded. Outliers must be presumed to be correct unless there is documented evidence of an equipment malfunction or other exceptional condition that renders the result unrepresentative. Special flags are available in the Aerometric Information Retrieval System (AIRS) indicating when data are suspect.

Identification and investigation of outliers is particularly important with PM<sub>2.5</sub> monitoring because the 24-hour compliance criterion is based on the upper 98th percentile point of the concentration distribution. Because operational problems can sometimes cause anomalously high readings, potential outliers in the upper tail of the concentration distribution should be validated carefully.

A monitoring agency should provide a detailed description of its data screening procedures in its QAPP or in an approved data validation SOP. Decision rules for data invalidation and for taking corrective actions should be clearly described. Corresponding AIRS data flags should be specified in the agency's written procedure.

#### 11.4.2 Validation of Software Used to Process PM<sub>2.5</sub> Data

Software used to process, manage, and report PM<sub>2.5</sub> data used for compliance purposes must be validated to ensure that it is free of incorrectly coded calculations and errors. The process of validating software is distinct from data validation discussed in the previous section.

A structured approach to software development, testing, and validation is strongly recommended for managing PM<sub>2.5</sub> compliance data. Information on structured software development, testing and validation can be found in EPA QA/R-5, *EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, Appendix G*, and in many other publications.

A few of the areas that should be tested during the development of PM<sub>2.5</sub> compliance and reporting software include the following:

- Correctness of calculations
- Correct assignment of input and output values (i.e., verify that the input and output values are correctly identified as to station, sample time period, sampler number, filter number, and so on)
- Correct calculation of statistics, including determination of the correct 98th percentile point for the 24-hour compliance criterion (40 CFR Part 50, Appendix N, Section 2.6).

- Correct operation across the turn of the century (year 2000 problem)
- Correct application of validation procedures, range checks, statistical control limits, and so on.

Use of spreadsheets for processing and managing large environmental data sets is strongly discouraged. Spreadsheets are difficult to test thoroughly and can develop new problems as more data are added and predefined data ranges overflow. Modern relational database products offer a much more reliable environment for large-scale data processing, although they are somewhat more difficult to learn at first.

Although software does not usually “break” or wear out after it has been written and tested, any change in its runtime environment may potentially cause problems. Therefore, periodic reverification of software is advisable, especially after significant changes to system hardware or software. Whenever individual software modules are changed, run test procedures to detect any unintended consequences of the changes. Software documentation that describes configuration changes and test results should be maintained.

All monitoring agencies are strongly encouraged to develop and comply with SOPs for software development, testing, maintenance, configuration control, documentation, and data archiving. The QAPP should identify the relevant data processing SOPs, and these should be addressed during periodic audits.

## 11.5 Data Reporting

The primary standards for particulate matter in ambient air are based on the measured mass concentration of PM<sub>2.5</sub>. Information on reporting and interpretation of PM<sub>2.5</sub> data with respect to the attainment of these standards is covered in 40 CFR Part 50, Appendix N.

### 11.5.1 Rounding

Raw data from the samplers are preferably recorded electronically and/or communicated by electronic means to a central computer where they are recorded. Electronically stored data and computerized calculations should not be rounded until final formatting for reports or for delivery to AIRS.

Filter weight data for calculating PM<sub>2.5</sub> concentrations should be recorded to the full readable precision of the microbalance. Hand-calculated weight differences should be carried to the same number of digits and should not be rounded.

For other hand-recorded data, including calibration and audit data, it is advisable to read and record somewhat more resolution than will be needed for calculating the final result. For example, to make a measurement that will be reported to the nearest 1 percent, attempt to read the data to at least the nearest 0.5 percent or better. It is usually not necessary to record a resolution more than a factor of 10 better than that needed in the final result, however.

Special care should be taken when recording data that will be used in a difference calculation: enough significant figures must be recorded so that the **result** will have the necessary resolution. The most obvious example is the determination of PM<sub>2.5</sub> mass by the difference of two filter

weights. Measurement of the filter weight to six significant figures is necessary in order to get adequate precision in the net particulate mass. All data recording forms should specify the appropriate number of digits, or the necessary resolution in physical units, for each parameter that must be manually recorded.

### 11.5.2 Rounding Rules for Comparison with the National Ambient Air Quality Standards

For comparing calculated concentration averages to National Ambient Air Quality Standards (NAAQS) levels, it is necessary to use round-off rules defined in Sections 2.5 and 2.6 of 40 CFR Part 50, Appendix N. For the annual PM<sub>2.5</sub> standard (currently 15 µg/m<sup>3</sup>), the 3-year average of the spatially averaged annual means is rounded to the nearest 0.1 mg/m<sup>3</sup>. Decimals 0.05 and greater are rounded up to the next 0.1, and any decimal lower than 0.05 is rounded down to the nearest 0.1. For the 24-hour PM<sub>2.5</sub> standard (currently 65 µg/m<sup>3</sup>), the 3-year average of the annual 98th percentile values is rounded to the nearest 1 mg/m<sup>3</sup>. Decimals 0.5 and greater are rounded up to the nearest whole number, and any decimal lower than 0.5 is rounded down to the nearest whole number.

A different method is required for determining the annual 98th percentile concentration values used for the 24-hour NAAQS. According to the method described in Section 2.6 of 40 CFR Part 50, Appendix N, the observation number used to represent the 98th percentile value is always rounded up, never down. The daily concentration observations for  $N$  yearly observations are arranged in order of size, from smallest to largest (i.e., from 1 to  $N$ ). The 98th percentile concentration value for a particular year is defined by the CFR as

$$P_{0.98,y} = X_{[i+1]}$$

where

- $P_{0.98,y}$  Is the 98th percentile concentration value for year  $y$
- $X_{[i+1]}$  Is the  $(i+1)$ th concentration in the ordered series of concentration values for year  $y$
- $i$  Is the integer part of the product of 0.98 times the total number of valid concentration values collected during year  $y$  (i.e.,  $0.98iN$ ).

Taking the  $(i+1)$ st value is equivalent to rounding the observation number up to the next highest integer, regardless of whether the fractional part of  $0.98iN$  is greater than or less than 0.5.

## 12.0 Data and Records Management

### 12.1 Overview

It is important to keep good records in any air pollution measurement program. This is particularly true for measurements of PM<sub>2.5</sub> by reference or Class I equivalent methods because these are manual methods that involve the transfer and handling of samples and data by several people. Automated methods, on the other hand, present the final, averaged data with much less human intervention and, thus, limit the possibilities of transcription errors and misplacement of data.

Keeping good records and managing the data obtained under PM<sub>2.5</sub> regulations are especially important because this is a newly designated criteria pollutant, and the samplers and methodology for its determination are new to most users. Thus, records of knowledge gained in the initial stage of network operation will be of great help in improving and refining all operational aspects of PM<sub>2.5</sub> determinations.

Section 14 of the U.S. EPA's *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Ambient Air Specific Methods, Part I*, gives guidance on data acquisition and information management (EPA 1997). Although it emphasizes automated data acquisition, much of the discussion is also applicable to manual methods. There are several reasons for maintaining complete, orderly records and properly managing data. Records can

- Provide information on mechanical problems that occur and document how the problems were corrected
- Provide a history of warranty repairs
- Provide a history of in-house repairs and preventive maintenance servicing
- Document date and site placement details for the primary and collocated samplers as well as the characteristics of the surrounding land areas, sources, and other features
- Be a useful source of information at the time of the annual network review to show proper sampler installation and operation, performance of quality control (QC) and quality assurance (QA) checks, traceability of equipment and standards, and proof that all systems were kept in control (use of control charts)
- Provide evidence to support the quality of PM<sub>2.5</sub> data submitted to regional and national databases.

### 12.2 Methodology for Data and Records Management

A systematic approach to maintaining records and managing data for all reported ambient air pollutant concentrations usually involves the following considerations:

- **Personnel**—One or more persons should be identified within the reporting organization to be responsible for maintaining the records and preparing and submitting the required data products. Education, training, and experience are requirements for the position. Standard Operating Procedures (SOPs) will also be required.

- **Quality assurance**—An individual or group should be assigned to prepare SOPs, to conduct internal systems and performance audits, and to review the data before submission.
- **Facilities and equipment**—A designated storage location for paper records and computer data files should be established. Using file cabinets that can be locked is suggested. A backup copy of data files should be kept in another location for safety. Computers and data entry stations, as well as connections to centralized databases (such as the Aerometric Information Retrieval System [AIRS]), are also required.

### 12.3 PM<sub>2.5</sub> Records to Create and Retain

A quality assurance project plan (QAPP) and SOPs for various aspects of the PM<sub>2.5</sub> sampling program should be prepared. Much of the required information can be drawn from that given in the applicable sections of the *Code of Federal Regulations (CFR)*, the sampler's instruction manual, and this document. Records to maintain are summarized below.

#### 12.3.1 Sampler Siting and Maintenance Records

Documentation regarding siting and maintenance of the PM<sub>2.5</sub> samplers should be retained in the files. These documents may include the following:

- Site selection criteria checklist; photographs or color slides of the site in the eight cardinal directions, with the PM<sub>2.5</sub> sampler in the center foreground; site sketch; U.S. Geological Survey (USGS) topographical map of the area; and a copy of the site documentation records that were submitted to the AIRS database when the site was first established and any updates made since that time.
- Procurement log for field equipment (see Figure 4-1). Notes on acceptance/rejection tests (see Table 4-1).
- Warranty and maintenance records for each PM<sub>2.5</sub> sampler. A dated maintenance checklist should be established for each sampler and separate maintenance logbooks should be used for the PM<sub>2.5</sub> samplers and the laboratory microbalance(s).
- Manufacturer-supplied calibration and traceability records for the thermometers, flow rate measuring devices, and pressure sensors used for calibrating, checking, or auditing PM<sub>2.5</sub> samplers. Maintain similar records for calibrations and traceability studies conducted in the reporting organization's laboratory or elsewhere.

#### 12.3.2 Analytical Laboratory Installation Records

Records to keep about the physical setup of the analytical laboratory include the following:

- Equipment inventory (microbalances, conditioning chambers, antistatic devices, calibration and check weights, and so on).
- Procurement log (see Figure 4-1). Notes on acceptance/rejection tests (see Table 4-1)
- Records (e.g., control charts) that demonstrate that the temperature and relative humidity (RH) within the weighing laboratory and conditioning chambers were held within specified limits

- Microbalance logbook.

### 12.3.3 Field Sampling Operation Records

PM<sub>2.5</sub> sampling operations generate a number of paper and/or computerized records that need to be reviewed, reported, and filed. Starting with the calibration of the sampler's sensors, these records will include at least the following items:

- Sampler calibration logbooks or data sheets for entering results of temperature, pressure, and flow rate checks, audits, and calibrations. Also include documentation of the devices used to calibrate the sampler(s) and these devices' traceability records or references to the traceability records. A separate section of the logbook should be used for each sampler in the network. Enter results of the sampling procedure checks (see Table 8-1), which include notes on filter inspection, and checks of flow rate and leaks.
- PM<sub>2.5</sub> sampler run data sheet (see Figure 8-1 for an example). The run data sheet should be prepared at least in duplicate by using carbonless paper or making copies. The original of the data sheet should accompany the filter sample to the laboratory, and a copy should be retained in the field site records files.
- PM<sub>2.5</sub> sampler flow check data sheet (see Figure 8-2 for an example).
- Computer disks and printouts of data downloaded from the data port of each PM<sub>2.5</sub> sampler.

### 12.3.4 Weighing Laboratory Operation Records

Data records from activities in the weighing laboratory are expected to include at least the following items:

- Records of temperature and humidity control in the laboratory and in the filter conditioning environment (if the latter is different from the laboratory). Use of control charts is recommended.
- Laboratory data form (see Figure 7-2 for an example).
- Laboratory internal QC log (see Figure 7-1 for an example).
- Results of microbalance calibrations and servicing. This information can be recorded in the microbalance logbook.
- Results of filter integrity checks and determinations of the conditioning periods required for various filter batches (refer to Sections 7-5 and 7-6).
- The completed PM<sub>2.5</sub> sampler run data sheet (see Figure 8-1 for an example).
- Records of sample numbers (identifications) and locations of archived PM<sub>2.5</sub> samples.

### 12.3.5 Quality Assurance Records

Quality assurance systems and performance audits require complete documentation as well. Records of audits may be similar to the following items:

- Flow rate, temperature, and pressure audit data sheet (see Figure 10-1 for an example)
- Systems audit questionnaire (see Figure 10-2 for an example).

Accuracy and precision audit results are based on comparing the data from the primary sampler (the one used regularly at the site) to data from collocated samplers. These data are reported to the EPA Regional Office and to the AIRS database as noted in Sections 11.0 and 12.4.

## 12.4 Data Reporting Requirements

The EPA Regional Office and the AIRS database require the following information be reported on a quarterly (generally, calendar-quarter) basis (EPA will calculate measurement uncertainties for the entire calendar year):

- Siting documentation (upon installation and any changes thereafter)
  - PM<sub>2.5</sub> concentration data,  $\mu\text{g}/\text{m}^3$ , or sample weight and volume sampled to allow electronic calculations (entered electronically into the AIRS database)
  - Information calculated and provided by the sampler (refer to Table 12-1 [Adapted from 40 CFR Part 50, Appendix L, Table L-1]). This information is retrieved from the data port of each sampler. It is generally downloaded in the field to a laptop computer or another data storage device and later processed in the laboratory or office. Data outputs to be provided to the AIRS database are indicated in Table 12-1 by the bullet symbol.
- C Results of all valid precision, bias, and accuracy tests performed during the quarter. Refer to Section 10.0 for details.

Features can be incorporated into the processing program to detect and correct typographical errors, such as out-of-sequence dates for start and end times, extremely high (or low) filter weights, flow rates, and temperatures.

**Table 12-1. Summary of Information to be Provided by Sampler**

| Information to be provided                                      | Appendix L section reference | Availability         |                            |                             |                          | Format                       |                |
|---|------------------------------|----------------------|----------------------------|-----------------------------|--------------------------|------------------------------|----------------|
|   |                              | Anytime <sup>a</sup> | End of period <sup>b</sup> | Visual display <sup>c</sup> | Data output <sup>d</sup> | Digital reading <sup>e</sup> | Units          |
| Flow rate, 30-sec maximum interval                              | 7.4.5.1                      | Y                    | —                          | Y                           | ρ                        | XX.X                         | L/min          |
| Flow rate, average for the sample period                        | 7.45.2                       | ρ                    | Y                          | ρ                           | Y                        | XX.X                         | L/min          |
| Flow rate, coefficient of variation (CV), for the sample period | 7.4.5.2                      | ρ                    | Y                          | ρ                           | Y □                      | XX.X                         | %              |
| Flow rate, 5-min average out of specification <sup>f</sup>      | 7.4.5.2                      | Y                    | Y                          | Y                           | Y □                      | On/off                       |                |
| Sample volume, total  | 7.4.5.2                      | ρ                    | Y                          | Y                           | Y □                      | XX.X                         | m <sup>3</sup> |
| Temperature, ambient, 30-sec interval                           | 7.4.8                        | Y                    | —                          | Y                           | —                        | XX.X                         | EC             |

Table 12-1 (continued)

| Information to be provided   | Appendix L section reference | Availability         |                            |                             |                          | Format                        |                                |
|--|------------------------------|----------------------|----------------------------|-----------------------------|--------------------------|-------------------------------|--------------------------------|
|  |                              | Anytime <sup>a</sup> | End of period <sup>b</sup> | Visual display <sup>c</sup> | Data output <sup>d</sup> | Digital reading <sup>e</sup>  | Units                          |
| Temperature, ambient, min., max., average for the sample period                      | 7.4.8                        | ρ                    | Y                          | Y                           | Y □                      | XX.X                          | EC                             |
| Barometric pressure, ambient, 30-sec interval  | 7.4.9                        | Y                    | —                          | Y                           | —                        | XXX                           | mmHg                           |
| Barometric pressure, ambient, min., max., average for the sample period              | 7.4.9                        | ρ                    | Y                          | Y                           | Y □                      | XXX                           | mmHg                           |
| Filter temperature, 30-sec interval  | 7.4.11                       | Y                    | —                          | Y                           | —                        | XX.X                          | EC                             |
| Filter temperature, differential, 30-min interval, out of specification <sup>f</sup> | 7.4.11                       | ρ                    | Y                          | Y                           | Y □                      | On/Off                        |                                |
| Filter temperature, maximum differential from ambient, date, time of occurrence      | 7.4.11                       | ρ                    | ρ                          | ρ                           | ρ                        | X.X,<br>YY/MM/D<br>D<br>HH:mm | EC,<br>yr/mo/<br>day hr<br>min |
| Date and time  | 7.4.12                       | Y                    | —                          | Y                           | —                        | YY/MM/D<br>D HH:mm            | yr/mo/<br>day hr<br>min        |
| Sample start and stop time settings  | 7.4.12                       | Y                    | Y                          | Y                           | Y                        | YY/MM/D<br>D HH:mm            | yr/mo/<br>day hr<br>min        |
| Sample period start time   | 7.4.12                       | —                    | Y                          | Y                           | Y □                      | YYYY/MM<br>M/DD<br>HH:mm      | yr/mo/<br>day hr<br>min        |
| Elapsed sample time  | 7.4.13                       | ρ                    | Y                          | Y                           | Y □                      | HH:mm                         | hr min                         |
| Elapsed sample time out of specification <sup>f</sup>                                | 7.4.13                       | —                    | Y                          | Y                           | Y □                      | On/Off                        |                                |
| Power interruptions >1 min, start time of first 10                                   | 7.4.15.5                     | ρ                    | Y                          | ρ                           | Y                        | 1HH:mm,<br>2HH:mm,<br>etc.    | hr min                         |
| User-entered information, such as sampler and site identification                    | 7.4.16                       | Y                    | Y                          | Y                           | Y □                      | As entered                    |                                |

See notes on following page.

**Table 12-1 (continued)**

Y Provision of this information is required.

ρ Provision of this information is optional. If information related to the entire sample period is optionally provided prior to the end of the sample period, the value provided should be the value calculated for the portion of the sampler period completed up to the time the information is provided.

□ This information is also required to be provided to the AIRS database.

<sup>a</sup> This information is required to be available to the operator any time the sampler is operating, whether sampling or not.

<sup>b</sup> This information relates to the entire sampler period and must be provided following the end of the sample period until reset manually by the operator or automatically by the sampler upon the start of a new sample period.

<sup>c</sup> This information shall be available to the operator visually.

<sup>d</sup> This information should be available as digital data at the sampler's data output port following the end of the sample period until reset manually by the operator or automatically by the sampler upon the start of a new sample period.

<sup>e</sup> Digital readings, both visual and data output, shall have no less than the number of significant digits and resolution specified.

<sup>f</sup> Flag warnings may be displayed to the operator by a single-flag indicator or each flag may be displayed individually. Only a set (on) flag warning must be indicated; an off (unset) flag may be indicated by the absence of a flag warning. Sampler users should refer to Section 10.12 of Appendix L regarding the validity of samples for which the sampler provided an associated flag warning.

## 13.0 Assessment of Measurement Uncertainty for Monitoring Data

### 13.1 Overview

State and Local Air Monitoring Stations (SLAMS) reporting organizations are required to assess the measurement uncertainty of their PM<sub>2.5</sub> monitoring data (EPA 1997). Three different procedures are used in this assessment:

- Flow rate audit—The accuracy of the PM<sub>2.5</sub> sampler's flow rate is assessed by performing a flow rate audit as described in Section 10.2.4. Performance of this audit is the responsibility of the State and/or the reporting organization. The goal for accuracy of the sampler flow rate is to be within  $\pm 2$  percent of the value indicated by the audit device (during multipoint verification/calibration) or  $\pm 4$  percent (during one-point verification checks and audits) and to be within  $\pm 5$  percent of the sampler's design flow rate (16.67 L/min).
- Bias—PM<sub>2.5</sub> measurement bias is assessed by conducting a Federal Reference Method (FRM) collocated sampler audit. The U.S. EPA Regional Offices will conduct this performance evaluation. The bias goal is to be between -10 percent and +10 percent (i.e., to have the site sampler's PM<sub>2.5</sub> concentration value agree within  $\pm 10$  percent of the audit value).
- Precision—PM<sub>2.5</sub> measurement precision is assessed by collocating (i.e., locating a second, duplicate sampler alongside the primary sampler used for measurement) samplers and reporting the results of both the measurement sampler and the collocated sampler. Operation of the duplicate samplers is the responsibility of the State and/or the reporting organization. The precision goal is to have a coefficient of variation (CV) of less than 10 percent.

These procedures are very similar to those used in the data quality assessment (DQA) of PM<sub>10</sub> monitoring data. The results of these procedures are used to screen individual samplers for bias or excessive imprecision. EPA also uses these results to assess measurement uncertainty for each reporting organization. The results from all SLAMS reporting organizations are used by EPA to assess the measurement uncertainty of data from each PM<sub>2.5</sub> reference or equivalent method on a national basis.

### 13.2 Flow Rate Audits

Flow rate audits consist of measuring the sampler's normal operating flow rate with a flow rate transfer standard. This procedure is described in Section 10.2.1. Manual PM<sub>2.5</sub> sampler methods must be audited each calendar quarter. Audits should be scheduled to avoid interference with regularly scheduled sampling periods. Whenever possible, these flow audits should be performed at randomly selected times with respect to time of day and day of week.

Results of the flow rate audit to be reported to EPA are as follows:

- The audit (true) flow rate as determined using the transfer standard.
- The corresponding flow rate as measured by the sampler. This rate should be that used by the sampler to calculate its reported total sampling volume and average flow rate. This information is typically obtained from the sampler's flow rate display or from its data

system.

The sampler's flow rate accuracy ( $A$ ) should be within  $\pm 4$  percent of the audit value. Furthermore, the audit measured flow rate accuracy ( $A_D$ ) should be within  $\pm 5$  percent of the sampler's design inlet flow rate (16.67 L/min). The sampler's flow rate accuracies ( $A$  and  $A_D$ ) are calculated as follows:

$$A(\%) = \frac{Q_{\text{Sampler}} \& Q_{\text{Audit}}}{Q_{\text{Audit}}} \times 100 \quad (1)$$

$$A_D(\%) = 100 \times (Q_{\text{sampler}} - 16.67) / 16.67 \quad (2)$$

where

- $A$  = flow rate accuracy (percent)
- $A_D$  = flow rate accuracy (percent) versus design flow rate
- $Q_{\text{Sampler}}$  = flow rate as measured by sampler (L/min)
- $Q_{\text{Audit}}$  = flow rate as measured by the flow rate transfer standard (L/min)
- 16.67 = design flow rate (L/min).

### 13.3 Bias Assessment

Sampler bias is assessed from the results of an FRM performance evaluation (PE) (Section 10.2.3) administered through the EPA Regional Offices. The goal for acceptable bias is between -10 percent and +10 percent (i.e., the reporting primary sampler's concentration should agree to within  $\pm 10$  percent of the FRM PE sampler's concentration). Minimum FRM PE requirements for SLAMS reporting organizations include the following:

- At least one sampler must be audited annually.
- At least 25 percent of each reference method and of each EPA-designated equivalent method must be audited each year (i.e., in most cases, 25 percent of each make and model of sampler will need to be audited). This percentage includes collocated sites, even those collocated with FRM instruments. Thus, if a reporting organization has three different makes of samplers, each with a different equivalency designation, the reporting organization will have to audit 25 percent of each make of sampler. If a fractional number of samplers is required, values of 0.5 or greater must be rounded up to the nearest whole number. For example, a reporting organization with 10 samplers of a given type must audit at least 3 of those samplers.
- Audits must occur at least four times a year.
- All samplers must be audited at least once every 4 years.

In addition to these requirements, areas that exceed the National Ambient Air Quality Standard (NAAQS) for  $\text{PM}_{2.5}$  should place special emphasis on those sites with the highest  $\text{PM}_{2.5}$  concentrations as follows:

- Reporting organizations with sites reporting  $\text{PM}_{2.5}$  concentrations equal to or exceeding 90 percent of the NAAQS (annual or 24 hour, as appropriate):

- 80 percent of FRM PEs should be performed at those sites reporting PM<sub>2.5</sub> concentrations equal to or exceeding 90 percent of the NAAQS (annual or 24 hour, as appropriate).
- The remaining 20 percent of FRM PEs should be performed at sites reporting concentrations less than 90 percent of the NAAQS (annual or 24 hour, as appropriate).
- Reporting organizations **without** sites reporting PM<sub>2.5</sub> concentrations exceeding 90 percent of the NAAQS (annual or 24 hour, as appropriate):
  - 60 percent of FRM PEs should be performed at sites that rank in the top 25 percent of the highest sites for PM<sub>2.5</sub> concentrations (annual or 24 hour, as appropriate).
  - The remaining 40 percent of FRM PEs should be performed at the remaining 75 percent of sites.

The individual sampler and audit measurements must be reported to EPA, which uses them to calculate the single sampler bias ( $B$ ) and the quarterly average bias ( $\bar{B}$ ) for a reporting organization. Reporting organizations may also want to calculate these parameters for their quality assurance (QA) use. The single sampler bias ( $B$ ) may be calculated as follows:

$$B(\%) = \frac{PM_{2.5\text{Sampler}} \& PM_{2.5\text{Audit}}}{PM_{\text{Audit}}} \times 100 \quad (3)$$

where

- $B$  = bias (percent)
- $PM_{2.5\text{Sampler}}$  = PM<sub>2.5</sub> concentration as measured by sampler (µg/m<sup>3</sup>)
- $PM_{2.5\text{Audit}}$  = PM<sub>2.5</sub> concentration as measured by audit device (µg/m<sup>3</sup>).

The quarterly average bias for a single sampler ( $\bar{B}$ ) may also be calculated as follows

$$\bar{B} = \frac{1}{n} \times \sum_{i=1}^n B_i \quad (4)$$

where

- $\bar{B}$  = average quarterly bias (percent)
- $B_i$  = bias for the  $i$ th measurement (percent)
- $n$  = number of measurements during quarter.

## 13.4 Precision

### 13.4.1 Overview

PM<sub>2.5</sub> precision is assessed by collocating samplers (i.e., locating a second sampler alongside the primary sampler used to report a measurement). Both samplers are run identically and the results

from both samplers are reported to EPA. The collocated (duplicate) sampler's inlet must be placed horizontally between 1 and 4 m from the reporting sampler and within 1 m of the same height (elevation) as the reporting sampler (Section 5.1.2). If there is a high volume sampler at the site, both the primary and duplicate PM<sub>2.5</sub> samplers must be separated from it by at least 2 m. The collocated (duplicate) sampler must be operated and its sample processed in exactly the same way as the primary reporting sampler and should be operated at least every sixth day on the same start time/end time schedule. It is important always to designate one sampler as the primary or reporting sampler and the other always as the collocated or duplicate sampler, even when the reporting sampler may be identical in make and model to the collocated sampler. The precision goal is to have a CV of less than 10 percent. Refer to the CFR Part 40, Section 58, Appendix A, Section 5.5, for detailed calculation procedures. Under no circumstances should data from the collocated sampler be reported as coming from the reporting sampler. However, collocated sampler data should always be reported even when the primary sampler's data are nonexistent or invalid.

Concentration data must be reported for both the primary and collocated sampler, no matter how low the concentration. Because agreement between the measurements of collocated samplers may be relatively poor at low concentrations, collocated measurement pairs are used by EPA to calculate precision only when both PM<sub>2.5</sub> measurement concentrations are above 6 µg/m<sup>3</sup>.

#### 13.4.2 Number of Collocated Samplers Required

To assess precision of their sampling data, SLAMS reporting organizations must meet the following collocation requirements:

- At least one reporting sampler must have a collocated sampler.
- At least one of the collocated samplers must be an FRM sampler (i.e., not a designated equivalent method sampler).
- At least 25 percent of all reporting samplers must have collocated samplers. If fractional numbers of collocated samplers are required, values of 0.5 or greater must be rounded up to the nearest sampler. For example, a reporting organization with 10 reporting samplers must provide collocated samplers for at least 3 of those reporting samplers.

In addition to these requirements, additional specifications must also be observed with respect to the different makes and models of samplers used as collocated samplers:

- Collocated samplers for FRM-designated reporting samplers shall always be of the identical FRM designation (i.e., the collocated and reporting samplers are of the same make and model).
- If the reporting sampler is a Federal Equivalent Method (FEM), half of the collocated samplers for the designated equivalent reporting samplers should have the identical equivalency designation (the same make and model), while the other half should be FRM-designated samplers. In cases where odd numbers of collocated samplers are required, the number of collocated FRM samplers should always be rounded up to the nearest sampler, with the remaining number being samplers of the same equivalent designation (samplers of the same make or model). For example, if five collocated samplers are required, then three should be FRM models and the remaining two should be models of the same equivalency

designation.

As an example, consider the minimum collocation requirements for an organization that has 43 reporting samplers composed of 25 FRM samplers, 10 equivalent designation A samplers, 2 equivalent designation B samplers, and 6 equivalent designation C samplers. Each type of sampler should be considered separately as shown in Table 13-1 for sampler types A, B, and C.

### 13.4.3 Location of Collocated Samplers

Collocated samplers should be placed at sites that have the highest PM<sub>2.5</sub> concentrations, with special emphasis on sites likely to be in violation of the NAAQS. Data from other particulate measurement methods, such as PM<sub>10</sub> samplers, can be helpful in selecting sites for collocation.

SLAMS reporting organizations that have areas in violation of the NAAQS should place their collocated samplers as follows:

- Reporting organizations with sites reporting PM<sub>2.5</sub> concentrations equal to or exceeding 90 percent of the NAAQS (annual or 24 hour, as appropriate):
  - 80 percent of the collocated samplers should be located at those sites that equal or exceed 90 percent of the NAAQS (annual or 24 hour, as appropriate).
  - The remaining 20 percent of collocated samplers should be located at sites that report less than 90 percent of the NAAQS (annual or 24 hour, as appropriate).
- Reporting organizations **without** sites reporting PM<sub>2.5</sub> concentrations exceeding 90 percent of the NAAQS (annual or 24-hour, as appropriate):
  - 60 percent of collocated samplers should be located at sites that rank in the top 25 percent of the highest sites for PM<sub>2.5</sub> concentrations (annual or 24 hour, as appropriate).
  - The remaining 40 percent of collocated samplers should be located in the remaining 75

**Table 13-1. Example of Collocated Sampler Requirements**

| Sampler type                     | Number of samplers | × 25 percent | Rounded to nearest sampler | Required collocated samplers | Required collocated samplers by type |                      |
|----------------------------------|--------------------|--------------|----------------------------|------------------------------|--------------------------------------|----------------------|
|                                  |                    |              |                            |                              | FRM                                  | Same equivalent type |
| FRM samplers                     | 25                 | 6.25         | 6                          | 6                            | 6                                    | NA                   |
| Equivalency-designated samplers  |                    |              |                            |                              |                                      |                      |
| Type A                           | 10                 | 2.5          | 3                          | 3                            | 2                                    | 1                    |
| Type B                           | 2                  | 0.5          | 1                          | 1                            | 1                                    | 0                    |
| Type C                           | 6                  | 1.5          | 2                          | 2                            | 1                                    | 1                    |
| Total for reporting organization | 43                 |              |                            | 12                           | 10                                   | 2                    |

NA = Not applicable.

percent of sites.

#### 13.4.4 Schedule for Operation of Collocated Samplers

Collocated samples should be taken to reflect the normal operation of the reporting sampler. The collocated samples should be evenly distributed across the various seasons and days of the week. A simple system to accomplish this is to run the collocated sampler every sixth day. Thus, if the first week's collocated sample was run on Monday, the second week's collocated sample would be run on Sunday, the third week's on Saturday, and so on. Remember to start and stop both the collocated and reporting samplers at exactly the same time for each sample run.

## 14.0 References

### Section 1.0

ANSI/ASQC. 1994. American national standard—Specifications and guidelines for quality systems for environmental data collection and environmental technology programs. American National Standard Institute/American Society for Quality Control. ANSI/ASQC E4-1994. Available from American Society for Quality Control, 611 East Wisconsin Avenue, Milwaukee, WI 53202.

Engelbrecht DR, Cahill T, Feeney PJ. 1980. Electrostatic effects on gravimetric analysis of membrane filters. *J Air Poll Control Assoc* 30:391-392.

EPA. 1995. Guidance for the preparation of standard operating procedures (SOPs). U.S. Environmental Protection Agency. Publication QA/G-6, U.S. EPA Quality Assurance Division, Washington, DC. November.

EPA. 1997a. Reference method for the determination of fine particulate matter as  $PM_{2.5}$  in the atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L.

EPA. 1997b. Ambient air quality surveillance. U.S. Environmental Protection Agency. 40 CFR Part 58, as amended July 18, 1997.

EPA. 1997c. Ambient air quality monitoring methodology. U.S. Environmental Protection Agency. 40 CFR Part 58, Appendix C, as amended July 18, 1997.

EPA. 1997d. Ambient air monitoring reference and equivalent methods. U.S. Environmental Protection Agency. 40 CFR Part 53, as amended July 18, 1997.

Lipfert FW. 1994. Filter artifacts associated with particulate measurements: recent evidence and effects on statistical relationships. *Atmos Environ* 28:391-392.

Papp ML, Elkins JB, Musick DR, Messner MJ. 1998. Data Quality Objectives for the  $PM_{2.5}$  Monitoring Data. U.S. Environmental Protection Agency, Research Triangle Park, NC. In preparation.

### Section 2.0

EPA. 1998. Quality assurance handbook for air pollution measurement systems. Volume II: Ambient air specific methods, Part I. U.S. Environmental Protection Agency. EPA-454/R-98-004, August 1998.

### Section 3.0

EPA. 1997a. Reference method for the determination of fine particulate matter as  $PM_{2.5}$  in the atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L.

EPA. 1997b. Procedure for testing physical (design) and performance characteristics of reference methods and Class I equivalent methods for  $PM_{2.5}$ . U.S. Environmental Protection Agency. 40 CFR Part 53, subpart E, Appendix A, as revised July 18, 1997.

## Section 4.0

- ASTM. 1992. Standard test method for inspection and verification of thermometers. American Society for Testing and Materials. Philadelphia, PA. ASTM Standard E77-92.
- ASTM. 1995a. Standard specification for ASTM thermometers. American Society for Testing and Materials. Philadelphia, PA. ASTM Standard E1-95.
- ASTM. 1995b. Standard test methods for measuring surface atmospheric pressure. American Society for Testing and Materials. Publication number ASTM D3631-95.
- ASTM. 1995c. Standard practice for evaluation of air assay media by the monodisperse DOP (Dioctylphthalate) smoke test. American Society for Testing and Materials. Philadelphia, PA. ASTM Standard D2986-95a.
- EPA. 1995. Quality assurance handbook for air pollution measurement systems. Volume IV: meteorological measurements. Section 4.3.5. U.S. Environmental Protection Agency.
- EPA. 1997a. Reference method for the determination of fine particulate matter as  $PM_{2.5}$  in the atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L.
- EPA. 1997b. Ambient air monitoring reference and equivalent methods. U.S. Environmental Protection Agency. 40 CFR Part 53, as amended July 18, 1997.
- Harris GL. 1994. State weights and measures laboratories: state standards program description. National Institute of Standards and Technology. Special publication 791. 130 pp.
- Nelson, G.O. 1992. *Gas Mixtures: Preparation and Control*. Lewis Publishers, Boca Raton, FL, 304 pp.
- NIST. 1986. Thermometer calibration: A model for State calibration laboratories. National Institute of Standards and Technology. NBS monograph 174. January.
- NIST. 1988. Liquid-in-glass thermometer calibration service. National Institute of Standards and Technology. Special publication 250-23. September.
- NIST. 1989. The calibration of thermocouples and thermocouple materials. National Institute of Standards and Technology. Special publication 250-35. April.
- White VR. 1997. National Voluntary Laboratory Accreditation Program: 1997 Directory. National Institute of Standards and Technology. Special publication 810. 225 pp.

## Section 5.0

- EPA. 1997a. Network design for State and local air monitoring stations (SLAMS), national air monitoring stations (NAMS), and photochemical assessment monitoring stations (PAMS). U.S. Environmental Protection Agency. 40 CFR Part 58, Appendix D, as amended July 18, 1997.
- EPA 1997b. Guidance for network design and optimum site exposure for  $PM_{2.5}$  and  $PM_{10}$ . U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Finalized version, December 1997.
- EPA. 1997c. Reference method for the determination of fine particulate matter as  $PM_{2.5}$  in the atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L.

## Section 6.0

- ASTM. 1977. Standard test methods for measuring surface atmospheric pressure. American Society for Testing and Materials. Philadelphia, PA. Standard D 3631-84.
- EPA. 1995. Quality Assurance Handbook for Air Pollution Measurement Systems Volume IV: Meteorological Measurements. U.S. Environmental Protection Agency. Document No. EPA/600/R-94/038d. Revised March.
- EPA. 1997. Reference method for the determination of fine particulate matter as  $PM_{2.5}$  in the atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 58, Appendix L, as amended July 18, 1997.
- EPA 1998. Quality Assurance Handbook for Air Pollution Measurement Systems. Volume II, Part I. Ambient Air Specific Methods. Appendix 12, "Calibration of Primary and Secondary Standards for Flow Measurements."
- NIST. 1976. Liquid-in-glass thermometry. National Institute of Standards and Technology. NBS Monograph 150. January.
- NIST. 1986. Thermometer calibration: a model for state calibration laboratories. National Institute of Standards and Technology. NBS Monograph 174. January.
- NIST. 1988. NIST measurement services: liquid-in-glass thermometer calibration service. National Institute of Standards and Technology. Special publication 250-23. September.
- NIST. 1989. The calibration of thermocouples and thermocouple materials. National Institute of Standards and Technology. Special publication 250-35. April.

## Section 7.0

- ASTM. 1993a. Standard practice for maintaining constant relative humidity by means of aqueous solutions. American Society for Testing and Materials. 1993 Annual Book of ASTM Standards, Vol 11.03, Designation E 104-85 (reapproved 1991), pp. 570–572.
- ASTM. 1993b. Standard specification for laboratory weights and precision mass standards. American Society for Testing and Materials. 1993 Annual Book of ASTM Standards, Vol 14.02, Designation E 617-91, pp. 280–295.
- Desert Research Institute. 1994. DRI Standard Operating Procedure. Gravimetric Analysis, DRI SOP No. 2-102.3, Reno, NV. 24 pp.
- Eisner AD. 1997. Personal communication. ManTech Environmental Technology, Inc., Research Triangle Park, NC.
- Engelbrecht DR, Cahill TA, Feeney PJ. 1980. Electrostatic effects on gravimetric analysis of membrane filters. *Journal of the Air Pollution Control Association* 30(4):319–392.
- EPA. 1997. Reference method for the determination of fine particulate matter as  $PM_{2.5}$  in the atmosphere. U.S. Environmental Protection Agency. 40 CFR Part 50, Appendix L.
- Harris G. 1993. Ensuring accuracy and traceability of weighing instruments. *ASTM Standardization News* 21(4):44–51.
- Harris GL. 1994. State weights and measures laboratories: State standards program description.

National Institute of Standards and Technology. Special publication 791. 130 pp.

Hawley RE, Williams CJ. 1974. Electrostatic effects in microbalances. I. General considerations of the effectiveness of a radioactive ionizing source under ambient conditions. *Journal of Vacuum Science and Technology* 11(1):419–422.

Kupper WE. 1990. High accuracy mass measurements, from micrograms to tons. *Instrument Society of America Transactions* 29(4):11–20.

Weil J. 1991. Static control for balances. Cahn Technical Note. Published by Cahn Instruments, Inc., Madison, WI.

White VR. 1997. National Voluntary Laboratory Accreditation Program: 1997 Directory. National Institute of Standards and Technology. Special publication 810. 225 pp.

### **Section 10.0**

EPA. 1994. EPA requirements for quality assurance project plans for environmental data operations. U.S. Environmental Protection Agency. Document EPA QA/R-5. External Review draft final, October 1998.

EPA. 1997. Ambient air quality surveillance. U.S. Environmental Protection Agency. 40 CFR Part 58, Appendix A—Quality assurance requirements for State and local air monitoring stations (SLAMS); Appendix E—Probe and monitoring path siting criteria for ambient air quality monitoring, as revised July 18, 1997.

### **Section 12.0**

EPA. 1998. Data acquisition and information management. Section 14 of Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Part I, Ambient Air Specific Methods. U.S. Environmental Protection Agency. EPA-454/R-98-004, August 1998.

### **Section 13.0**

EPA. 1997. Quality assurance requirements for State and local air monitoring stations (SLAMS). U.S. Environmental Protection Agency. 40 CFR Part 58, Appendix A, as amended July 18, 1997.

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## Appendix A—Measuring Alkalinity of Filters

*Note to the reader. The procedure given here is one created for and extensively tested for use with glass fiber or quartz high-volume filters. It is presented as guidance to the analyst for devising and testing a similar procedure for determining the acid-base character of PM<sub>2.5</sub> sampling filters.*

### A.1 History

Alkalinity of hi-vol filters is measured because alkaline sites react with SO<sub>2</sub>, NO<sub>x</sub> and other acid gases in the air forming sulfates, nitrates, etc. Such compounds' masses are indistinguishable from particulate mass collected by the filter. In turn, the total particulate weight, sulfate, content, nitrate content, etc., would be falsely high in proportion to filter alkalinity.

### A.2. Scope and Applicability

This method describes procedures for measuring the alkalinities of glass and quartz fiber filters. New filters are tested for compliance with EPA alkalinity specifications and older filters are tested in order to evaluate appropriate correction factors for previously obtained particulate data. Table A-1 outlines the procedure.

### A.3 Equipment and Reagents

Table A-2 lists the equipment and reagents. The supplier is always Fisher Scientific Company, unless otherwise specified. (This information is for reference purposes only.)

**TABLE A-1. GENERAL DESCRIPTION OF EPA FILTER ALKALINITY PROCEDURES**

| Parameter                              | EPA specification  |
|--|--|
| Sample container                       | 250-mL Phillips beaker   |
| H <sub>2</sub> O extraction volume, mL | 100  |
| Heating technique                      | Hotplate, reflux condenser (sample temperature about 100 EC)                   |
| Extraction time, min                   | 20   |
| Mixer                                  | Magnetic stirrer with 2-in stirring bar  |
| Filtration equipment                   | 7-cm ID unheated Buchner funnel 250-mL filter flask                            |
| Filtration procedure                   | Twice through Whatman 41 or 541  |
| Titration vessel                       | 250-mL filter flask (no transfer needed)                                       |
| Titration technique                    | Potentiometric without temperature control or exclusion of atm CO <sub>2</sub> |
| Endpoint                               | pH = 7.00 ± 0.01 after 5 min equilibration (electrode stationary)              |

TABLE A-2. EQUIPMENT AND REAGENTS

| Minimum Qty      | Description   | Supplier, Cat. No.           |
|------------------|---|------------------------------|
| <b>Equipment</b> |   |                              |
| 1 roll           | Heavy duty aluminum foil or stainless steel surface   | 01-213-11                    |
| 1 pair           | Scissors or shears  | 14-275C                      |
| 1 pair           | Forceps, 300 mm in length   | 10-316C                      |
| 1                | Ruler, 12 in. (300 mm) hardwood or metal  | 12-090                       |
| 2 pairs          | Lint-free gloves  | 11-393-5                     |
| 1                | Analytical balance (accurate to 1 mg)   |                              |
| 100              | Aluminum weighing dishes  | 08-732                       |
| 1 pair           | Heat-resistant gloves   | Lab Safety Supply<br>H1955-2 |
| 1                | pH Meter (accurate to 0.01 pH units)  |                              |
| 2                | Combination pH electrode with AgCl reference and ceramic junction   |                              |
| 2                | Electrode storage containers  | 13-640-300                   |
| 1                | Rectangular porcelain stand   | 14-667                       |
| 1                | Double buret clamp for above  | 05-779                       |
| 2                | Buret, 10 mL graduated in 0.05 mL, Class A  | 03-700-22A                   |
| 1                | Buret, 25 mL or 50 mL graduates in 0.1 mL, Class A, 3-way stopcock optional                               | 03-724-10A                   |
| 3                | Beakers, 10 mL or buret covers  | 02-539C                      |
| 2 (optional)     | Aspirator bottle with tubulation, 1,000 mL (storage reservoir for H <sub>2</sub> SO <sub>4</sub> titrant) | 02-972D                      |
| 3                | Volumetric flasks, 1,000 mL, Class A  | 10-210G                      |
| 1                | Volumetric flask, 500 mL Class A  | 10-210F                      |
| 1                | Pipet, 1.00 mL, Class A   | 13-650-2B                    |
| 1                | Pipet, 4.00 mL, Class A   | 13-650-2E                    |
| 1                | Pipet, 10.00 mL, Class A  | 13-650-2L                    |
| 1                | Pipet, 100.00 mL, Class A   | 13-650-2U                    |
| 1                | Extension-type ring, 2–3 in. or clamp   | 14-055B                      |
| 2                | 3-prong clamp, 57 mm grip size  | 05-740                       |
| 3                | 3-prong clamp, 89 mm grip size  | 05-742                       |
| 6                | Clamp holders   | 05-757 or 05-759             |
| 1 or 2           | Ring stands   |                              |
| 1 or 2           | Lead bricks for above (or similar weighted object)  |                              |
| 1                | Hotplate/stirrer, Corning PC-351 or equivalent  | 11-495-50                    |
| 2                | Hotplates, Corning PC-35 or equivalent  | 11-495                       |
| 1                | Watch glass, 90-mm diameter   | 02-612C                      |
| 1                | Erlenmeyer flask, 2,000 mL  | 10-040M                      |

(continued)

TABLE A-2 (continued)

| Minimum Qty      | Description   | Supplier, Cat. No.                      |
|------------------|---|---|
| 1                | Neoprene or rubber stoppers #6 ½, with hole to fit Buchner funnel                     | 14-141H                                 |
| 2 (optional)     | #8, for aspirator bottle  | 14-141K                                 |
| 1                | #7, with hole to fit condenser  | 14-130L                                 |
| 1                | #7, with hole to fit tube below   | 14-130L                                 |
| 1                | 1/4 in. to 3/8 in. O.D. heavy-walled glass or metal tube approximately 5 to 6 in long |   |
| As needed        | Vacuum tubing   |   |
|                  | Rubber  | 14-173C                                 |
|                  | Tygon   | 14-169-2B                               |
| 1 (2nd optional) | Tubing connector  |   |
|                  | Y-type  | 15-320-10C                              |
|                  | T-type  | 15-319C                                 |
| 1                | Vacuum source (house vacuum, water aspirator, etc.)                                   |   |
| 1                | Buchner funnel for 7 cm filter paper  | 10-356C                                 |
| 1                | Box of filter paper, Whatman 41 (or 541), 7 cm  | 09-850A                                 |
| 1                | Forceps, 150 mm L   | 10-275                                  |
| 1                | Stopwatch or equivalent timer, accurate to 1 sec or better                            | 14-648 or 14-649-5                      |
| 1                | Spatula   |   |
| 2                | Wash bottles, polyethylene  | 03-409-10E or 03-409-22C                |
| 2                | Tall form beakers, 200 mL or 150 mL beakers   | 02-545B                                 |
| 1                | Graduated cylinder, 100 mL  | 08-549-5E                               |
| 9                | 250 mL Phillips beakers or 250 mL wide mouth flasks                                   | 02-566B                                 |
| 10               | Filter flasks, 250 mL   | 10-181D                                 |
| Various          | Assorted stir bars Teflon <sup>®</sup> coated or glass beads                          | 10-181D                                 |
| 1                | Magnetic stir bar \$2 in length, Teflon coated  | 14-511-65                               |
| 1                | Beaker, 400 mL  | 02-540L                                 |
| 1                | Water-cooled condenser, must be fitted to Phillips beakers or wide mouth flasks above | 07-723B                                 |
| 1                | Filter flask 500 mL   | 10-180E                                 |
| As needed        | Laboratory tissues and towels, sizes as available                                     |   |
| 1                | Box glassine envelopes, #7 4 1/8 in. × 6 1/4 in., ungummed flap                       | Worcester Envelope Co., #3525           |
| 1                | Face shield   | 11-409-5                                |
| 1                | Box hose clamps   | American Scientific Products, C6096-232 |

(continued)

| Minimum Qty     | Description  | Supplier, Cat. No.             |
|-----------------|--|--------------------------------|
| <b>Reagents</b> |  |                                |
| 1               | Case pH standard buffer solution, pH 7.00  | SB107-500                      |
| 1               | Case pH standard buffer solution, pH 10.00   | SB115-500                      |
| 1               | 0.0500 N, Sodium Hydroxide NaOH, Fisher CERTIFIED, C.A.S. Reg. 1310-73-2, 1-L bottle                         | SS278-1                        |
| 1               | 1.000 N, Sulfuric Acid, H <sub>2</sub> SO <sub>4</sub> , Fisher CERTIFIED, C.A.S. Reg. 7664-93-9, 1-L bottle | SA212-1                        |
| 1               | Potassium Chloride, CKI, Fisher CERTIFIED, Crystals, C.A.S. Reg. No. 7447-40-7, 500-g bottle                 | P-217-500                      |
| 1               | Hydrochloric Acid, HCl, ACS reagent, C.A.S. Reg. No. 7647-01-0 ½ gal bottle                                  | A-144-212                      |
|                 | Deionized water  | Laboratory Deionization System |

## A.4 Procedure

### A.4.1 Sample Preparation

Filters should be handled only with clean lint-free gloves or clean metal utensils such as forceps, tongs, scissors, etc.

- (1) After initial receipt, place the Hi-vol filters in a metal rack with separate slots for each filter. Place this rack in a controlled humidity (<50 percent relative humidity), controlled temperature (25 EC), balance room and allow the filters to equilibrate for 24-48 hours. At the end of that period, carefully weigh each filter using a balance adapted for this purpose. Weigh to the nearest milligram or better. Then place the filters in a storage container.
- (2) Remove one filter at a time from the storage container.
- (3) Place the filter on a clean metal surface (aluminum foil spread on a regular lab bench, clean stainless steel surface, etc.). Measure its length and width on all edges. Calculate the area by multiplying the average length by the average width. Cut out the filter number printed in the upper right corner, in a single rectangular piece. Save the number and place it in a glassine envelope.
- (4) Using a ruler, scissors, and lint-free gloves, cut the filter into ½ in. squares. This makes approximately 320 squares.
- (5) Mix the squares and randomly select a sample of 70-80 squares (approximately 1 g). Do not select and analyze the piece of filter containing the filter number. Weigh the filter sample in a tared disposable aluminum weighing dish. The final sample weight must be within one square of 1 g. Record the weight to the nearest milligram.
- (6) Store the remaining squares in a glassine envelope with the filter number showing. NOTE: Filters may be cut a few days before analysis and stored in glassine envelopes.
- (7) Transfer the weighed sample into a 250-mL Phillips beaker or a 250-mL wide-mouth flask. Cover the beaker or flask with the aluminum weighing dish and seal.

- (8) Clean the gloves, utensils and work area of any filter fibers or lint before taking out the next filter.
- (9) Repeat Steps 2-8 until seven Phillips beakers containing filter squares have been prepared. Leave two Phillips beakers empty as blanks. Label the Phillips beakers and 250-mL filter flasks consecutively from 1 to 9 and pair by number for each sample or blank.

#### A.4.2 Solution Preparation

- (1) Prepare 0.005 N NaOH titrant by diluting a certified 0.0500 N solution 1:10 with degassed deionized water (i.e., pipet 100.0 mL of certified NaOH into a 1,000-mL volumetric flask, dilute to the mark, and mix well). Degas the deionized water by bubbling  $N_2$  through 1 L of water at a rate of approximately 1 L/min for 1 hour. Take care to protect the solution from the air during storage in order to prevent carbonate formation.
- (2) Prepare 0.0100 N  $H_2SO_4$  titrant by diluting certified 1.000 N  $H_xSO_4$  titrant by diluting certified 1.000 N  $H_2SO_4$  solution 1:100 with deionized water (i.e., pipet 10.00 mL into a 1,000-mL volumetric flask, dilute to the mark with deionized water, and mix well).
- (3) Prepare 0.1 M KCl/0.0001 M HCl electrode storage solution by the following process. First, pipet 1 mL of reagent grade concentrated HCl into a 500-mL volumetric flask, dilute to the mark with deionized water, and mix well. Second, place 7.456 g of KCl in a 1,000-mL volumetric flask, add approximately 500 mL deionized water, swirl to dissolve the KCl crystals, pipet 4 mL of the diluted HCl solution into the flask, dilute to the mark with deionized water, and mix well. The solution may be stored in the volumetric flask or similar glass container.

#### A.4.3 General Notes and Pre-run Setup

- (1) Set up the titration stand and burets well in advance of sample analysis. Place one buret clamp on the titration stand and adjust the clamp height. In the right-hand buret holder, place a 10-mL buret. Check to see that the stopcock is closed. Fill the buret with 0.005 N NaOH solution and place a small beaker or buret cover on top of the buret:

#### CAUTION

The NaOH solution should not be stored in the buret for any length of time due to its tendency to form carbonates when exposed to the air.

Place the other buret in the other side of the holder, fill the buret with 0.01 N  $H_2SO_4$ , and cover the top of the buret with a small beaker or buret cover.

- (2) (Optional: If using buret with 3-way stopcock.) Attach one end of a length of acid-resistant, flexible tubing to the curved tube, A, at the bottom of the buret and connect the other end to a 3-way tubing connector. Securely fasten both ends using hose clamps. Attach the other two branches of the tubing connector with flexible tubing to the tubes at the base of the aspirator (reservoir) bottles. If a single reservoir bottle is used, curved tube A may be attached directly by a single line. Again, secure the connections with hose clamps. Check the

buret stopcock and make sure it is closed. Fill the reservoir bottle(s) with 0.0100 N H<sub>2</sub>SO<sub>4</sub> and fill the buret by adjustment of the 3-way stopcock to the proper position. During analysis, cap the reservoirs with beakers. For overnight and longer storage, stopper the bottles with the appropriate sized stoppers. A regular 25- or 50-mL buret may also be used with a Nalgene self zero buret kit #364-2550.

- (3) The filtration system should also be set up in advance. Clamp a 500-mL filter flask (serves as a trap) to a ring stand stabilized by a lead brick. Attach a piece of vacuum tubing to the vacuum source and to a 3-way connector. To one branch of the connector attach a short piece of vacuum tubing leaving the end open. Another piece of vacuum tubing must connect the remaining branch of the connector with the side arm of the 500-mL filter flask. Insert a 5-6 in. heavy-walled tube through the one-hole #7 stopper. Seat the stopper with tube in the flask and attach a piece of vacuum tubing to the tube. Connect an extension ring support to the stand (used for holding the Buchner funnel when not in use). Insert the Buchner funnel through a #6.5 neoprene stopper, set it in the ring stand, and cover the funnel with a 90-mm or larger watch glass.
- (4) Insert the lower end of a condenser into a filter adapter or stopper that fits the Phillips beakers snugly. Connect the water supply to the bottom side arm of the condenser using a length of tubing. Also attach a drainage line (which runs to a sink) to the upper side arm of the condenser.

#### **CAUTION**

Securely fasten all connections using hose clamps, condenser hose clips or wire, to prevent the hoses from coming loose, causing flooding (especially dangerous around electrical equipment).

Clamp the condenser to a ring stand, turn the water on, and adjust the flow rate.

- (5) At the beginning of each day's analysis, fill the 2,000-mL Erlenmeyer flask with deionized water, add a stir bar, place a watch glass over the mouth of the flask, and set the flask on a hotplate.

#### **WARNING**

A stir bar or glass bead must be placed in the flask to prevent violent bumping.

Set the heat on high until the water begins to boil. After boiling starts, turn the heat down sufficiently to just maintain boiling. Also, at this time set a hot plate/stirrer by the ring stand with the condenser and adjust the heat setting to high. Set the third hotplate (to be used during the cleanup step) in a convenient place and set on high heat. Generally, a 45- to 60-min warmup period is required for the water and hotplates.

#### **A.4.4 pH Meter Calibration and Electrode Storage**

- (1) Calibrate the pH meter at the beginning and end of a run as described in the instrument manual and record the calibration data.
- (2) Store electrodes in the 0.1 M KCl/0.0001 M HCl solution described in Section 4.2, Step 3. For short-term storage and between samples the electrode may be placed in a beaker containing approximately 100 mL of storage solution. For long-term storage (weekend or longer) slip the electrode into an O-ring sealed electrode storage bottle approximately three-fourths full of storage solution and tighten the cap to seal the electrode body to the bottle.

#### A.4.5 Analysis of Glass Fiber Filters

**WARNING**

Protective equipment must be worn to prevent serious burns.  
The sample is hot and caustic and will adhere to the skin.

- (1) Clamp a clean, 250-mL filter flask to the filtration stand and connect the tubing from the vacuum trap to the flask side arm. Set the Buchner funnel on top of the filter flask, place a single 7-cm Whatman 41 or 541 filter circle in the funnel using tweezers, and cover the funnel.
- (2) Set the first Phillips beaker, containing filter squares, near the hotplate/stirrer and add a 2-in. magnetic stir bar.
- (3) Pour 100 mL of boiling water from the 2-L flask into a graduated cylinder. (Use heat-resistant gloves to handle the hot glassware.)
- (4) Set aside the aluminum weighing dish cover, place the Phillips beaker on the hotplate, and turn on the stirrer.
- (5) Add the 100 mL of water to the Phillips beaker, begin timing the extraction, quickly lower the condenser into the beaker and clamp loosely.
- (6) At approximately 19 minutes and 30 seconds after the start of the extraction, don the face mask, turn on the vacuum, and put on heat-resistant gloves.
- (7) At about 19 minutes and 55 seconds quickly lift the condenser out of the Phillips beaker and clamp in a raised position.

**NOTE**

The stirrer may be stopped at this time.

- (8) At exactly 20 minutes, using heat-resistant gloves, remove the Phillips beaker from the hotplate, swirl, and pour the contents rapidly, but carefully, through the Buchner funnel. Set the beaker on the bench and apply vacuum until the filter pad is just dry on top. The unconnected length of vacuum tubing may be used to regulate the vacuum strength by placing a finger over the hole in the tubing or by leaving it open.

- (9) Remove the vacuum tubing and the funnel from the filter flask. Pour the hot extract back into the Phillips beaker carefully washing down any residual pulp. Reclamp the filter flask, replace the funnel, and reattach the vacuum tubing. Swirl and pour the extract through the filter. Apply vacuum as needed.

**NOTE**

Steps 8 and 9 should be completed in 2 minutes or less. Typical times have been 1 minute and 30 seconds to 1 minute and 50 seconds.

- (10) Remove the fact shield, detach the vacuum tubing, set aside the Buchner funnel, place the filter flask on the titration stand, and remove gloves.

**NOTE**

Before titrating the extract just finished, a more efficient use of time may be made by starting the next sample at this point as follows: Remove the stir bar and wipe it clean of filter material adhering to it. Immediately start another extraction by performing Steps 2 through 5 on the next sample.

- (11) Rinse the pH electrode with deionized water, shake off excess, pat dry with a laboratory tissue, and place in the filter flask. Push pH button (meter in Manual Temperature Compensation mode, set to 25E) and take an initial pH reading; this reading should be between pH 9 and 11 for glass filters and between 7 and 9 for quartz. Swirl the flask and begin titration.
- (12) Titrate samples with 0.0100 N H<sub>2</sub>SO<sub>4</sub>. Add the titrant in increments of several milliliters at a time until the pH drops below 8. At that point slow the addition rate to smaller increments of 0.1–0.5 mL of titrant. Stop the titrant flow when the pH reaches 7.00 and stop swirling; the pH should rise by 0.1–0.2 units. If the final calm pH is greater than 7.00, add enough titrant to compensate. If the titration cannot be stopped at pH 7.00 ±0.02, record the calm pH and the volume of titrant for points around pH 7 and calculate the extract volume of titrant required to reach pH 7 by linear regression or extrapolation methods.

**A.4.6 Analysis of Quartz Fiber Filters**

In order to reduce background alkalinity retained in the funnel, rinse the Buchner funnel once with boiling water before each analysis of a quartz filter. The alkalinity of quartz filters is more than an order of magnitude lower than that of glass filters; therefore, a lower background is required.

- (1) Clamp in place a clean, dry, 250-mL filter flask and a 250-mL filter flask labeled “rinse.”
- (2) Seat the funnel on the rinse flask.

- (3) Fill a graduated cylinder with 100 mL boiling deionized water, pour the water through the funnel and cover the funnel with a watch glass.
- (4) through (9)  
Same as Steps 1-5 for glass fiber filters.
- (10) During the extraction, remove the cover from the funnel, pat the funnel bowl and tip dry with laboratory tissues, move the funnel to the clean dry flask, using forceps place a 7 cm Whatman 41 or 541 filter circle in the funnel and recover. Remove the rinse flask and drain.
- (11) through (17)  
Same as Steps 6-12 for glass fiber filters.

#### A.4.7 Analysis of Blanks

Blank flasks are empty flasks that do not contain any filter squares.

- (1) through (11)  
Same as Steps 1-11 for glass fiber filters.
- (12a) FOR BASIC BLANKS: If the initial pH is above 7, titrate slowly with the  $\text{H}_2\text{SO}_4$  used for samples. See Step 12 for glass fiber filters.
- (12b) FOR ACIDIC BLANKS: If the initial pH is below 7, titrate slowly with the 0.005 N NaOH from a 10-mL buret, instead of  $\text{H}_2\text{SO}_4$ . Slow the titrant flow when the pH goes above 6 and stop the flow when pH reaches 6.8–6.9; when swirling is ceased the pH should rise 0.1 to 0.2 units. See Step 12 for glass fiber filters.

#### A.4.8 Cleanup after Analysis

- (1) Wipe and rinse the stir bars, then place in a clean dry place.
- (2a) FOR GLASS FILTERS: Get the next clean filter flask, mount it on the ring stand, and attach the vacuum line. Remove the used filter with forceps, clean the funnel with laboratory tissues, seat it on the filter flask, add a fresh Whatman 41 or 541 filter with forceps, and cover the funnel.
- (2b) FOR QUARTZ FILTERS: Mount the next filter flask and the rinse flask on the ring stand. Attach the vacuum line to the filter flask. Remove the used filter with forceps, clean the funnel with laboratory tissues, seat the funnel on the rise flask and cover.
- (3) After each titration, rinse the pH electrode into a waste beaker, then with a laboratory tissue wipe off any condensation on the electrode, and place it in the storage beaker.
- (4) As time permits, rinse the Phillips beakers and filter flask thoroughly with a stream of deionized water. Then fill each twice with fresh deionized water discarding the water each time. Fill each a third time with fresh deionized water, add a small Teflon-coated stir bar or clean glass bead and place on a hotplate to boil (as time and space permit).

**WARNING**

Be sure to use heat-resistant gloves or tongs when handling the hot glassware to prevent burns.

After boiling for at least 5 minutes, discard the water, taking care not to lose the stir bar, drain, and allow to dry thoroughly before reusing. Once per week, or more often as needed, scrub the sides and spout of the Phillips beaker with a clean brush, then rinse thoroughly and boil as above. Store the flask in a clean dry place.

**A.5. Calculations**

- (1) If the titration was not stopped at exactly pH 7.00, then the titrant volume corresponding to pH 7.00 is calculated by extrapolation or linear regression.
- (2) Calculate the microequivalents for the samples and the blanks using the following equation:

$$\text{microequivalents} = (\text{mL of titrant}) \times (\text{normality of titrant}) \times 1,000.$$

The sign of the blank (or sample) is negative if the titrant was NaOH and positive if the titrant was H<sub>2</sub>SO<sub>4</sub>.

- (3) Next average the blanks for the day:

$$\text{average blank} = \frac{\text{blank}_1 + \text{blank}_2}{2} \text{ (microequivalents)}$$

- (4) Then calculate alkalinity in microequivalents per gram of sample using the following equation:

$$\frac{\mu\text{eq}}{\text{g}} = \frac{\mu\text{eq of sample} - \mu\text{eq of average blank}}{\text{gram weight of sample}}$$

- (5) If units of microequivalents per square centimeter are required:

$$\frac{\mu\text{eq}}{\text{cm}^2} = \frac{\mu\text{eq}}{\text{g}} \times \frac{\text{weight of entire filter (g)}}{\text{surface of entire filter (cm}^2\text{)}}$$

- (6) Sample calculation

A filter weighing 4.0432 g was found to have an area of 517.2 cm<sup>2</sup>. A 1.005-g filter sample was extracted and the extract titrated with 0.0100 N H<sub>2</sub>SO<sub>4</sub>. The volume 10.99 mL of titrant corresponded to pH 7.08 and 11.12 mL of titrant corresponded to pH 6.92. Both blanks required H<sub>2</sub>SO<sub>4</sub> and had values of 1.0 and 0.8 microequivalents, respectively. Alkalinity is calculated as follows:

Step 1 using linear regression:

The estimated volume of titrant at pH 7.00 in this case equals 11.06 mL; or

Step 1 using extrapolation

The estimated volume of titrant also equals 11.06 mL.

Step 2

$$\begin{aligned}\mu\text{eq} &= (11.06 \text{ mL}) \times (0.0100 \text{ N}) \times 1,000 \\ &= 110.6 \text{ microequivalents}\end{aligned}$$

$$\begin{aligned}\text{Step 3} \\ \text{average blank} &= \frac{1.0 + 0.8}{2} = 0.9 \mu\text{eq} \\ \text{Step 4} \\ \text{alkalinity } (\mu\text{eq/g}) &= \frac{(110.6 - 0.9) \mu\text{eq}}{1.005 \text{ g}}\end{aligned}$$

Step 5

$$\text{alkalinity } \frac{\mu\text{eq}}{\text{cm}^2} = \frac{4.0432 \text{ g}}{517.2 \text{ cm}^2} \times \frac{109.2 \mu\text{eq}}{\text{g}} = 0.854 \frac{\mu\text{eq}}{\text{cm}^2}$$

## A.6 Quality Control

Prepare a total of nine Phillips beakers for analysis each day; leave two empty as blanks. The sample analysis order is: two samples, one blank, three samples, one blank, two samples. Two samples should be from filters previously analyzed. Number the Phillips beakers and filter flasks from 1 to 9 and rotate the position each day so all glassware pairs eventually are used for a blank analysis. An example of the glassware and sample order for a week is given in Table A-3.

**TABLE A-3. GLASSWARE AND SAMPLE ORDER**

| Day 1 |        | Day 2 |        | Day 3 |        | Day 4 |        | Day 5 |        |
|-------|--------|-------|--------|-------|--------|-------|--------|-------|--------|
| Glass | Sample |
| 1     | S-1    | 2     | S-8    | 3     | S-13   | 4     | S-18   | 5     | S-23   |
| 2     | S-2    | 3     | S-9    | 4     | S-14   | 5     | S-19   | 6     | S-24   |
| 3     | B-1    | 4     | B-3    | 5     | B-5    | 6     | B-7    | 7     | B-9    |
| 4     | S-3    | 5     | S-10   | 6     | S-15   | 7     | S-20   | 8     | S-25   |
| 5     | S-4    | 6     | S-1    | 7     | S-9    | 8     | S-16   | 9     | S-18   |
| 6     | S-5    | 7     | S-11   | 8     | S-16   | 9     | S-21   | 1     | S-26   |
| 7     | B-2    | 8     | B-4    | 9     | B-6    | 1     | B-8    | 2     | B-10   |
| 8     | S-6    | 9     | S-12   | 1     | S-17   | 2     | S-22   | 3     | S-27   |
| 9     | S-7    | 1     | S-5    | 2     | S-1    | 3     | S-13   | 4     | S-4    |

