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AP-42 Section 11.28
Reference 8
Report Sect. BR
Reference 9



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COMMONWEALTH of VIRGINIA

State Air Pollution Control Board

ROOM 1106, NINTH STREET OFFICE BUILDING
RICHMOND, VIRGINIA 23219
TELEPHONE: (804) 786-2378

W. R. MEYER
EXECUTIVE DIRECTOR

July 7, 1980

Mr. Robert L. Sansom
Virginia Vermiculite, Ltd.
Energy and Environmental Analysis, Inc.
General Partner
1111 N. 19th Street, Suite 600
Arlington, Virginia 22049

Dear Mr. Sansom:

Review of the particulate and asbestos emissions test performed by RTP Environmental Associates on October 27, 1979 indicates that your facility is in compliance with the particulate emissions requirements of the Virginia State Air Pollution Control Board. Tests done by Walter C. McCrone Associates, Inc. also indicate that no asbestiform minerals were present in those ore samples taken at the plant.

Thank you for your patience in waiting for our official report on these tests.

Sincerely,

William M. Jewell, Jr.
Director, Division of Compliance

WMJ/RCJ/jld

cc: Director, Region IV



RTP Environmental Associates

Air Measurement and Control

P.O. Box 12564

Research Triangle Park, N.C. 27709

Telephone (919) 471-8366

ROTARY DRYER
PARTICULATE EMISSIONS TESTING

PERFORMED FOR:

VIRGINIA VERMICULITE LIMITED

BOSWELL'S TAVERN, VIRGINIA

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INTRODUCTION

The Virginia Vermiculite mining operation in Boswell's Tavern, Virginia, employs a rotary dryer to burn and evaporate the flotation medium used to separate vermiculite from undesirable mica and silicate deposits. The exhaust gases from the rotary dryer are drawn by an induced fan through a low efficiency cyclone in series with a cylindrical water spray tower with a wire mesh demister; combustion waste gases are then vented to the atmosphere.

RTP Environmental Associates performed particulate and asbestos emissions testing at the waste gas stack of this rotary dryer. The testing objectives were to determine the total process particulate emissions on a lbs/hr basis and qualitative asbestos measurements of the particles emitted from the rotary dryer. Three samples for asbestos determination were collected from three locations: (1) raw ore feed into plant, (2) vermiculite feed into dryer, and (3) rotary dryer exhaust. These three samples will be analyzed by Walter C. McCrone Associates, Inc. All particulate emissions procedures compiled with the August 18, 1977 Federal Reference Method 5 and the April 21, 1977 Commonwealth of Virginia testing requirements.

Test procedures at Virginia Vermiculite were observed by representatives of the Virginia State Air Pollution Control Board. These representatives were Ward Butler, Air Pollution Control Office; Nick Buckholtz, Division of Compliance; and Renaldo Jenkins, Engineering Division.

SUMMARY OF RESULTS

The Reference Method 5 procedures given in the August 18, 1977 Federal Register has been adopted as the particulate compliance testing method in the State of Virginia. Confirmation of the approved methodology was made through Mr. Thomas Creasy of the Virginia State Air Resources Board.

The rotary dryer process rate varied throughout the testing regime. A summary of the process rates occurring during the compliance testing is shown below.

TABLE 1
PROCESS FEED RATES

<u>Time</u>	<u>Process Rate</u> <i>lb/hr</i>
11:30	6.62
12:00	8.27
12:30	9.45
1:00	8.27
1:30	6.62
2:00	7.35
2:30	8.27
3:00	11.03
3:30	8.27
4:00	9.45

8.36 AVG

Virginia State particulate emission compliance regulations state that particulate emissions must not exceed a specified limit "E" calculated from the equation $E = 4.10P^{0.67}$ where E = emission rate in lbs/hr and P

= process weight rate in tons/hr (this equation is valid up to a process weight rate of 30 tons/hr). The average process feed rate during each particulate test and its representative particulate emission limit are given in Table 2.

TABLE 2
PARTICULATE EMISSION LIMITS

<u>Test Number</u>	<u>Process Rate (Ton/hr)</u>	<u>Emission Limit (lb/hr)</u>
1	8.11	16.7
2	7.41	15.7
3	9.19	18.1

Table 3 gives the individual test summaries for the particulate compliance tests performed on the dryer exhaust stack.

Visible emissions determinations were attempted by Ward Butler of the State Air Pollution Control Board and Robert Y. Purcell of RTP Environmental Associates. Both personnel agreed that the existing meteorological conditions prevented an accurate estimate.

TABLE 3
SUMMARY OF TEST RESULTS

TEST*	1	2	3	
Date	10/27/79	10/27/79	10/27/79	
Stack Temperature (°F)	131	133	130	12
Percent Isokinetic (%)	103.5	92.0	101.3	93.1
Stack Flowrate (SDCFM)**	10,764	11,202	10,123	10696.5
Gas Sampled (SDCF)**	46.8	43.3	43.1	44.9
Filter Catch (mg)	49.3	47.1	46.5	47.6
Particulate Emissions (lbs/hr)	1.50	1.61	1.44	1.51 _{AV}

*TEST 1: Particulate Emissions Test (11:20-12:34)

TEST 2: Particulate Emissions Test (13:12-14:21)

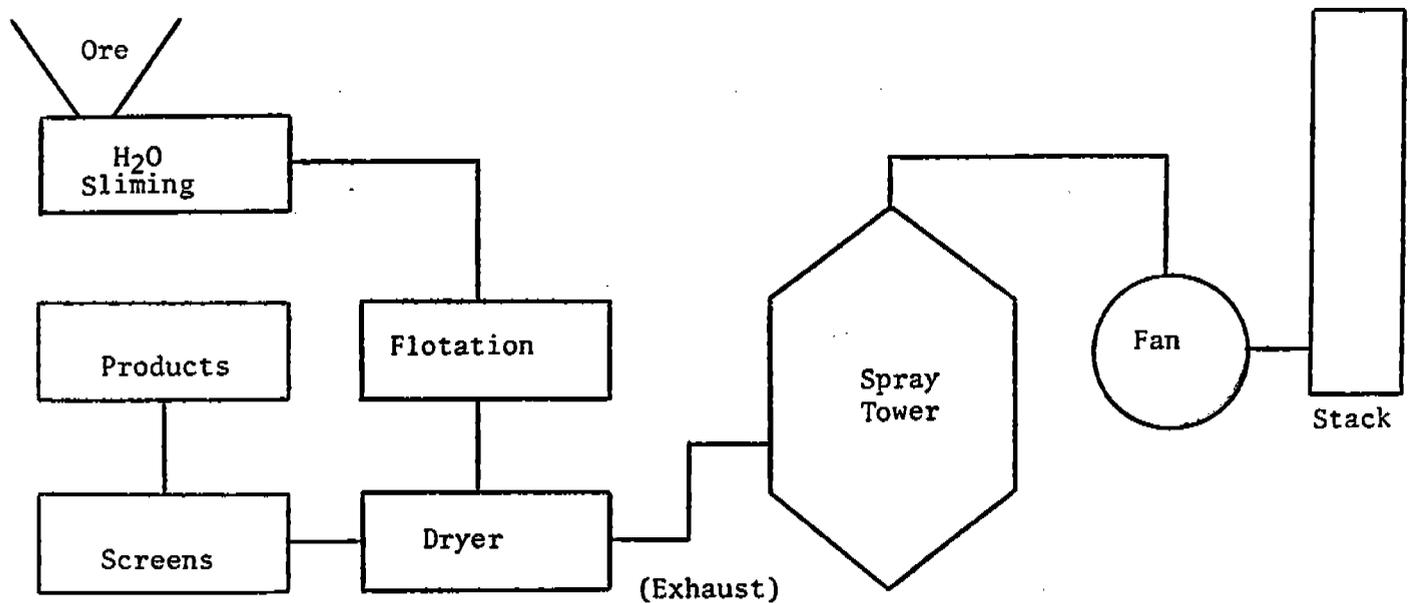
TEST 3: Particulate Emissions Test (14:48-15:54)

**68°F, 29.92 in. Hg

PROCESS DESCRIPTION

The Virginia Vermiculite mining operation removes clay, mica, and silicate deposits from vermiculite laden ore at a site in Boswell's Tavern, Virginia. Clay deposits are removed by a water sliming procedure; the mica and silicates are separated from the vermiculite by flotation in a mixture of #2 fuel oil and other chemicals. The vermiculite is then dried in the rotary dryer and divided into two commercial grades.

Particulate emissions from the rotary dryer are controlled by a low energy water spray tower. The pressure drop across the tower is less than five inches of water. A wire demister prevents excess entrained water from being emitted from the tower. The following block diagram illustrates the process operation.



The rotary dryer normally operates at a process feed rate of eight tons per hour. The maximum operating capacity is rated at 10 tons per hour, but numerous difficulties arise as this feed rate is maintained. The dryer utilizes #2 fuel oil as a fuel; this fuel is rated at 138,000 BTU per gallon with a 0.3 percent sulfur content. The normal operating fuel useage is 60 gallons per hour with a maximum useage of 75 gallons per hour.

TESTING METHODOLOGY

All sampling and analytical procedures are those recommended by the United States Environmental Protection Agency and the Commonwealth of Virginia. Complete details of the equipment and procedures used are described in Appendix C.

Method 1 was used to determine the number and location of the sampling points. The dryer exhaust stack was divided into 12 equal areas. Three ports were cut into the stack (see Figure 1 and 2) so that the center of each area could be tested. Every point was sampled for five minutes, giving a total testing time of 60 minutes for each test run on the stack.

Velocity measurements were made in accordance with Method 2. Molecular weights for the stack gases were determined by Method 3 procedures. Particulate emissions were determined using Method 5 analytical and sampling procedures.

The two process feed samples (asbestos determination) were collected via intermittent grab samples, while the dryer exhaust asbestos sample was collected using the EPA Method 5 sampling train with a 0.5 μm Millipore filter. These samples were sent to Walter C McCrone Associates, Inc. for sample analysis. The results of these analyses will be sent as a separate enclosure with this report.

All sampling equipment used was manufactured by Nutech Corporation or RTP Environmental Associates.

Method 5 Particulate Test - #1

Calculation Form

I. Necessary Data

- A. No. of Traverse Points 12
- B. Total Test Time (t) 60 minutes
- C. Water Collected
1. Impinger H₂O 105 ml
2. Silica Gel 12 gm
- D. Particulate Weight (m_n) 0.0493 gm
- E. Volume Metered
- V_m = 47.164 CF X DG MCF = 47.164 CF
- F. Average $\sqrt{\Delta P}$ 1.638 in H₂O
- G. Average ΔH 1.975 in H₂O
- H. Average Meter Temperature T_m 79 °F + 460 = 539 °R
- I. Average Stack Temperature T_s 131 °F + 460 = 591 °R
- J. Stack Absolute Pressure 30.21 in. Hg
- K. Barometric Pressure 30.17 in. Hg
- L. %CO₂ 1.0 ; %O₂ 18.0 ; %CO 0 ; %N₂ 81.0 ;
- M. Area of Stack 2.25 ft.²
- N. Area of Nozzle 1.5763x10⁻⁴ ft.²

II. Calculations

A. Standard Volume Metered

Y = Dry Gas meter calibration factor

$$V_{m(\text{std})} = V_m Y \frac{T_{\text{std}}}{P_{\text{std}}} \frac{P_b + \frac{\Delta H}{13.6}}{T_m}$$

$$V_{m(\text{std})} = (47.164 \text{ CF}) \left(\frac{528^\circ \text{R}}{29.92 \text{ in. Hg}} \right) \left(\frac{30.17 + 0.1452 \text{ in. Hg}}{539^\circ \text{R}} \right) = 46.812 \text{ dscf}$$

B. Moisture Content of Stack Gas

1. H₂O collected in impingers in standard cubic feet

$$V_{wc(std)} = K (V_f - V_i)$$

$$V_{wc(std)} = 0.04707 \text{ ft.}^3/\text{ml} \left(\underline{105} \text{ ml} \right) = \underline{4.9424} \text{ scf}$$

2. H₂O collected in Silica Gel in standard cubic feet

$$V_{wsg(std)} = K (W_f - W_i)$$

$$V_{wsg(std)} = 0.04715 \text{ ft.}^3/\text{gm.} \left(\underline{12} \text{ gm} \right) = \underline{0.5658} \text{ scf}$$

3. Moisture Content of Stack Gas (B_{ws})

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}}$$

$$B_{ws} = \frac{(\underline{4.9424} \text{ scf}) + (\underline{0.5658} \text{ scf})}{(\underline{4.9424} \text{ scf}) + (\underline{0.5658} \text{ scf}) + (\underline{46.812} \text{ scf})} = \underline{0.1053}$$

C. Molecular Weight of Stack Gas (1B/1B-mole)

1. M_d (Dry Molecular Weight) = $\sum M_x B_x$

$$M_d = (44) \underline{1.0} \% \text{CO}_2 + (32) \underline{18.0} \% \text{O}_2 + (28) \underline{0} \% \text{CO} + (28) \underline{81.0} \% \text{N}_2 = \underline{28.880} \text{ 1B/1B-mole}$$

2. M_s (Wet Molecular Weight) = M_d (1-B_{ws}) + 18 B_{ws}

$$M_s = \underline{28.880} (1 - \underline{0.1053}) + 18(\underline{0.1053}) = \underline{27.735} \text{ 1B/1B-mole}$$

D. Average Stack Gas Velocity

$$\bar{V}_s = K C_p \left[\frac{T_s}{P_s M_s} \right]^{1/2} \left[\sqrt{\Delta P} \right]_{AVE}$$

$$\bar{V}_s = 35.48 \text{ ft./sec.} \left[\frac{\text{1B/1B-mole(in.Hg)}}{\text{°R(in.H}_2\text{O)}} \right]^{1/2} \frac{0.84}{\left[\frac{(\underline{591})^\circ\text{R}}{(\underline{30.2})\text{in.Hg}(\underline{27.735})\text{1B/1B-mole}} \right]^{1/2}} \left[\underline{1.638} \text{ in H}_2\text{O} \right] = \underline{98.79} \text{ ft./sec}$$

E. Average Stack Gas Volumetric Flow Rate

$$\bar{Q}_s = (3600 \text{ sec/hr})(\bar{V}_s)(A_s)(1-B_{ws}) \frac{T_{std}}{P_{std}} \frac{P_s}{T_s}$$

$$\bar{Q}_s = (3600 \text{ sec/hr})(98.79 \text{ ft./sec})(2.25 \text{ ft.}^2)(1-0.1053) \frac{528^\circ\text{R}}{29.92 \text{ in.Hg}}$$

$$\left[\frac{30.21 \text{ in.Hg}}{591^\circ\text{R}} \right] = \frac{645,819}{\text{ft.}^3/\text{hr}} \text{ DEL STANDARD CONDITIONS}$$

F. Pollutant Mass Rate

$$\text{PMR} = \left[\frac{M_n}{V_m(\text{std})} \right] \times Q_s$$

$$\text{PRM} = \frac{(0.0493) \text{ gm}}{46.812 \text{ dscf}} \times 645,819 \text{ dscf/HR} \times \frac{1}{454 \text{ gm/lb}} = 1.4981 \text{ lb/HR}$$

G. % Isokinetic Variation (Intermediate Data)

$$\%I = \frac{T_s V_m(\text{std}) P_{std}}{\theta V_s A_n P_s T_{std} (60)(1-B_{ws})} \times 100$$

$$\%I = \frac{(591^\circ\text{R})(46.812 \text{ dscf})(29.92 \text{ in.Hg}) \times 100}{(1.5763 \text{ ft.}^2) \times 10^{-4} (60 \text{ min.})(98.79 \text{ ft./sec.})(30.21 \text{ in.Hg})(528^\circ\text{R})(60 \text{ sec./min.})(1-0.1053)}$$

$$= 103.46$$

Method 5 Particulate Test - #2

Calculation Form

I. Necessary Data

A. No. of Traverse Points 12

B. Total Test Time (t) 60 minutes

C. Water Collected

1. Impinger H₂O 86 ml

2. Silica Gel 10 gm

D. Particulate Weight (m_n) 0.0471 gm

E. Volume Metered

$$V_m = \underline{44.256} \text{ CF} \times \text{DGMCF} = \underline{44.256} \text{ CF}$$

F. Average $\sqrt{\Delta P}$ 1.692 in H₂O

G. Average ΔH 1.724 in H₂O

H. Average Meter Temperature T_m 86 °F + 460 = 546 °R

I. Average Stack Temperature T_s 133 °F + 460 = 593 °R

J. Stack Absolute Pressure 30.21 in. Hg

K. Barometric Pressure 30.17 in. Hg

L. %CO₂ 1.2; %O₂ 18.5; %CO 0; %N₂ 80.3;

M. Area of Stack 2.25 ft.²

N. Area of Nozzle 1.5763x10⁻⁴ ft.²

II. Calculations

A. Standard Volume Metered

Y = Dry Gas meter calibration factor

$$V_{m(\text{std})} = V_m Y \frac{T_{\text{std}}}{P_{\text{std}}} \frac{P_b + \frac{\Delta H}{13.6}}{T_m}$$

$$V_{m(\text{std})} = (44.256 \text{ CF}) \left(\frac{528^\circ\text{R}}{29.92 \text{ in.Hg}} \right) \left(\frac{30.17 + 0.1268 \text{ in.Hg}}{546^\circ\text{R}} \right) = \underline{43.336} \text{ dscf}$$

B. Moisture Content of Stack Gas

1. H₂O collected in impingers in standard cubic feet

$$V_{wc(std)} = K (V_f - V_i)$$

$$V_{wc(std)} = 0.04707 \text{ ft.}^3/\text{ml} (\underline{86} \text{ ml}) = \underline{4.0480} \text{ scf}$$

2. H₂O collected in Silica Gel in standard cubic feet

$$V_{wsg(std)} = K (W_f - W_i)$$

$$V_{wsg(std)} = 0.04715 \text{ ft.}^3/\text{gm.} (\underline{10} \text{ gm}) = \underline{0.4715} \text{ scf}$$

3. Moisture Content of Stack Gas (B_{ws})

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}}$$

$$B_{ws} = \frac{(\underline{4.0480} \text{ scf}) + (\underline{0.4715} \text{ scf})}{(\underline{4.0480} \text{ scf}) + (\underline{0.4715} \text{ scf}) + (\underline{43.336} \text{ scf})} = \underline{0.0944}$$

C. Molecular Weight of Stack Gas (lb/lb-mole)

1. M_d (Dry Molecular Weight) = Σ M_x B_x

$$M_d = (44) \underline{1.2} \% \text{CO}_2 + (32) \underline{18.5} \% \text{O}_2 + (28) \underline{0} \% \text{CO} + (28) \underline{70.3} \% \text{N}_2 = \underline{28.932} \text{ lb/lb-mole}$$

2. M_s (Wet Molecular Weight) = M_d (1-B_{ws}) + 18 B_{ws}

$$M_s = \underline{28.932} (1 - \underline{0.0944}) + 18(\underline{0.0944}) = \underline{27.900} \text{ lb/lb-mole}$$

D. Average Stack Gas Velocity

$$\bar{V}_s = K C_P \left[\frac{T_s}{P_s M_s} \right]^{1/2} \left[\sqrt{\Delta P} \right]_{ave}$$

$$\bar{V}_s = 35.48 \text{ ft./sec.} \left[\frac{\text{lb/lb-mole (in.Hg)}}{^\circ\text{R (in.H}_2\text{O)}} \right]^{1/2} \frac{0.84}{\left[\frac{(\underline{593})^\circ\text{R}}{(\underline{30.121} \text{ in.Hg}) (\underline{27.9} \text{ lb/lb-mole})} \right]^{1/2}} \left[\underline{1.692} \text{ in H}_2\text{O}^h \right] = \underline{101.92} \text{ ft./sec}$$

E. Average Stack Gas Volumetric Flow Rate

$$\bar{Q}_s = (3600 \text{ sec/hr})(\bar{V}_s)(A_s)(1-B_{ws}) \frac{T_{std}}{P_{std}} \frac{P_s}{T_s}$$

$$\bar{Q}_s = (3600 \text{ sec/hr})(\underline{101.92 \text{ ft./sec}})(\underline{2.25 \text{ ft.}^2})(1-\underline{0.0944}) \frac{528^\circ\text{R}}{29.92 \text{ in.Hg}}$$

$$\left[\frac{\underline{30.21 \text{ in.Hg}}}{\underline{593}^\circ\text{R}} \right] = \underline{672.124} \text{ (ft.}^3\text{/hr.) DEL STANDARD CONDITIONS}$$

F. Pollutant Mass Rate

$$\text{PMR} = \left[\frac{M_n}{V_m(\text{std})} \right] \times Q_s$$

$$\text{PRM} = \frac{(\underline{0.0471}) \text{ gm}}{\underline{43.336} \text{ dscf}} \times \underline{672.124} \text{ dscf/HR} \times \frac{1}{454 \text{ gm/lb}} = \underline{1.6090} \text{ lb/HR}$$

G. % Isokinetic Variation (Intermediate Data)

$$\%I = \frac{T_s V_m(\text{std}) P_{std}}{\theta V_s A_n P_s T_{std} (60)(1-B_{ws})} \times 100$$

$$\%I = \frac{(\underline{593}^\circ\text{R})(\underline{43.336} \text{ dscf})(\underline{29.92 \text{ in.Hg}}) \times 100}{(\underline{1.5763 \text{ ft.}^2}) \times 10^{-4} (\underline{60} \text{ min.})(\underline{101.92 \text{ ft./sec.}})(\underline{30.21 \text{ in.Hg}})(\underline{528^\circ\text{R}})(\underline{60 \text{ sec./min.}})(1-\underline{0.0944})} = 92.03$$

Method 5 Particulate Test- #3

Calculation Form

I. Necessary Data

- A. No. of Traverse Points 12
- B. Total Test Time (t) 60 minutes
- C. Water Collected
1. Impinger H₂O 110 ml
2. Silica Gel 13 gm
- D. Particulate Weight (m_n) 0.0465 gm
- E. Volume Metered
- $V_m = 44.658$ CF X $\frac{1.00}{DGMCF} = 44.658$ CF
- F. Average $\sqrt{\Delta P}$ 1.559 in H₂O
- G. Average ΔH 1.736 in H₂O
- H. Average Meter Temperature T_m 94 °F + 460 = 554 °R
- I. Average Stack Temperature T_s 130 °F + 460 = 590 °R
- J. Stack Absolute Pressure 30.21 in. Hg
- K. Barometric Pressure 30.17 in. Hg
- L. %CO₂ 1.2; %O₂ 18.3; %CO 0; %N₂ 80.5;
- M. Area of Stack 2.25 ft.²
- N. Area of Nozzle 1.5763x10⁻⁴ ft.²

II. Calculations

A. Standard Volume Metered

Y = Dry Gas meter calibration factor

$$V_{m(std)} = V_m Y \frac{T_{std}}{P_{std}} \frac{P_b + \frac{\Delta H}{13.6}}{T_m}$$

$$V_{m(std)} = (44.658 \text{ CF}) \left(\frac{528^\circ R}{29.92 \text{ in.Hg}} \right) \left(\frac{30.17 + 0.1276 \text{ in.Hg}}{554^\circ R} \right) = 43.099 \text{ dscf}$$

B. Moisture Content of Stack Gas

1. H₂O collected in impingers in standard cubic feet

$$V_{wc(std)} = K (V_f - V_i)$$

$$V_{wc(std)} = 0.04707 \text{ ft.}^3/\text{ml} (110 \text{ ml}) = 5.1777 \text{ scf}$$

2. H₂O collected in Silica Gel in standard cubic feet

$$V_{wsg(std)} = K (W_f - W_i)$$

$$V_{wsg(std)} = 0.04715 \text{ ft.}^3/\text{gm.} (13 \text{ gm}) = 0.6130 \text{ scf}$$

3. Moisture Content of Stack Gas (B_{ws})

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}}$$

$$B_{ws} = \frac{(5.1777 \text{ scf}) + (0.6130 \text{ scf})}{(5.1777 \text{ scf}) + (0.6130 \text{ scf}) + (43.099 \text{ scf})} = 0.1184$$

C. Molecular Weight of Stack Gas (1B/1B-mole)

1. M_d (Dry Molecular Weight) = $\sum M_x B_x$

$$M_d = (44) 1.2 \%CO_2 + (32) 18.3 \%O_2 + (28) 0 \%CO + (28) 80.5 \%N_2 = 28.924 \text{ 1B/1B-mole}$$

2. M_s (Wet Molecular Weight) = M_d (1-B_{ws}) + 18 B_{ws}

$$M_s = 28.924 (1 - 0.1184) + 18(0.1184) = 27.629 \text{ 1B/1B-mole}$$

D. Average Stack Gas Velocity

$$\bar{V}_s = K C_p \left[\frac{T_s}{P_s M_s} \right]^{1/2} \left[\sqrt{\Delta P} \right]_{ave}$$

$$\bar{V}_s = 35.48 \text{ ft./sec.} \left[\frac{1\text{B/1B-mole(in.Hg)}}{^\circ\text{R(in.H}_2\text{O)}} \right]^{1/2} \frac{0.84}{\left[\frac{(590)^\circ\text{R}}{(30.21\text{Hg})(27.629\text{1B/1B-mole})} \right]^{1/2}} \left[\frac{1.559 \text{ in H}_2\text{O}^3}{\text{}} \right] = 94.13 \text{ ft./sec}$$

E. Average Stack Gas Volumetric Flow Rate

$$\bar{Q}_s = (3600 \text{ sec/hr})(\bar{V}_s)(A_s)(1-B_{ws}) \frac{T_{std}}{P_{std}} \frac{P_s}{T_s}$$

$$\bar{Q}_s = (3600 \text{ sec/hr})(94.13 \text{ ft./sec})(2.25 \text{ ft.}^2)(1-0.1184) \frac{528^\circ\text{R}}{29.92 \text{ in.Hg}}$$

$$\left[\frac{30.21 \text{ in.Hg}}{590^\circ\text{R}} \right] = \frac{607373}{\text{ft.}^3/\text{hr.}} \text{ DEN. STANDARD CONDITIONS}$$

F. Pollutant Mass Rate

$$\text{PMR} = \left[\frac{M_n}{V_{m(\text{std})}} \right] \times Q_s$$

$$\text{PRM} = \frac{(0.0465) \text{ gm}}{43.099 \text{ dscf}} \times 607,373 \text{ dscf/HR} \times \frac{1}{454 \text{ gm/lb}} = 1.4434 \text{ lb/HR}$$

G. % Isokinetic Variation (Intermediate Data)

$$\%I = \frac{T_s V_{m(\text{std})} P_{std}}{\theta V_s A_n P_s T_{std} (60)(1-B_{ws})} \times 100$$

$$\%I = \frac{(590^\circ\text{R})(43.099 \text{ dscf})(29.92 \text{ in.Hg})}{\left(\frac{1.576 \text{ ft.}^2}{\times 10^{-4}} \right) (60 \text{ min.}) (94.13 \text{ ft./sec.}) (30.21 \text{ in.Hg}) (528^\circ\text{R}) (60 \text{ sec./min.}) (1-0.1184)}$$

$$= 101.29$$

APPENDIX B
FIELD AND ANALYTICAL DATA

Orsat Identification No. 1 Date Reagents Added 10/26/79
 Checked By DAVE BEACHLER
 Plant Location LOUISA, VIRGINIA Sampling Date 10/27/79
 Operator(s) R. PORCELL
 Sampling Location ROTARY DRYER EXHAUST Average Pyrite CO₂ —
 Moisture Content of Stack Gas (B_{WS}) ~ 10.9% Fuel Used #2 FUEL OIL
 Fuel Feed Rate 60 gal/hr Combustion Source Description ROTARY DRYER
 Process Production Rate ~ 8 ton/hr Steam Production Rate —
 Comments: —

Test No.	Sample Time		Analysis Time	Burette Readings		
	Start	Stop		CO ₂	O ₂	CO
1	11:29	12:30	12:35	1.0	19.0	—
2	13:12	14:20	14:30	1.2	19.5	—
3	14:48	15:55	16:00	1.2	19.7	—

Component	Mole Fraction = % Composition			
CO ₂	1.0	1.2	1.2	
O ₂ -CO ₂	18.0	18.3	18.5	
CO-O ₂	0	0	0	
100-CO = N ₂ *	81.0	80.5	80.3	

Dry Molecular Weight of Stack Gas (M_d) = ΣM_xB_x

M_d = 44gm/m (ZCO₂) + 32gm/m (ZO₂) + 28gm/m (ZCO) + 28gm/m (ZN₂) = gm/m

Wet Molecular Weight of Stack Gas (M_w) = M_d(1-B_{WS}) + 18gm/m(B_{WS})

M_w = (gm/m) (1 -) + 18gm/m () = gm/m

% Excess Air in the Duct (ZEA) =
$$\frac{\% O_2 - 0.5(\% CO) \times 100}{(0.264)(\% N_2) - (\% O_2) + 0.5(\% CO)}$$

Z EA =
$$\frac{(18.3 ZO_2) - 0.5(0 ZCO) \times 100}{(0.264)(80.5 ZN_2) - (18.3 ZO_2) + 0.5(0 ZCO)} = 619.9 \%$$

* If CO reading is less than O₂ reading use O₂ reading for calculating ZN₂

PARTICULATE FIELD DATA

VERY IMPORTANT - FILL IN ALL PLACES

Plant Virginia Power Site Test Start Time: 1129
 Run No. 1 Stop Time: 1234
 Location Scrubber Stack Filter No. 108
 Date 10/24/79 Filter Tare Weight 0.4143 gm
 Operator JA Orsat No. 1
 Sample Box No. 8 Date Rebuilt 10/16/79
 Meter Box No. 7 Fyrite No. _____
 Meter AH₂ 1.63 Date Rebuilt _____
 C Factor 0.85 Nomogr. ID No. 509
 Ambient Temp. °F 60
 Bar. Press. in.Hg. 30.17
 Assumed Moisture % 10
 Heater Box Setting, °F 250-7
 Probe Tip Dia., In. 0.170
 Probe length 5 ft.
 Probe Heater Setting 7 - 250°F
 Avg. AD 2.4 Avg. SD 1.84
 Leak Rate - 150 In. 0.0 Leak Rate - 300 In. 0.0

Point	Clock Time	Flow Rate (m ³ /hr)	Filter	Orifice In.Hg.	Actual In.Hg.	Inlet Temp. °F	Outlet Temp. °F	Pump Vacuum In. Hg.	Dry Temp. °F	Wet Temp. °F	Wet Bulb Temp. °F	Wet Bulb Depression °F	Relative Humidity %
A-1	0	365.514	2.30	1.78	1.30	62	62	1	250	65	65	0.05	125
2	5	313.11	2.60	1.50	1.50	62	62	1	250	65	65	0.05	125
3	10	316.92	2.40	1.75	1.75	61	61	1	250	66	66	0.05	120
4	15	320.50	2.10	1.55	1.55	61	61	1	250	66	66	0.05	130
B-1	20	389.044	3.00	1.90	2.20	80	80	2	250	66	66	0.05	135
2	25	381.28	3.00	2.20	2.20	80	80	2	250	66	66	0.05	130
3	30	347.05	3.00	2.10	2.20	80	80	2	250	66	66	0.05	130
4	35	346.22	2.90	2.10	2.10	80	80	2	250	66	66	0.05	135
C-1	40	100.23	2.50	1.85	1.85	81	81	2	250	66	66	0.05	135
2	45	404.15	2.80	1.05	2.05	81	81	2	250	66	66	0.05	135
3	50	408.15	3.10	2.30	2.30	81	81	2	250	66	66	0.05	135
4	55	412.45	3.00	2.20	2.20	81	81	2	250	66	66	0.05	135
5	60	416.43	3.00	2.20	2.20	81	81	2	250	66	66	0.05	135
Volume = 47.164													
(VAR) = 1.63%													
AVG = 78.8													

Scrubber cleaned 10/21/79. First operation after cleaning

Test Observers: Ward Butler } Variable
 Nick Buckle }
 Rendell Senkin }

PARTICULATE SOURCE SAMPLE

Analysis Date(s) 10/27/79 - 10/29/79 Analytical Chemist E. Porcell

Plant Sampled VIRGINIA VERMICULITE LTD. Location LOUISA, VIRGINIA

Sampling Location ROTARY DRYER EXHAUST Sampling Date(s) 10/27/79

Sample Run No. 2 Sampling Case No. 9

Sample Labels

H₂O _____ Silica Gel _____ Filter _____ Probe _____

Impinger Rinse _____ Dry Particulate _____ Other _____

Analysis Performed _____

Reference Method _____ Analytical Sample Temp. 70 °F

Comments:

<p><u>Moisture Data</u></p> <p>Final Volume H₂O in Impingers <u>286</u> ml Initial Volume H₂O in Impingers <u>200</u> ml Volume H₂O condensed <u>86</u> ml</p> <p>Final Weight Silica Gel <u>210</u> gm Initial Weight Silica Gel <u>200</u> gm (1gmH₂O=1mlH₂O) Volume H₂O Absorbed <u>10</u> ml</p>	<p><u>Total Moisture</u></p> <p>H₂O Condensed <u>86</u> ml H₂O Absorbed <u>10</u> ml H₂O Total <u>96</u> ml</p>
<p><u>Particulate Data</u></p> <p>CH₃-CH₂-O-CH₂-CH₃/CHCL₃ extract Flask No: _____ Final Weight _____ mg Initial Weight _____ mg Organic Fraction _____ mg</p>	<p><u>Total Particulate Sampled</u></p> <p>Organic Fraction _____ mg Inorganic Fraction _____ mg Front Half Particulates _____</p>
<p>Extracted H₂O Flask No. _____ Final Weight _____ mg Initial Weight _____ mg Inorganic Fraction _____ mg</p>	<p><u>Total Particulates</u></p> <p>Run No. <u>2</u></p> <p align="center"><u>47.1</u> mg</p>
<p>Filter Flask No. _____</p> <p>Final Weight _____ mg Initial Weight _____ mg Filter and Particulates <u>436.2</u> mg Filter No. <u>104</u> Tare Weight <u>423.8</u> mg Particulates <u>12.4</u> mg Dry Particulates and Probe <u>34.7</u> mg Front Half Particulates <u>47.1</u> mg</p>	

PARTICULATE FIELD DATA

VERY IMPORTANT - FILL IN ALL BLANKS

Plant Virginia Vermiculite Test Start Time: 1448 Ambient Temp. °F 65
 Run No. 3 Stop Time: 1551 Ear. Press. in. Hg. 30.17
 Location Southern Stack Filter No. 109 Assumed Moisture % 10
 Date 10/27/71 Filter Tare Weight 6.4225 gm Heater Box Setting, °F 250-7
 Operator CA Orsat No. 1 Probe Tip Dia., In. 0.170
 Sample Box No. 8 Date Rebuilt 10/26/79 Probe Length 5 ft.
 Meter Box No. 7 Tyline No. _____ Probe Heater Setting 250-7
 Meter ID# 1.63 Date Rebuilt _____ Avg. ΔP 2.6 Avg. ΔH 1.84
 O Factor 0.85 Sample No. 501 Leak Rate @ 15" Hg. 0.002 (cm³/min) @ 0.001CF

Point	Clock Time (min)	Dry to Wet Ratio	Dry to Wet Ratio	Dry Gas Temp.	Filter Outlet Gauge	Filter Inlet Gauge	Imp. Temp. °C	Stack Temp. °C	Filter Temp. °C	Filter Inlet	Filter Outlet	Filter Inlet	Filter Outlet	Filter Inlet	Filter Outlet
C-1	0	401.224	1.55		95	95	67	130	250	40.5	40.5	1.40	1.40	1.70	1.70
C-2	5	416.57	2.00		96	96	67	130	250	40.5	40.5	1.86	1.86	1.70	1.70
C-3	10	422.71	2.70		97	97	67	130	250	40.5	40.5	1.90	1.90	1.70	1.70
C-4	15	426.165	2.60		97	97	67	130	250	40.5	40.5	1.84	1.84	1.70	1.70
B-1	20	430.0	2.80		98	98	65	130	250	40.5	40.5	2.0	2.0	1.70	1.70
B-2	25	433.91	2.70		98	98	65	130	250	40.5	40.5	1.70	1.70	1.70	1.70
B-3	30	437.93	2.70		98	98	65	130	250	40.5	40.5	1.70	1.70	1.70	1.70
A-1	40	441.811	2.20		99	99	66	130	250	40.5	40.5	1.60	1.60	1.70	1.70
A-2	45	445.42	2.40		99	99	66	130	250	40.5	40.5	1.70	1.70	1.70	1.70
A-3	50	447.12	2.20		99	99	66	130	250	40.5	40.5	1.60	1.60	1.70	1.70
A-4	55	502.69	1.75		99	99	66	130	250	40.5	40.5	1.25	1.25	1.70	1.70
A-5	60	505.832	1.75		99	99	66	130	250	40.5	40.5	1.25	1.25	1.70	1.70
Uplume		44.658													
Moisture		(VAP) No.	1.551		73.5							6.736			130.0

Test Observer: Baker

PARTICULATE SOURCE SAMPLE

Analysis Date(s) 10/27/79 - 10/29/79 Analytical Chemist Z. Porcell
 Plant Sampled VIRGINIA VERMICULITE LTD. Location LOUISA, VIRGINIA
 Sampling Location ROTARY DRYER EXHAUST Sampling Date(s) 10/27/79
 Sample Run No. 3 Sampling Case No. 8

Sample Labels

H₂O _____ Silica Gel _____ Filter _____ Probe _____
 Impinger Rinse _____ Dry Particulate _____ Other _____

Analysis Performed _____

Reference Method _____ Analytical Sample Temp. 70 °F

Comments:

<p><u>Moisture Data</u></p> <p>Final Volume H₂O in Impingers <u>310</u> ml Initial Volume H₂O in Impingers <u>200</u> ml Volume H₂O condensed <u>110</u> ml</p> <p>Final Weight Silica Gel <u>213</u> gm Initial Weight Silica Gel <u>200</u> gm (1gmH₂O=1mlH₂O) Volume H₂O Absorbed <u>13</u> ml</p>	<p><u>Total Moisture</u></p> <p>H₂O Condensed <u>110</u> ml H₂O Absorbed <u>13</u> ml H₂O Total <u>123</u> ml</p>
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<p><u>Particulate Data</u></p> <p>CH₃-CH₂-O-CH₂-CH₃/CHCL₃ extract Flask No. _____ Final Weight _____ mg Initial Weight _____ mg Organic Fraction _____ mg</p>	<p><u>Total Particulate Sampled</u></p> <p>Organic Fraction _____ mg Inorganic Fraction _____ mg Front Half Particulates _____</p>
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<p>Extracted H₂O Flask No. _____</p> <p>Final Weight _____ mg Initial Weight _____ mg Inorganic Fraction _____ mg</p>	<p><u>Total Particulates</u></p> <p>Run No. <u>3</u></p>
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<p>Filter Flask No. _____</p> <p>Final Weight _____ mg Initial Weight _____ mg Filter and Particulates <u>428.3</u> mg Filter No. <u>109</u> Tare Weight <u>422.5</u> mg Particulates <u>5.8</u> mg Dry Particulates and Probe <u>40.7</u> mg Front Half Particulates <u>46.5</u> mg</p>	<p><u>46.5</u> mg</p>
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PARTICULATE FIELD DATA
VERY IMPORTANT - FILL IN ALL BLANKS

Plant Virginia Vermiculite Ambient Temp. °F 65
 Run No. Asbestos Sample Bar. Press. in. Hg. _____
 Location Scrubber Stack Assumed Moisture % 10.00
 Date 10/27/79 Heater Box Setting, °F _____
 Operator GA Probe Tip Dia., In. 0.500
 Sample Box No. 9 Probe Length 5 ft
 Meter Box No. 7 Probe Heater Setting _____
 Meter Amp. 1.63 Avg. Sp. 2.6 Avg. W. 1.84
 C Factor 0.85 Log Rate of Flow 0.0052

Point	Clock Time (min)	Pilot Meter Reading	Pilot Flow in L/min	Orifice Air in H ₂ O	Dry Gas Temp. °F	Inlet Outlet Gauge	Push Vacuum in. Hg.	Box Temp. °F	Impulse Count	Stack Temp. in. Hg.	Stack Temp. °F
B-3	0	526.047	2.40	4.00	86	3	250	65	10.50	130	
	5			4.00	89	3	250	65	10.50	130	
	10	517.18	2.7	4.00	90	3	250	65			
	15			4.00	91	6	250	65			
	20	528.48	2.4	4.00	94	6	250	65			
	25	534.07	2.3								
	30	539.17									
Volume		33.68									

Comments: Maximum flow rate obtainable and throughout
Test: 0.3 m Millipore Filter
 Test Observer: _____

APPENDIX C
SAMPLING AND ANALYTICAL PROCEDURES

Title 40—Protection of Environment

CHAPTER I—ENVIRONMENTAL
PROTECTION AGENCY

(FRL 754-5)

PART 60—STANDARDS OF PERFORMANCE FOR NEW STATIONARY SOURCES

Revision to Reference Methods 1-8

AGENCY: Environmental Protection Agency.

ACTION: Final Rule.

SUMMARY: This rule revises Reference Methods 1 through 8, the detailed requirements used to measure emissions from affected facilities to determine whether they are in compliance with a standard of performance. The methods were originally promulgated December 23, 1971, and since that time several revisions became apparent which would clarify, correct and improve the methods. These revisions make the methods easier to use, and improve their accuracy and reliability.

EFFECTIVE DATE: September 19, 1977.

ADDRESSES: Copies of the comment letters are available for public inspection and copying at the U.S. Environmental Protection Agency, Public Information Reference Unit (EPA Library), Room 2922, 401 M Street, S.W., Washington, D.C. 20460. A summary of the comments and EPA's responses may be obtained upon written request from the EPA Public Information Center (PM-215), 401 M Street, S.W., Washington, D.C. 20460 (specify "Public Comment Summary: Revisions to Reference Methods 1-8 in Appendix A of Standards of Performance for New Stationary Sources").

FOR FURTHER INFORMATION CONTACT:

Don R. Goodwin, Emission Standards and Engineering Division, Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone No. 919-541-5271.

SUPPLEMENTARY INFORMATION: The amendments were proposed on June 8, 1976 (40 FR 23069). A total of 55 comment letters were received during the comment period—34 from industry, 15 from governmental agencies, and 6 from other interested parties. They contained numerous suggestions which were incorporated in the final revisions.

Changes common to all eight of the reference methods are: (1) the clarification of procedures and equipment specifications resulting from the comments, (2) the addition of guidelines for alternative procedures and equipment to make prior approval of the Administrator unnecessary and (3) the addition of an introduction to each reference method discussing the general use of the method and delineating the procedure for using alternative methods and equipment.

Specific changes to the methods are:

METHOD 1

1. The provision for the use of more than two traverse diameters, when specified by the Administrator, has been deleted. If one traverse diameter is in a plane containing the greatest expected concentration variation, the intended purpose of the deleted paragraph will be fulfilled.

2. Based on recent data from Fluidyne (Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow, EPA-600/2-76-170, June 1976) and Entropy Environmentalists (Determination of the Optimum Number of Traverse Points: An Analysis of Method 1 Criteria (draft), Contract No. 68-01-3172), the number of traverse points for velocity measurements has been reduced and the 2:1 length to width ratio requirement for cross-sectional layout of rectangular ducts has been replaced by a "balanced matrix" scheme.

3. Guidelines for sampling in stacks containing cyclonic flow and stacks smaller than about 0.31 meter in diameter or 0.071 m² in cross-sectional area will be published at a later date.

4. Clarification has been made as to when a check for cyclonic flow is necessary; also, the suggested procedure for determination of unacceptable flow conditions has been revised.

METHOD 2

1. The calibration of certain pitot tubes has been made optional. Appropriate construction and application guidelines have been included.

2. A detailed calibration procedure for temperature gauges has been included.

3. A leak check procedure for pitot lines has been included.

METHOD 3

1. The applicability of the method has been confined to fossil-fuel combustion processes and to other processes where it has been determined that components other than O₂, CO₂, CO, and N₂ are not present in concentrations sufficient to affect the final results.

2. Based on recent research information (Particulate Sampling Strategies for Large Power Plants Including Nonuniform Flow, EPA-600/2-76-170, June 1976), the requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling. Proportional and constant rate sampling have been found to give essentially the same result.

3. The "three consecutive" requirement has been replaced by "any three" for the determination of molecular weight, CO₂ and O₂.

4. The equation for excess air has been revised to account for the presence of CO.

5. A clearer distinction has been made between molecular weight determination and emission rate correction factor determination.

6. Single point, integrated sampling has been included.

METHOD 4

1. The sampling time of 1 hour has been changed to a total sampling time which will span the length of time the pollutant emission rate is being determined or such time as specified in an applicable subpart of the standards.

2. The requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling.

3. The leak check before the test run has been made optional; the leak check after the run remains mandatory.

METHOD 5

1. The following alternatives have been included in the method:

a. The use of metal probe liners.

b. The use of other materials of construction for filter holders and probe liner parts.

c. The use of polyethylene wash bottles and sample storage containers.

d. The use of desiccants other than silica gel or calcium sulfate, when appropriate.

e. The use of stopcock grease other than silicone grease, when appropriate.

f. The drying of filters and probe-filter catches at elevated temperatures, when appropriate.

g. The combining of the filter and probe washes into one container.

2. The leak check prior to a test run has been made optional. The post-test leak check remains mandatory. A method for correcting sample volume for excessive leakage rates has been included.

3. Detailed leak check and calibration procedures for the metering system have been included.

METHOD 6

1. Possible interfering agents of the method have been delineated.

2. The options of: (a) using a Method 8 impinger system, or (b) determining SO₂ simultaneously with particulate matter, have been included in the method.

3. Based on recent research data, the requirement for proportional sampling has been dropped and replaced with the requirement for constant rate sampling.

4. Tests have shown that isopropanol obtained from commercial sources occasionally has peroxide impurities that will cause erroneously low SO₂ measurements. Therefore, a test for detecting peroxides in isopropanol has been included in the method.

5. The leak check before the test run has been made optional; the leak check after the run remains mandatory.

6. A detailed calibration procedure for the metering system has been included in the method.

METHOD 7

1. For variable wave length spectrophotometers, a scanning procedure for determining the point of maximum absorbance has been incorporated as an option.

METHOD 8

1. Known interfering compounds have been listed to avoid misapplication of the method.

2. The determination of filterable particulate matter (including acid mist) simultaneously with SO₂ and SO₃ has been allowed where applicable.

3. Since occasionally some commercially available quantities of isopropanol

have peroxide impurities that will cause erroneously high sulfuric acid mist measurements, a test for peroxides in isopropyl alcohol has been included in the method.

4. The gravimetric technique for moisture content (rather than volumetric) has been specified because a mixture of isopropyl alcohol and water will have a volume less than the sum of the volumes of its content.

5. A closer correspondence has been made between similar parts of Methods 8 and 5.

MISCELLANEOUS

Several commenters questioned the meaning of the term "subject to the approval of the Administrator" in relation to using alternate test methods and procedures. As defined in § 60.2 of subpart A, the "Administrator" includes any authorized representative of the Administrator of the Environmental Protection Agency. Authorized representatives are EPA officials in EPA Regional Offices or State, local, and regional governmental officials who have been delegated the responsibility of enforcing regulations under 40 CFR 60. These officials in consultation with other staff members familiar with technical aspects of source testing will render decisions regarding acceptable alternate test procedures.

In accordance with section 117 of the Act, publication of these methods was preceded by consultation with appropriate advisory committees, independent experts, and Federal departments and agencies.

(Secs. 111, 114 and 301(a) of the Clean Air Act, sec. 4(a) of Pub. L. No. 91-604, 84 Stat. 1683; sec. 4(a) of Pub. L. No. 91-604, 84 Stat. 1687; sec. 2 of Pub. L. No. 90-148, 81 Stat. 504 [42 U.S.C. 1857c-6, 1857c-9, 1857g(a)].)

NOTE.—The Environmental Protection Agency has determined that this document does not contain a major proposal requiring preparation of an Economic Impact Analysis under Executive Orders 11821 and 11949 and OMB Circular A-107.

Dated: August 10, 1977.

DOUGLAS M. COSTLE,
Administrator.

Part 60 of Chapter I of Title 40 of the Code of Federal Regulations is amended by revising Methods 1 through 8 of Appendix A—Reference Methods as follows:

APPENDIX A—REFERENCE METHODS

The reference methods in this appendix are referred to in § 60.6 (Performance Tests) and § 60.11 (Compliance With Standards and Maintenance Requirements) of 40 CFR Part 60, Subpart A (General Provisions). Specific uses of these reference methods are described in the standards of performance contained in the subparts, beginning with Subpart I.

Within each standard of performance, a section titled "Test Methods and Procedures" is provided so (1) identify the test methods applicable to the facility subject to the respective standard and (2) identify any special instructions or conditions to be followed when applying a method to the respective facility. Such instructions (for example, establish sampling rates, volumes, or temperatures) are to be used either in addition to, or as a substitute for procedures in a reference method. Similarly, for sources subject to emission monitoring requirements, specific instructions pertaining to any use of a reference method are provided in the subpart or in Appendix B.

Inclusion of methods in this appendix is not intended as an endorsement or denial of their applicability to sources that are not subject to standards of performance. The methods are potentially applicable to other sources; however, applicability should be confirmed by careful and appropriate evaluation of the conditions prevalent at such sources.

The approach followed in the formulation of the reference methods involves specifications for equipment, procedures, and performance. In concept, a performance specification approach would be preferable in all methods because this allows the greatest flexibility to the user. In practice, however, this approach is impractical in most cases because performance specifications cannot be established. Most of the methods described herein, therefore, involve specific equipment specifications and procedures, and only a few methods in this appendix rely on performance criteria.

Minor changes in the reference methods should not necessarily affect the validity of the results and it is recognized that alternative and equivalent methods exist. Section 60.8 provides authority for the Administrator to specify or approve (1) equivalent methods, (2) alternative methods, and (3) minor changes in the methodology of the reference methods. It should be clearly understood that unless otherwise identified all such methods and changes must have prior approval of the Administrator. An owner employing such methods or deviations from the reference methods without obtaining prior approval does so at the risk of subsequent disapproval and retesting with approved methods.

Within the reference methods, certain specific equipment or procedures are recognized as being acceptable or potentially acceptable and are specifically identified in the methods. The items identified as acceptable options may be used without approval but must be identified in the test report. The potentially approvable options are cited as "subject to the approval of the Administrator" or as "or equivalent." Such potentially approvable techniques or alternatives may be used at the discretion of the owner without prior approval. However, detailed descriptions for applying these potentially approvable techniques or alternatives are not provided in the reference methods. Also, the potentially approvable options are not necessarily acceptable in all applications. Therefore, an owner electing to use such potentially approvable techniques or alternatives is responsible for: (1) assuring that the techniques or alternatives are in fact applicable and are properly executed; (2) including a written description of the alternative method in the test report (the written method must be clear and must be capable of being performed without additional instruction, and the degree of detail should be similar to the detail contained in the reference methods); and (3) providing any rationale or supporting data necessary to show the validity of the alternative in the particular application. Failure to meet these requirements can result in the Administrator's disapproval of the alternative.

METHOD 1—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A traverse point is then located within each of these equal areas.

1.2 Applicability. This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 m² (113 in.²) in cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

2. Procedure

2.1 Selection of Measurement Site. Sampling or velocity measurement is performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter (D_e) shall be calculated from the following equation, to determine the upstream and downstream distances:

$$D_e = \frac{2LW}{L+W}$$

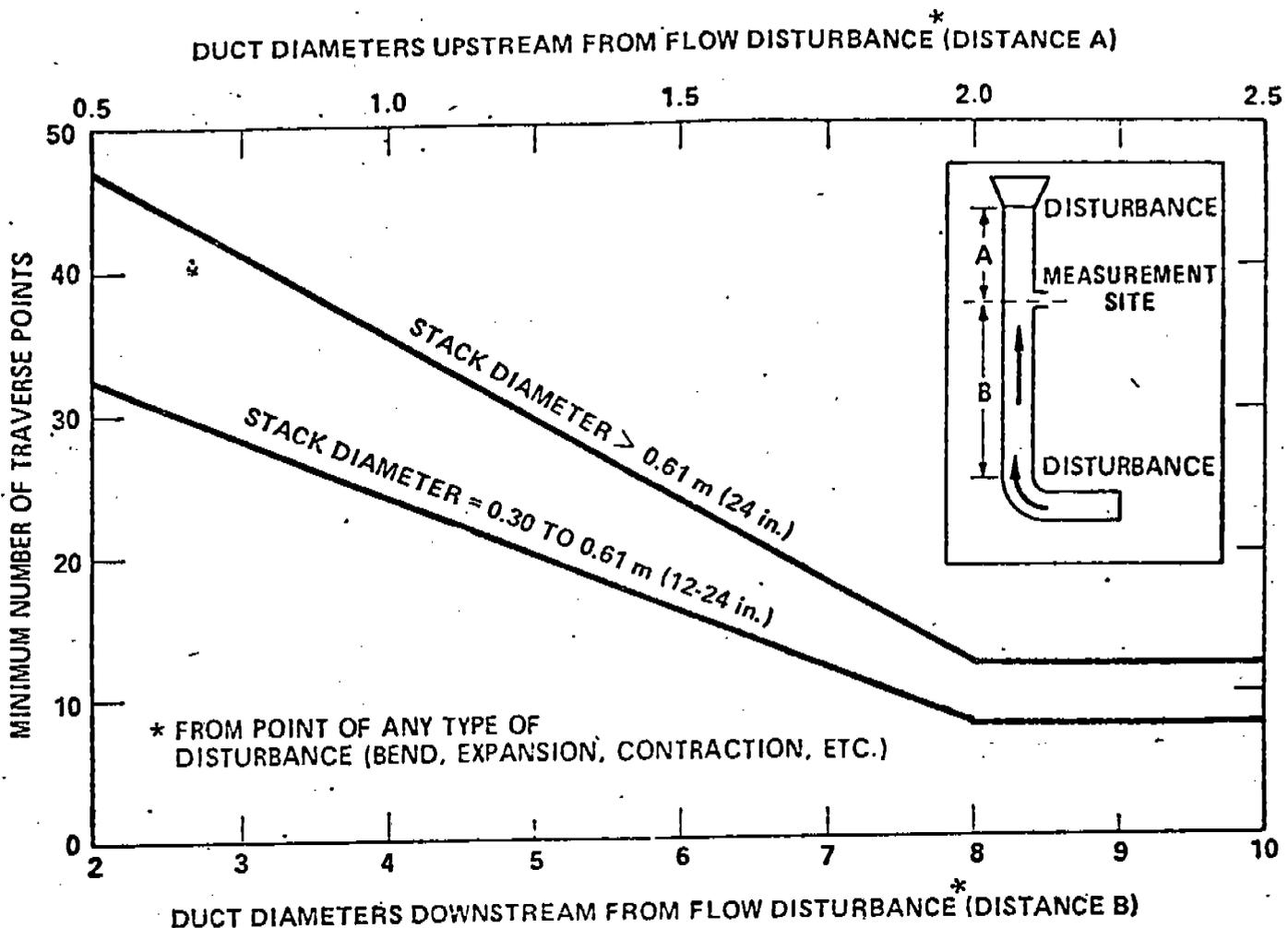


Figure 1-1. Minimum number of traverse points for particulate traverses.

where L=length and W=width.

2.2 Determining the Number of Traverse Points.

2.2.1 Particulate Traverses. When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12-24 in.); (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12-24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1. Before referring to the figure, however, determine the distances from the chosen measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1.

TABLE 1-1. Cross-sectional layout for rectangular stacks

Number of traverse points:	Mo- stic lay- out
8	3x3
12	4x3
16	4x4
20	5x4
25	5x5
30	6x5
36	6x6
42	7x6
48	7x7

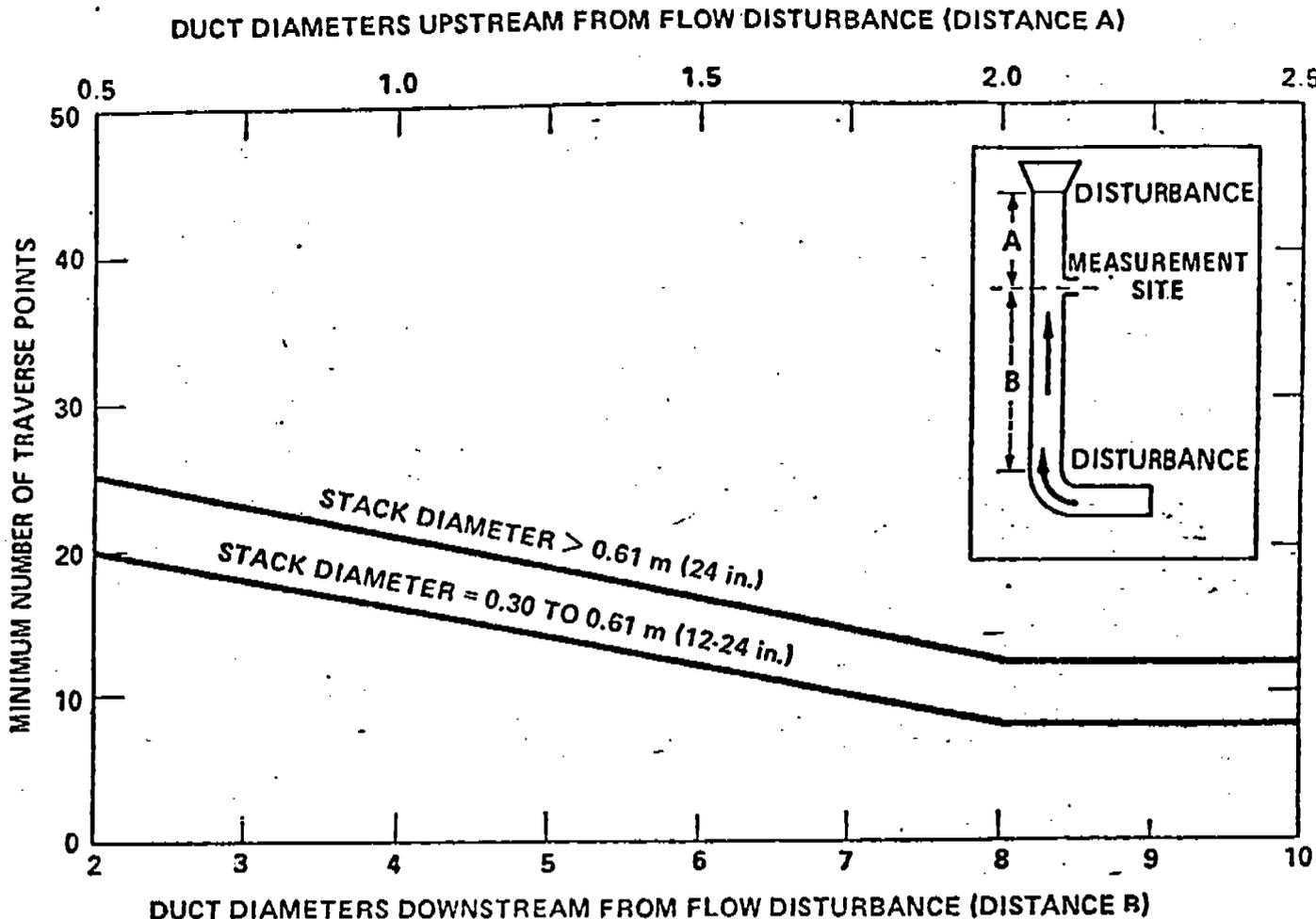


Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

2.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that for particulate traverses (Section 2.2.1) is followed, except that Figure 1-2 may be used instead of Figure 1-1.

2.3 Cross-Sectional Layout and Location of Traverse Points.

2.3.1 Circular Stacks. Locate the traverse points on two perpendicular diameters according to Table 1-2 and the example shown in Figure 1-3. Any equation (for examples, see Citations 2 and 3 in the Bibliography) that gives the same values as those in Table 1-2 may be used in lieu of Table 1-2.

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to approval of the Administrator.

In addition, for stacks having diameters greater than 0.61 m (24 in.) no traverse points shall be located within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls. To meet these criteria, observe the procedures given below.

2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.). When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.

RULES AND REGULATIONS

TRAVERSE POINT	DISTANCE, % of diameter
1	4.4
2	14.7
3	29.5
4	70.5
5	85.3
6	95.6

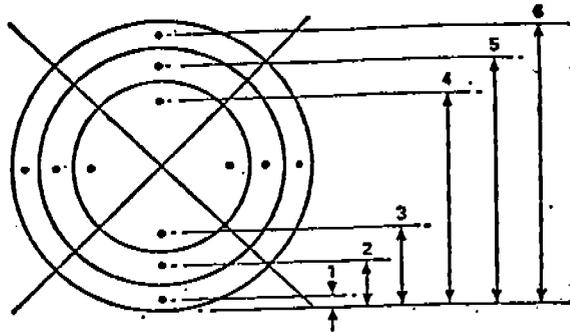


Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

(2) In stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.

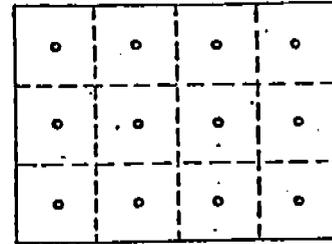


Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

Table 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS (Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1
8				96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0
10					97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.8	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.6	92.1
22											98.9	94.5
23												96.8
24												98.9

Level and zero the manometer. Connect a Type B pitot tube to the manometer. Position the Type B pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane; when the Type B pitot tube is in this position, it is at "0° reference." Note the differential pressure (Δp) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to $\pm 90^\circ$ yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle (α) to the nearest degree. After the null technique has been applied at each traverse point, calculate the average of the absolute values of α ; assign α values of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of α is greater than 10°, the overall flow condition in the stack is unacceptable and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.

3. Bibliography

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METHOD 2—DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE (TYPE S PITOT TUBE)

1. Principle and Applicability

1.1 Principle. The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stausscheibe or reverse type) pitot tube.

1.2 Applicability. This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable at measurement sites which fail to meet the criteria of Method 1, Section 2.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams; Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U.S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

2. Apparatus

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

2.3.1.2 Stacks With Diameters Equal to or Less Than 0.61 m (24 in.). Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

2.3.2 Rectangular Stacks. Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points,

and then locate a traverse point at the centroid of each equal area according to the example in Figure 1-4.

The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

2.4 Verification of Absence of Cyclonic Flow. In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or

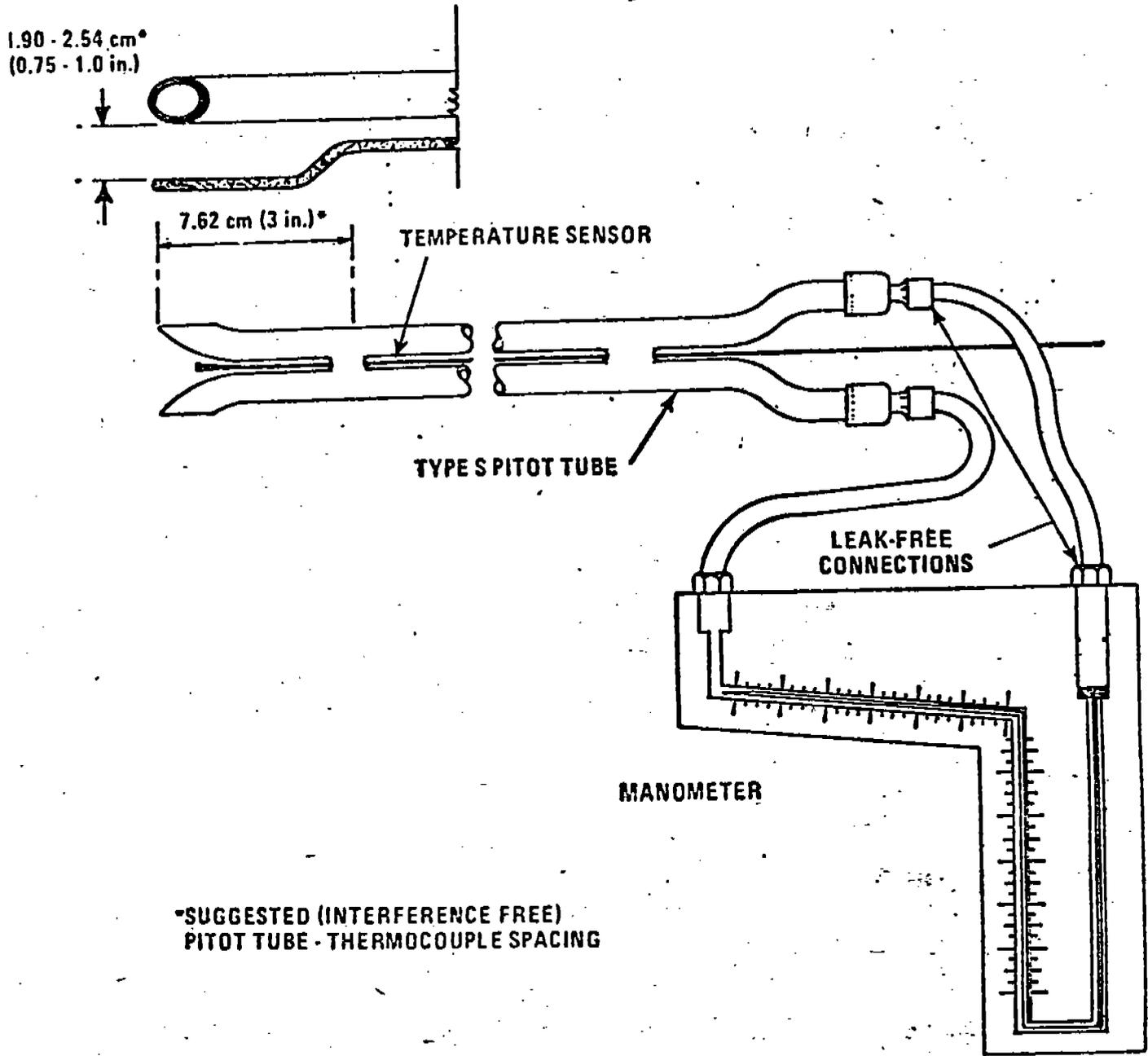


Figure 2-1. Type S pitot tube manometer assembly.

2.1 Type S Pitot Tube. The Type S pitot tube (Figure 2-1) shall be made of metal tubing (e.g., stainless steel). It is recommended that the external tubing diameter (dimension D , Figure 2-2b) be between 0.48 and 0.95 centimeters ($3/16$ and $3/8$ inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions P_1 and P_2 , Figure 2-2b); it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2-2; however, slight misalignments of the openings are permissible (see Figure 2-3).

The Type S pitot tube shall have a known coefficient, determined as outlined in Section 4. An identification number shall be assigned to the pitot tube; this number shall be permanently marked or engraved on the body of the tube.

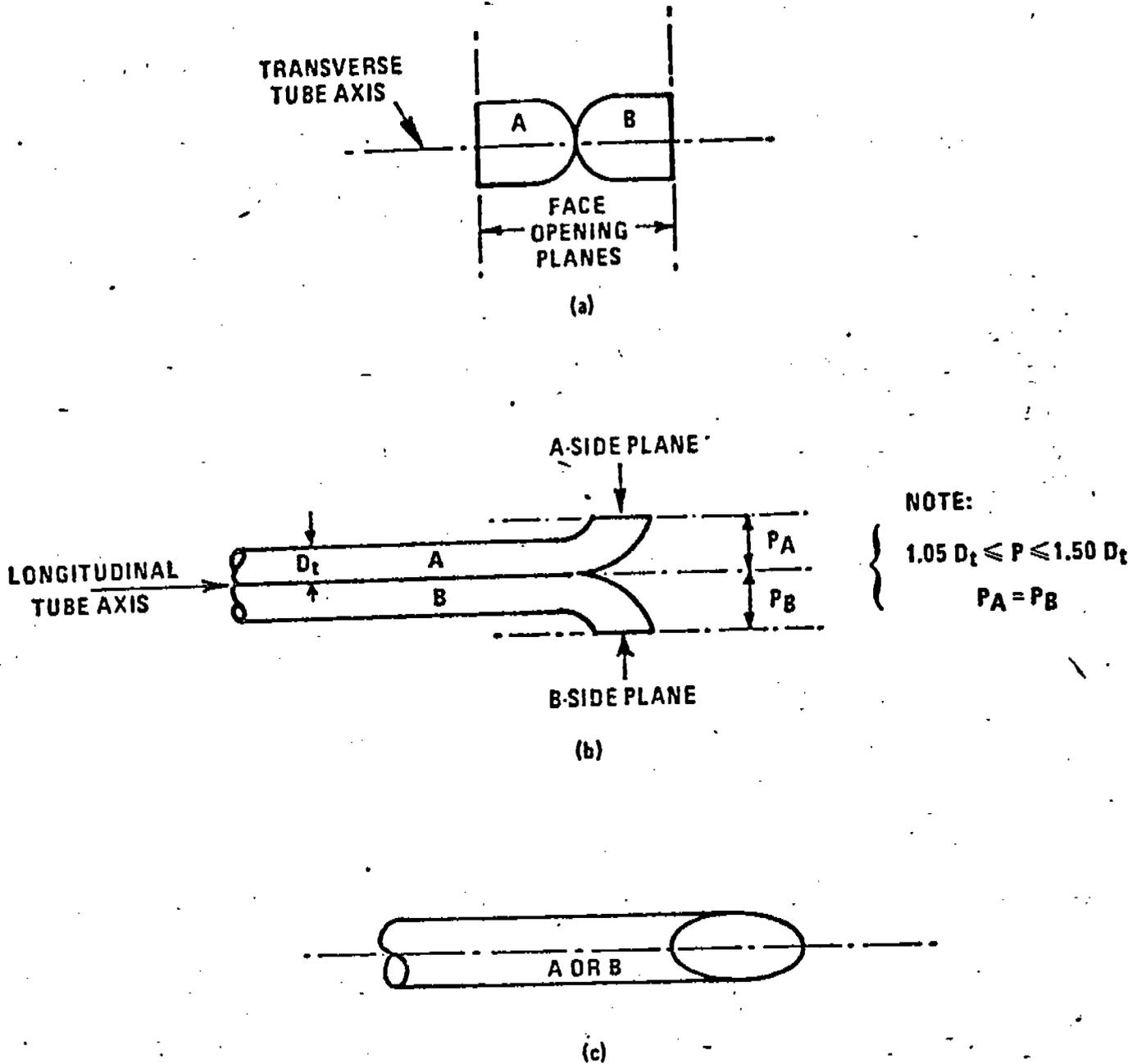


Figure 2-2. Properly constructed Type S pitot tube, shown in: (a) end view; face opening planes perpendicular to transverse axis; (b) top view; face opening planes parallel to longitudinal axis; (c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way.

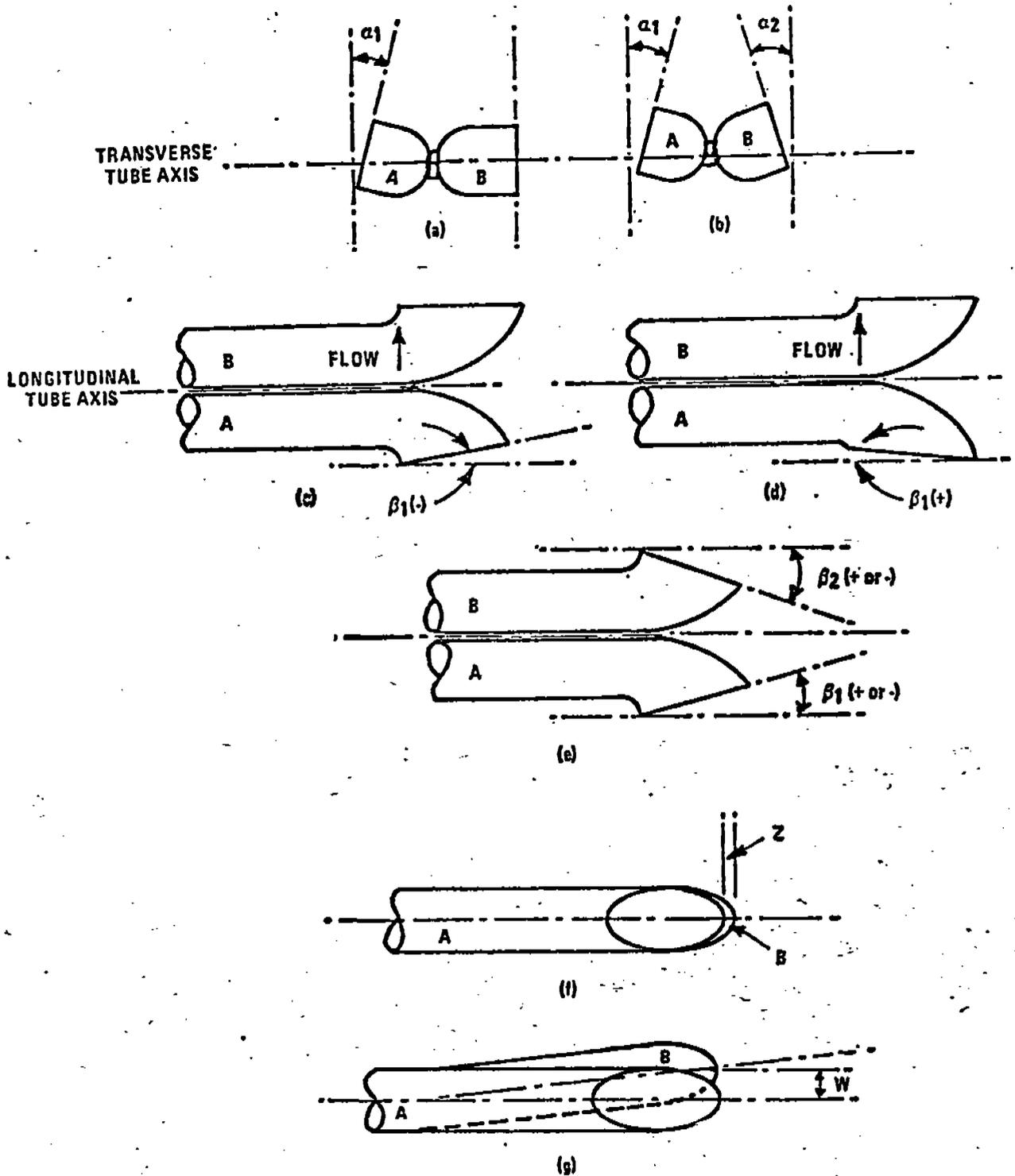


Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of $\bar{C}_p(s)$ so long as α_1 and $\alpha_2 < 10^\circ$, β_1 and $\beta_2 < 5^\circ$, $z < 0.32$ cm (1/8 in.) and $w < 0.08$ cm (1/32 in.) (citation 11 in Section 6).

A standard pitot tube may be used instead of a Type B, provided that it meets the specifications of Sections 2.7 and 4.2; note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period; this can be done by taking a velocity head (Δp) reading at the final traverse point, cleaning out head (Δp) reading at the standard pitot tube by the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another Δp reading. If the Δp readings made before and after the air purge are the same (± 5 percent), the traverse after the air purge is acceptable. Note that if Δp is acceptable. Otherwise, reject the run. Note that if Δp at the final traverse point is unsuitably low, another point may be selected. If "back-purging" at regular intervals is part of the procedure, then comparative Δp readings shall be taken, as above, for the last two back readings at which suitably high Δp readings are observed.

2.2 Differential Pressure Gauge. An inclined manometer or equivalent device is used. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. H_2O divisions on the 0- to 1-in. inclined scale, and 0.1-in. H_2O divisions on the 1- to 10-in. vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of Δp values as low as 1.3 mm (0.05 in.) H_2O . However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all Δp readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) H_2O ; (2) for traverses of 12 or more points, more than 10 percent of the individual Δp readings are below 1.3 mm (0.05 in.) H_2O ; (3) for traverses of fewer than 12 points, more than one Δp reading is below 1.3 mm (0.05 in.) H_2O . Citation 18 in Section 6 describes commercially available instrumentation for the measurement of low-range gas velocities.

As an alternative to criteria (1) through (3) above, the following calculation may be performed to determine the necessity of using a more sensitive differential pressure gauge:

$$T = \frac{\sum_{i=1}^n \sqrt{\Delta p_i + K}}{\sum_{i=1}^n \sqrt{\Delta p_i}}$$

where:

Δp_i = Individual velocity head reading at a traverse point, mm H_2O (in. H_2O).

n = Total number of traverse points.

K = 0.13 mm H_2O when metric units are used and 0.005 in. H_2O when English units are used.

If T is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

NOTE.—If differential pressure gauges other than inclined manometers are used (e.g., magnetic gauges), their calibration must be checked after each test series. To check the calibration of a differential pressure gauge, compare Δp readings of the gauge with those of a gauge-oil manometer at a minimum of three points, approximately representing the range of Δp values in the stack. If, at each point, the values of Δp as read by the differential pressure gauge and gauge-oil manometer agree to within 5 percent, the differential pressure gauge shall be considered to be in proper calibration. Otherwise, the test series shall either be voided, or procedures to adjust the measured Δp values and final results shall be used, subject to the approval of the Administrator.

2.3 Temperature Gauge. A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature shall be used. The temperature gauge shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2-1 and also Figure 2-7 in Section 4). Alternate positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of Section 4. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the tem-

perature gauge need not be attached to the pitot tube; this alternative is subject to the approval of the Administrator.

2.4 Pressure Probe and Gauge. A piezometer tube and mercury- or water-filled U-tube manometer capable of measuring stack pressure to within 2.5 mm (0.1 in.) Hg is used. The static tap of a standard type pitot tube or one leg of a Type X pitot tube with the face opening planes positioned parallel to the gas flow may also be used as the pressure probe.

2.5 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30-meter (100 foot) elevation increase, or vice-versa for elevation decrease.

2.6 Gas Density Determination Equipment. Method 3 equipment, if needed (see Section 3.6), to determine the stack gas dry molecular weight, and Reference Method 4 or Method 5 equipment for moisture content determination; other methods may be used subject to approval of the Administrator.

2.7 Calibration Pitot Tube. When calibration of the Type B pitot tube is necessary (see Section 4), a standard pitot tube is used as a reference. The standard pitot tube shall, preferably, have a known coefficient, obtained either (1) directly from the National Bureau of Standards, Route 270, Quince Orchard Road, Gaithersburg,

Maryland, or (2) by calibration against another standard pitot tube with an NBS-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given in 2.7.1 through 2.7.5 below and illustrated in Figure 2-4 (see also Citations 7, 8, and 17 in Section 6) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of about 0.99 ± 0.01 .

2.7.1 Hemispherical (shown in Figure 2-4), ellipsoidal, or conical tip.

2.7.2 A minimum of six diameters straight run (based upon D , the external diameter of the tube) between the tip and the static pressure holes.

2.7.3 A minimum of eight diameters straight run between the static pressure holes and the centerline of the external tube, following the 90 degree bend.

2.7.4 Static pressure holes of equal size (approximately $0.1 D$), equally spaced in a piezometer ring configuration.

2.7.5 Ninety degree bend, with curved or mitered junction.

2.8 Differential Pressure Gauge for Type B Pitot Tube Calibration. An inclined manometer or equivalent is used. If the single-velocity calibration technique is employed (see Section 4.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in. H_2O). For multiveLOCITY calibrations, the gauge shall be readable to the nearest 0.13 mm H_2O (0.005 in. H_2O) for Δp values between 1.3 and 25 mm H_2O (0.05 and 1.0 in. H_2O), and to the nearest 1.3 mm H_2O (0.05 in. H_2O) for Δp values above 25 mm H_2O (1.0 in. H_2O). A special, more sensitive gauge will be required to read Δp values below 1.3 mm H_2O [0.05 in. H_2O] (see Citation 18 in Section 6).

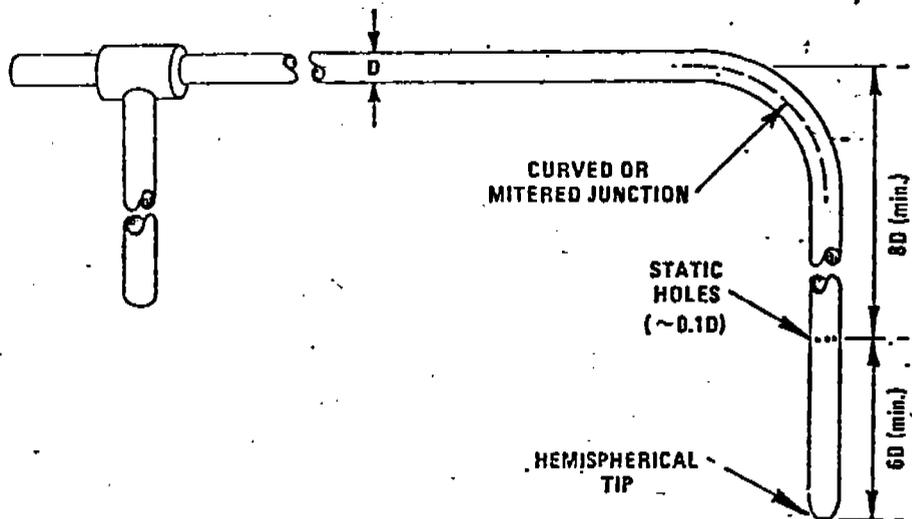


Figure 2-4. Standard pitot tube design specifications.

3. Procedure

3.1 Set up the apparatus as shown in Figure 2-1. Capillary tubing or surge tanks installed between the manometer and pitot tube may be used to dampen Δp fluctuations. It is recommended, but not required, that a pretest leak-check be conducted, as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3 in.) H_2O velocity pressure registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3 in.) H_2O . Other leak-check procedures, subject to the approval of the Administrator, may be used.

3.2 Level and zero the manometer. Because the ma-

nometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Record all necessary data as shown in the example data sheet (Figure 2-5).

3.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of Δp values encountered (see Section 2.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the Δp and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 3.1 above, to validate the traverse run.

3.4 Measure the static pressure in the stack. One reading is usually adequate.

3.5 Determine the atmospheric pressure.

3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially CO_2 , O_2 , CO , and N_2 , use Method 2. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

3.7 Obtain the moisture content from Reference Method 4 (or equivalent) or from Method 5.

3.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.

4. Calibration

4.1 Type S Pitot Tube. Before its initial use, carefully examine the Type S pitot tube in top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figure 2-6 or 2-8. The pitot tube shall not be used if it fails to meet these alignment specifications.

After verifying the face opening alignment, measure and record the following dimensions of the pitot tube:

(a) the external tubing diameter (dimension D_t , Figure 2-2b); and (b) the base-to-opening plane distances (dimensions P_1 and P_2 , Figure 2-2b). If D_t is between 0.48 and 0.95 cm ($3/16$ and $3/8$ in.) and if P_1 and P_2 are equal and between 1.06 and 1.50 P_t , there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the pitot tube is part of an assembly, calibration may still be required, despite knowledge of the baseline coefficient value (see Section 4.1.1). If D_t , P_1 , and P_2 are outside the specified limits, the pitot tube must be calibrated as outlined in 4.1.2 through 4.1.5 below.

4.1.1 Type S Pitot Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Citation 9 in Section 6); therefore an assigned (or otherwise known) baseline coefficient

value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2-6 through 2-8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm ($3/16$ and $3/8$ in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2-6 through 2-8 shall be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, and prior to calibration, the values of the inter-component spacings (pitot-nozzle, pitot-thermocouple, pitot-probe sheath) shall be measured and recorded.

Note.—Do not use any Type S pitot tube assembly which is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2-6b).

4.1.2 Calibration Setup. If the Type S pitot tube is to be calibrated, one leg of the tube shall be permanently marked A, and the other, B. Calibration shall be done in a flow system having the following essential design features:

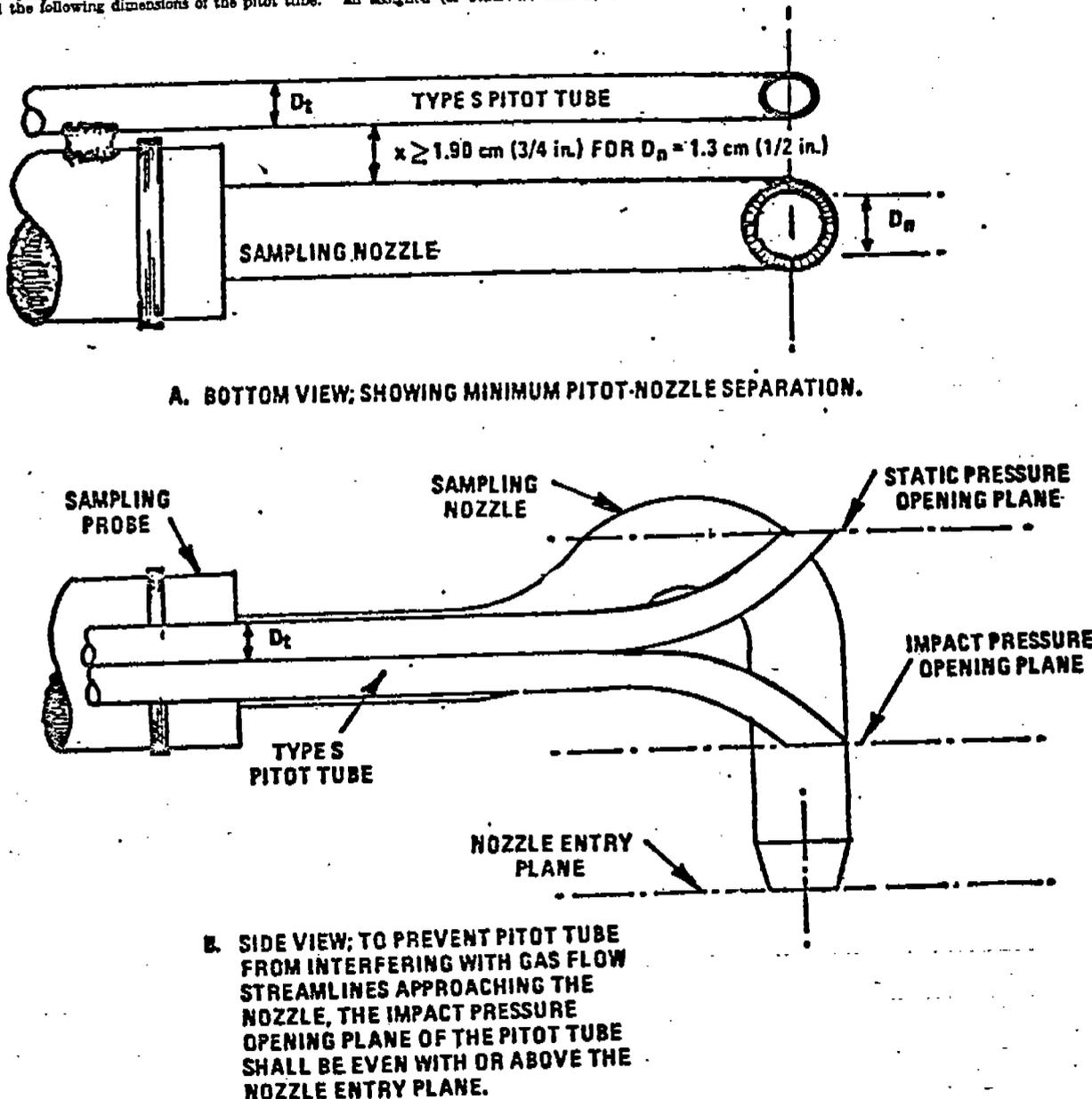


Figure 2-6. Proper pitot tube - sampling nozzle configuration to prevent aerodynamic interference; buttonhook - type nozzle; centers of nozzle and pitot opening aligned; D_t between 0.48 and 0.95 cm ($3/16$ and $3/8$ in.).

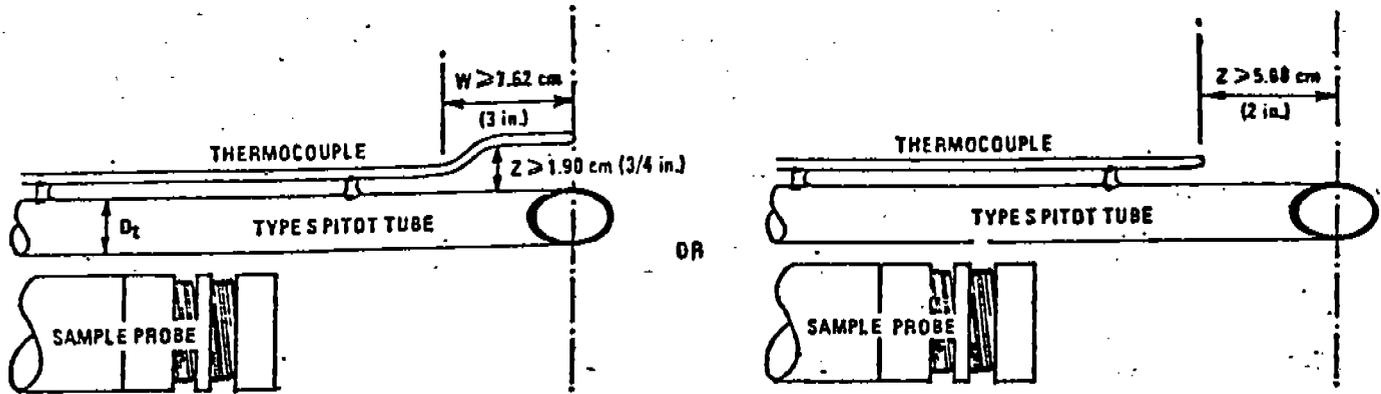


Figure 2-7. Proper thermocouple placement to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

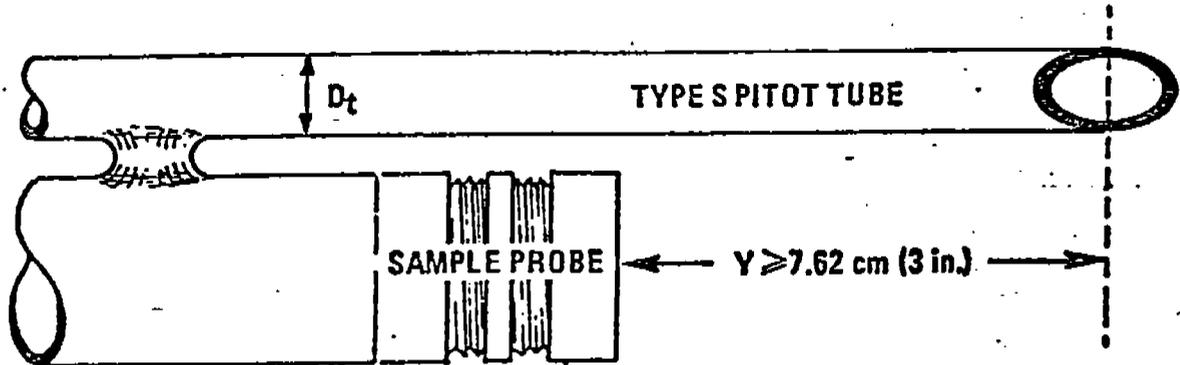


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

4.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For circular cross-sections, the minimum duct diameter shall be 30.5 cm (12 in.); for rectangular cross-sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

4.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross-section, use an equivalent diameter, calculated from the following equation, to determine the number of duct diameters:

$$D_e = \frac{2LW}{L+W}$$

Equation 2-1

where:

D_e = Equivalent diameter
 L = Length
 W = Width

To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

NOTE.—The eight- and two-diameter criteria are not absolute; other test section locations may be used (subject to approval of the Administrator), provided that the flow at the test site is stable and demonstrably parallel to the duct axis.

4.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 915 m/min (3,000

f/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 915 m/min (3,000 f/min) will generally be valid to within ± 3 percent for the measurement of velocities above 395 m/min (1,000 f/min) and to within ± 5 to 6 percent for the measurement of velocities between 180 and 305 m/min (600 and 1,000 f/min). If a more precise correlation between C_p and velocity is desired, the flow system shall have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,525 m/min (600 to 5,000 f/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in Section 6 for details).

4.1.2.4 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 4.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

4.1.3.1 Make sure that the manometer is properly filled and that the oil is free from contamination and is of the proper density. Inspect and leak-check all pitot lines; repair or replace if necessary.

4.1.3.2 Level and zero the manometer. Turn on the fan and allow the flow to stabilize. Seal the Type S entry port.

4.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in Section 4.1.5.1), and align the tube so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.4 Read Δp_{s1} and record its value in a data table similar to the one shown in Figure 2-9. Remove the standard pitot tube from the duct and disconnect it from the manometer. Seal the standard entry port.

4.1.3.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its A side impact opening is at the same point as was the standard pitot tube and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.6 Read Δp_s and enter its value in the data table. Remove the Type S pitot tube from the duct and disconnect it from the manometer.

4.1.3.7 Repeat steps 4.1.3.3 through 4.1.3.6 above until three pairs of Δp readings have been obtained.

4.1.3.8 Repeat steps 4.1.3.3 through 4.1.3.7 above for the B side of the Type S pitot tube.

4.1.3.9 Perform calculations, as described in Section 4.1.4 below.

4.1.4 Calculations.

4.1.4.1 For each of the six pairs of Δp readings (i.e., three from side A and three from side B) obtained in Section 4.1.3 above, calculate the value of the Type S pitot tube coefficient as follows:

PITOT TUBE IDENTIFICATION NUMBER: _____ DATE: _____
 CALIBRATED BY: _____

"A" SIDE CALIBRATION				
RUN NO.	Δp_{std} cm H ₂ O (in. H ₂ O)	$\Delta p(s)$ cm H ₂ O (in. H ₂ O)	$C_p(s)$	DEVIATION $C_p(s) - \bar{C}_p(A)$
1				
2				
3				
			\bar{C}_p (SIDE A)	

"B" SIDE CALIBRATION				
RUN NO.	Δp_{std} cm H ₂ O (in. H ₂ O)	$\Delta p(s)$ cm H ₂ O (in. H ₂ O)	$C_p(s)$	DEVIATION $C_p(s) - \bar{C}_p(B)$
1				
2				
3				
			\bar{C}_p (SIDE B)	

$$\text{AVERAGE DEVIATION} = \sigma \text{ (A OR B)} = \frac{1}{3} \sum |C_p(s) - \bar{C}_p(A \text{ OR } B)| \leftarrow \text{MUST BE } \leq 0.01$$

$$|\bar{C}_p \text{ (SIDE A)} - \bar{C}_p \text{ (SIDE B)}| \leftarrow \text{MUST BE } \leq 0.01$$

Figure 2-9. Pitot tube calibration data.

$$\bar{C}_p(s) = C_{p(Std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_s}}$$

Equation 2-2

where:
 $C_{p(s)}$ = Type S pitot tube coefficient
 $C_{p(Std)}$ = Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tube is designed

according to the criteria of Sections 2.7.1 to 2.7.5 of this method.
 Δp_{std} = Velocity head measured by the standard pitot tube, cm H₂O (in. H₂O)
 Δp_s = Velocity head measured by the Type S pitot tube, cm H₂O (in. H₂O)

4.1.4.2 Calculate \bar{C}_p (side A), the mean A-side coefficient, and \bar{C}_p (side B), the mean B-side coefficient; calculate the difference between these two average values.

4.1.4.3 Calculate the deviation of each of the three A-side values of $C_{p(s)}$ from \bar{C}_p (side A), and the deviation of each B-side value of $C_{p(s)}$ from \bar{C}_p (side B). Use the following equation:

$$\text{Deviation} = C_{p(s)} - \bar{C}_p \text{ (A or B)} \quad \text{Equation 2-3}$$

4.1.4.4 Calculate σ , the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma \text{ (side A or B)} = \frac{\sum |C_{p(s)} - \bar{C}_p \text{ (A or B)}|}{3} \quad \text{Equation 2-4}$$

4.1.4.5 Use the Type S pitot tube only if the values of σ (side A) and σ (side B) are less than or equal to 0.01 and if the absolute value of the difference between \bar{C}_p (A) and \bar{C}_p (B) is 0.01 or less.

4.1.5 Special considerations.

4.1.5.1 Selection of calibration point.

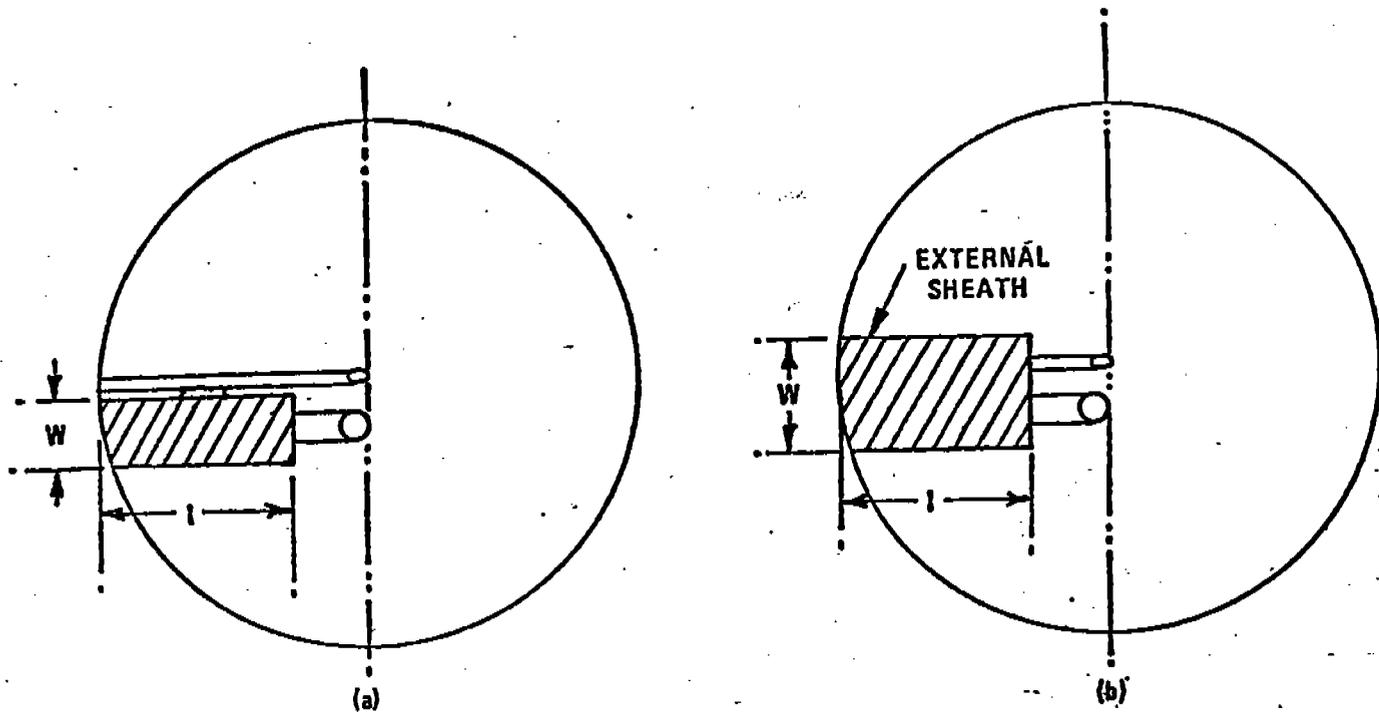
4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., \bar{C}_p (side A) and \bar{C}_p (side B), will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2-6 through 2-8).

4.1.5.1.2 For Type S pitot tube-thermocouple combinations (without sample probe), select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2-6 and 2-6).

4.1.5.1.3 For assemblies with sample probes, the calibration point should be located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area blockage and yield incorrect coefficient values (Citation 9 in Section 6). Therefore, to minimize the blockage effect, the calibration point may be a few inches off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2-10a), and 3 percent or less for assemblies with external sheaths (Figure 2-10b).

4.1.5.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance fails to meet the specification illustrated in Figure 2-6a), the value of $C_{p(s)}$ depends upon the amount of free-space between the tube and nozzle, and therefore is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or 1/4 in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3,000 ft/min), which is the calibration velocity; note also that it is not necessary to draw an isokinetic sample during calibration (see Citation 19 in Section 6).

4.1.5.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2-2 or 2-3, however, and must have an average deviation (σ) value of 0.01 or less (see Section 4.1.4.4).



$$\text{ESTIMATED SHEATH BLOCKAGE (\%)} = \left[\frac{l \times W}{\text{DUCT AREA}} \right] \times 100$$

Figure 2-10. Projected-area models for typical pitot tube assemblies.

4.1.6 Field Use and Recalibration.
 4.1.6.1 Field Use.
 4.1.6.1.1 When a Type B pitot tube (isolated tube or assembly) is used in the field, the appropriate coefficient value (whether assigned or obtained by calibration) shall be used to perform velocity calculations. For calibrated Type B pitot tubes, the A side coefficient shall be used when the A side of the tube faces the flow, and the B side coefficient shall be used when the B side faces the flow; alternatively, the arithmetic average of the A and B side coefficient values may be used, irrespective of which side faces the flow.
 4.1.6.1.2 When a probe assembly is used to sample a small duct (12 to 36 in. in diameter), the probe sheath sometimes blocks a significant part of the duct cross-section, causing a reduction in the effective value of C_p . Consult Citation 9 in Section 6 for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 12 inches (Citation 16 in Section 6).
 4.1.6.2 Recalibration.
 4.1.6.2.1 Isolated Pitot Tubes. After each field use, the pitot tube shall be carefully reexamined in top, side, and end views. If the pitot face openings are still aligned within the specifications illustrated in Figure 2-2 or 2-3, it can be assumed that the baseline coefficient of the pitot tube has not changed. If, however, the tube has been damaged to the extent that it no longer meets the specifications of Figure 2-2 or 2-3, the damage shall either be repaired to restore proper alignment of the face openings or the tube shall be discarded.
 4.1.6.2.2 Pitot Tube Assemblies. After each field use, check the face opening alignment of the pitot tube, as in Section 4.1.6.2.1; also, remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figures 2-2 or 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings or recalibrate the assembly.
 4.2 Standard pitot tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 2.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly,

the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).
 4.3 Temperature Gauges. After each field use, calibrate dial thermometers, liquid-filled bulb thermometers, thermocouple-potentiometer systems, and other gauges at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405° C (761° F), use an ASTM mercury-in-glass reference thermometer, or equivalent, as a reference; alternatively, either a reference thermocouple and potentiometer (calibrated by NBS) or thermometric fixed points, e.g., ice bath and boiling water (corrected for barometric pressure) may be used. For temperatures above 405° C (761° F), use an NBS-calibrated reference thermocouple-potentiometer system or an alternate reference, subject to the approval of the Administrator.
 If, during calibration, the absolute temperatures measured with the gauge being calibrated and the reference gauge agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.
 4.4 Barometer. Calibrate the barometer used against a mercury barometer.
 5. Calculations
 Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.
 5.1 Nomenclature.
 A = Cross-sectional area of stack, m² (ft²).
 B_{wv} = Water vapor in the gas stream (from Method 5 or Reference Method 4), proportion by volume.
 C_p = Pitot tube coefficient, dimensionless.
 K_p = Pitot tube constant,

$$34.97 \frac{\text{m}}{\text{sec}} \left[\frac{(\text{g/g-mole})(\text{mm Hg})}{(\text{°K})(\text{mm H}_2\text{O})} \right]^{1/2}$$
 for the metric system and

$$85.49 \frac{\text{ft}}{\text{sec}} \left[\frac{(\text{lb/lb-mole})(\text{in. Hg})}{(\text{°R})(\text{in. H}_2\text{O})} \right]^{1/2}$$

for the English system.
 M_d = Molecular weight of stack gas, dry basis (see Section 3.6) g/g-mole (lb/lb-mole).
 M_w = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).

$$= M_d(1 - B_{wv}) + 18.0 B_{wv}$$
 Equation 2-5
 P_{sw} = Barometric pressure at measurement site, mm Hg (in. Hg).
 P_s = Stack static pressure, mm Hg (in. Hg).
 P_t = Absolute stack gas pressure, mm Hg (in. Hg).

$$= P_{sw} + P_s$$
 Equation 2-6
 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
 Q_{sv} = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).
 t_s = Stack temperature, °C (°F).
 T_s = Absolute stack temperature, °K (°R).

$$= 273 + t_s$$
 for metric Equation 2-7

$$= 460 + t_s$$
 for English Equation 2-8
 T_{std} = Standard absolute temperature, 293° K (538° R).
 v_s = Average stack gas velocity, m/sec (ft/sec).
 Δp = Velocity head of stack gas, mm H₂O (in. H₂O).
 3,600 = Conversion factor, sec/hr.
 18.0 = Molecular weight of water, g/g-mole (lb-lb-mole).
 5.2 Average stack gas velocity.

$$v_s = K_p C_p (\sqrt{\Delta p})^{0.5} \sqrt{\frac{T_s(t_{std})}{P_{std} M_d}}$$
 Equation 2-9
 5.3 Average stack gas dry volumetric flow rate.

$$Q_{sd} = 3,600(1 - B_{wv}) v_s A \left(\frac{T_{std}}{T_s(t_{std})} \right) \left(\frac{P_s}{P_{std}} \right)$$
 Equation 2-10
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Probes. Prepared by the University of Windsor for the Ministry of the Environment, Toronto, Canada. February 1975.

METHOD 3—GAS ANALYSIS FOR CARBON DIOXIDE, OXYGEN, EXCESS AIR, AND DRY MOLECULAR WEIGHT

1. Principle and Applicability

1.1 Principle. A gas sample is extracted from a stack, by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent carbon dioxide (CO₂), percent oxygen (O₂), and, if necessary, percent carbon monoxide (CO). If a dry molecular weight determination is to be made, either an Orsat or a Fyrite analyzer may be used for the analysis; for excess air or emission rate correction factor determination, an Orsat analyzer must be used.

1.2 Applicability. This method is applicable for determining CO₂ and O₂ concentrations, excess air, and dry molecular weight of a sample from a gas stream of a fossil-fuel combustion process. The method may also be applicable to other processes where it has been determined that compounds other than CO₂, O₂, CO, and nitrogen (N₂) are not present in concentrations sufficient to affect the results.

Other methods, as well as modifications to the procedure described herein, are also applicable for some or all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point; (2) a method using CO₂ or O₂ and stoichiometric calculations to determine dry molecular weight and excess air; (3) assigning a value of 30.0 for dry molecular weight, in lieu of actual measurements, for processes burning natural gas, coal, or oil. These methods and modifications may be used, but are subject to the approval of the Administrator.

2. Apparatus

As an alternative to the sampling apparatus and systems described herein, other sampling systems (e.g., liquid displacement) may be used provided such systems are capable of obtaining a representative sample and maintaining a constant sampling rate, and are otherwise capable of yielding acceptable results. Use of such systems is subject to the approval of the Administrator.

2.1 Grab Sampling (Figure 3-1)

2.1.1 Probe. The probe should be made of stainless steel or borosilicate glass tubing and should be equipped with an in-stack or out-stack filter to remove particulate matter (a plug of glass wool is satisfactory for this purpose). Any other material inert to O₂, CO₂, CO, and N₂ and resistant to temperature at sampling conditions may be used for the probe; examples of such material are aluminum, copper, quartz glass and Teflon.

2.1.2 Pump. A one-way squeeze bulb, or equivalent, is used to transport the gas sample to the analyzer.

2.2 Integrated Sampling (Figure 3-2)

2.2.1 Probe. A probe such as that described in Section 2.1.1 is suitable.

1. Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

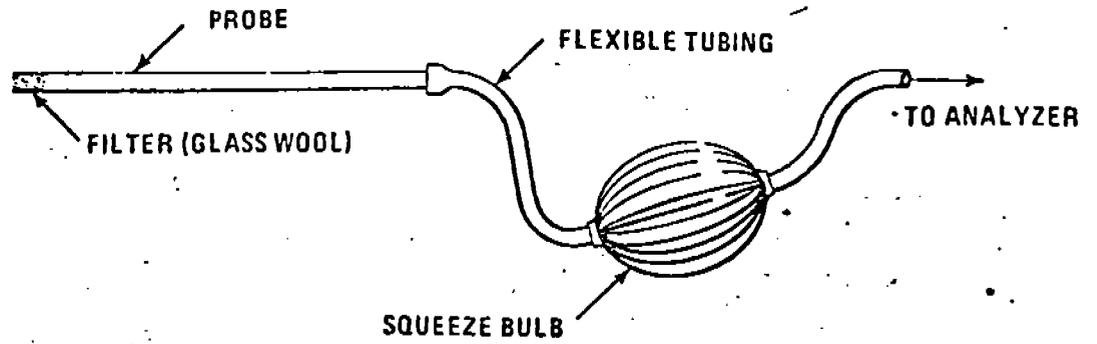


Figure 3-1. Grab-sampling train.

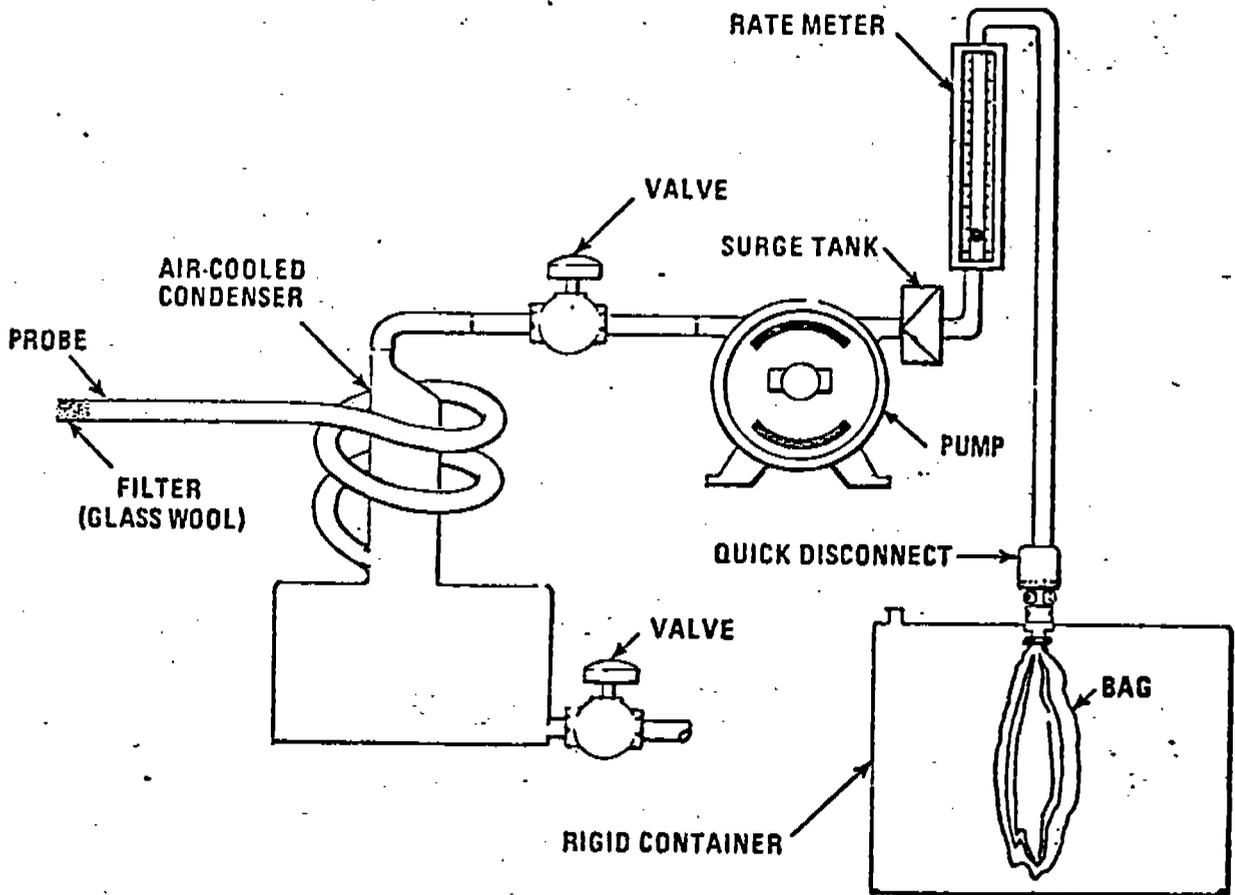


Figure 3-2. Integrated gas-sampling train.

2.2.2 Condenser. An air-cooled or water-cooled condenser, or other condenser that will not remove O_2 , CO_2 , and N_2 , may be used to remove excess moisture which would interfere with the operation of the pump and flow meter.

2.2.3 Valve. A needle valve is used to adjust sample gas flow rate.

2.2.4 Pump. A leak-free, diaphragm-type pump, or equivalent, is used to transport sample gas to the flexible bag. Install a small surge tank between the pump and rate meter to eliminate the pulsation effect of the diaphragm pump on the rotameter.

2.2.5 Rate Meter. The rotameter, or equivalent rate meter, used should be capable of measuring flow rate to within ± 2 percent of the selected flow rate. A flow rate range of 50 to 1000 cm^3/min is suggested.

2.2.6 Flexible Bag. Any leak-free plastic (e.g., Tedlar, Mylar, Teflon) or plastic-coated aluminum (e.g., aluminized Mylar) bag, or equivalent, having a capacity consistent with the selected flow rate and time length of the test run, may be used. A capacity in the range of 65 to 90 liters is suggested.

To leak-check the bag, connect it to a water manometer and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. An alternative leak-check method is to pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O) and allow to stand overnight. A deflated bag indicates a leak.

2.2.7 Pressure Gauge. A water-filled U-tube manometer, or equivalent, of about 25 cm (12 in.) is used for the flexible bag leak-check.

2.2.8 Vacuum Gauge. A mercury manometer, or equivalent, of at least 760 mm Hg (30 in. Hg) is used for the sampling train leak-check.

2.3 Analysis. For Orsat and Fyrite analyzer maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

2.3.1 Dry Molecular Weight Determination. An Orsat analyzer or Fyrite type combustion gas analyzer may be used.

2.3.2 Emission Rate Correction Factor or Excess Air Determination. An Orsat analyzer must be used. For low CO_2 (less than 4.0 percent) or high O_2 (greater than 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions.

3. Dry Molecular Weight Determination

Any of the three sampling and analytical procedures described below may be used for determining the dry molecular weight.

3.1 Single-Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall either be at the centroid of the cross section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

3.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. If an Orsat analyzer is used, it is recommended that the analyzer be leak-checked by following the procedure in Section 5; however, the leak-check is optional.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer and immediately analyze it for percent CO_2 and percent O_2 . Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2 from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.1.4 Repeat the sampling, analysis, and calculation procedures, until the dry molecular weights of any three grab samples differ from their mean by no more than 0.3 g/mole (0.3 lb/mole). Average these three molecular weights, and report the results to the nearest 0.1 g/mole (lb/mole).

3.2 Single-Point, Integrated Sampling and Analytical Procedure.

3.2.1 The sampling point in the duct shall be located as specified in Section 3.1.1.

3.2.2 Leak-check (optional) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (optional) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then turning off the pump. The vacuum should remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

3.2.3 Sample at a constant rate. The sampling run should be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collection of at least 30 liters (1.00 ft³) of sample gas is recommended; however, smaller volumes may be collected, if desired.

3.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. Within 8 hours after the sample is taken, analyze it for percent CO_2 and percent O_2 using either an Orsat analyzer or a Fyrite-type combustion gas analyzer. If an Orsat analyzer is used, it is recommended that the Orsat leak-check described in Section 5 be performed before this determination; however, the check is optional. Determine the percentage of the gas that is N_2 and CO by subtracting the sum of the percent CO_2 and percent O_2

from 100 percent. Calculate the dry molecular weight as indicated in Section 6.3.

3.2.5 Repeat the analysis and calculation procedures until the individual dry molecular weights for any three analyses differ from their mean by no more than 0.3 g/mole (0.3 lb/mole). Average these three molecular weights, and report the results to the nearest 0.1 g/mole (0.1 lb/mole).

3.3 Multi-Point, Integrated Sampling and Analytical Procedure.

3.3.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used for all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to approval of the Administrator.

3.3.2 Follow the procedures outlined in Sections 3.2.2 through 3.2.5, except for the following: traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

TIME	TRAVERSE PT.	Q 1pm	% DEV. ^a
AVERAGE			

$$^a \% \text{ DEV} = \left(\frac{Q - Q_{\text{avg}}}{Q_{\text{avg}}} \right) 100 \quad (\text{MUST BE } \leq 10\%)$$

Figure 3-3. Sampling rate data.

4.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Draw a sample into the analyzer. For emission rate correction factor determination, immediately analyze the sample, as outlined in Sections 4.1.4 and 4.1.5, for percent CO_2 or percent O_2 . If excess air is desired, proceed as follows: (1) immediately analyze the sample, as in Sections 4.1.4 and 4.1.5, for percent CO_2 , O_2 , and CO ; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2 , percent O_2 , and percent CO from 100 percent; and (3) calculate percent excess air as outlined in Section 6.2.

4.1.4 To ensure complete absorption of the CO_2 , O_2 , or if applicable, CO , make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.1.5 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis. NOTE.—Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis. Although in most cases only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.2 Single-Point, Integrated Sampling and Analytical Procedure.

4.2.1 The sampling point in the duct shall be located as specified in Section 4.1.1.

4.2.2 Leak-check (mandatory) the flexible bag as in Section 2.2.6. Set up the equipment as shown in Figure 3-2. Just prior to sampling, leak-check (mandatory) the train by placing a vacuum gauge at the condenser inlet, pulling a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet at the quick disconnect, and then

4. Emission Rate Correction Factor or Excess Air Determination

NOTE.—A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determination, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight.

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.

4.1 Single-Point, Grab Sampling and Analytical Procedure.

4.1.1 The sampling point in the duct shall either be at the centroid of the cross-section or at a point no closer to the walls than 1.00 m (3.3 ft), unless otherwise specified by the Administrator.

4.1.2 Set up the equipment as shown in Figure 3-1, making sure all connections ahead of the analyzer are tight and leak-free. Leak-check the Orsat analyzer according to the procedure described in Section 5. This leak-check is mandatory.

turning off the pump. The vacuum shall remain stable for at least 0.5 minute. Evacuate the flexible bag. Connect the probe and place it in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line. Next, connect the bag and make sure that all connections are tight and leak free.

4.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 30 liters (1.00 ft³) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

4.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Sections 4.2.5 through 4.2.7). The Orsat analyzer must be leak-checked (see Section 5) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 4.2.5 through 4.2.7) for percent CO_2 , O_2 , and CO ; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2 , percent O_2 , and percent CO from 100 percent; (3) calculate percent excess air, as outlined in Section 6.2.

4.2.5 To ensure complete absorption of the CO_2 , O_2 , or if applicable, CO , make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.)

4.2.6 Repeat the analysis until the following criteria are met:

4.2.6.1 For percent CO_2 , repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average the three acceptable values of percent CO_2 and report the results to the nearest 0.1 percent.

4.2.6.2 For percent O_2 , repeat the analytical procedure until the results of any three analyses differ by no more

than (a) 0.3 percent by volume when O₂ is less than 15.0 percent or (b) 0.2 percent by volume when O₂ is greater than 15.0 percent. Average the three acceptable values of percent O₂ and report the results to the nearest 0.1 percent.

4.2.6.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO and report the results to the nearest 0.1 percent.

4.2.7 After the analysis is completed, leak-check (mandatory) the Orsat analyzer once again, as described in Section 5. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis. Note: Although in most instances only CO₂ or O₂ is required, it is recommended that both CO₂ and O₂ be measured, and that Citation 5 in the Bibliography be used to validate the analytical data.

4.3 Multi-Point, Integrated Sampling and Analytical Procedure.

4.3.1 Both the minimum number of sampling points and the sampling point location shall be as specified in Section 3.3.1 of this method. The use of fewer points than specified is subject to the approval of the Administrator.

4.3.2 Follow the procedures outlined in Sections 4.2.2 through 4.2.7, except for the following: Traverse all sampling points and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3.

5. Leak-Check Procedure for Orsat Analyzers

Moving an Orsat analyzer frequently causes it to leak. Therefore, an Orsat analyzer should be thoroughly leak-checked on site before the flue gas sample is introduced into it. The procedure for leak-checking an Orsat analyzer is:

5.1.1 Bring the liquid level in each pipette up to the reference mark on the capillary tubing and then close the pipette stopcock.

5.1.2 Raise the leveling bulb sufficiently to bring the confining liquid meniscus onto the graduated portion of the burette and then close the manifold stopcock.

5.1.3 Record the meniscus position.

5.1.4 Observe the meniscus in the burette and the liquid level in the pipette for movement over the next 4 minutes.

5.1.5 For the Orsat analyzer to pass the leak-check, two conditions must be met.

5.1.5.1 The liquid level in each pipette must not fall below the bottom of the capillary tubing during this 4-minute interval.

5.1.5.2 The meniscus in the burette must not change by more than 0.2 ml during this 4-minute interval.

5.1.6 If the analyzer fails the leak-check procedure, all rubber connections and stopcocks should be checked until the cause of the leak is identified. Leaking stopcocks must be disassembled, cleaned, and regreased. Leaking rubber connections must be replaced. After the analyzer is reassembled, the leak-check procedure must be repeated.

6. Calculations

6.1 Nomenclature.

M_d = Dry molecular weight, g/g-mole (lb/lb-mole).

%FA = Percent excess air.

%CO₂ = Percent CO₂ by volume (dry basis).

%O₂ = Percent O₂ by volume (dry basis).

%CO = Percent CO by volume (dry basis).

%N₂ = Percent N₂ by volume (dry basis).

%O₂ = Ratio of O₂ to N₂ in air, 7/8.

0.264 = Ratio of molecular weight of N₂ or CO, divided by 100.

0.280 = Molecular weight of O₂ divided by 100.

0.440 = Molecular weight of CO₂ divided by 100.

6.2 Percent Excess Air. Calculate the percent excess air (if applicable) by substituting the appropriate values of percent O₂, CO, and N₂ (obtained from Section 4.1.3 or 4.2.4) into Equation 3-1.

$$\%EA = \left[\frac{\%O_2 - 0.5\%CO}{0.264\%N_2(\%O_2 - 0.5\%CO)} \right] 100$$

Equation 3-1

NOTE.—The equation above assumes that ambient air is used as the source of O₂ and that the fuel does not contain appreciable amounts of N₂ (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N₂ are present (coal, oil, and natural gas do not contain appreciable amounts of N₂) or when oxygen enrichment is used, alternate methods, subject to approval of the Administrator, are required.

6.3 Dry Molecular Weight. Use Equation 3-2 to calculate the dry molecular weight of the stack gas

$$M_d = 0.440(\%CO_2) + 0.320(\%O_2) + 0.260(\%N_2 + \%CO)$$

Equation 3-2

NOTE.—The above equation does not consider argon in air (about 0.9 percent, molecular weight of 39.7). A negative error of about 0.4 percent is introduced. The tester may opt to include argon in the analysis using procedures subject to approval of the Administrator.

7. Bibliography

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METHOD 4—DETERMINATION OF MOISTURE CONTENT IN STACK GASES

1. Principle and Applicability

1.1 Principle. A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

1.2 Applicability. This method is applicable for determining the moisture content of stack gas.

Two procedures are given. The first is a reference method, for accurate determinations of moisture content (such as are needed to calculate emission data). The second is an approximation method, which provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content, e.g., drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc., are also acceptable.

The reference method is often conducted simultaneously with a pollutant emission measurement run; when it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent; these calculations shall not be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, U.S. Environmental Protection Agency, to be capable of yielding results within 1 percent H₂O of the reference method.

NOTE.—The reference method may yield questionable results when applied to saturated gas streams or to streams that contain water droplets. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor (capable of measuring to ±1° C (±2° F)) to the reference method probe. Measure the stack gas temperature at each traverse point (see Section 2.2.1) during the reference method traverse; calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternate methods, subject to the approval of the Administrator, shall be used.

2. Reference Method

The procedure described in Method 5 for determining moisture content is acceptable as a reference method.

2.1 Apparatus. A schematic of the sampling train used in this reference method is shown in Figure 4-1. All components shall be maintained and calibrated according to the procedure outlined in Method 5.

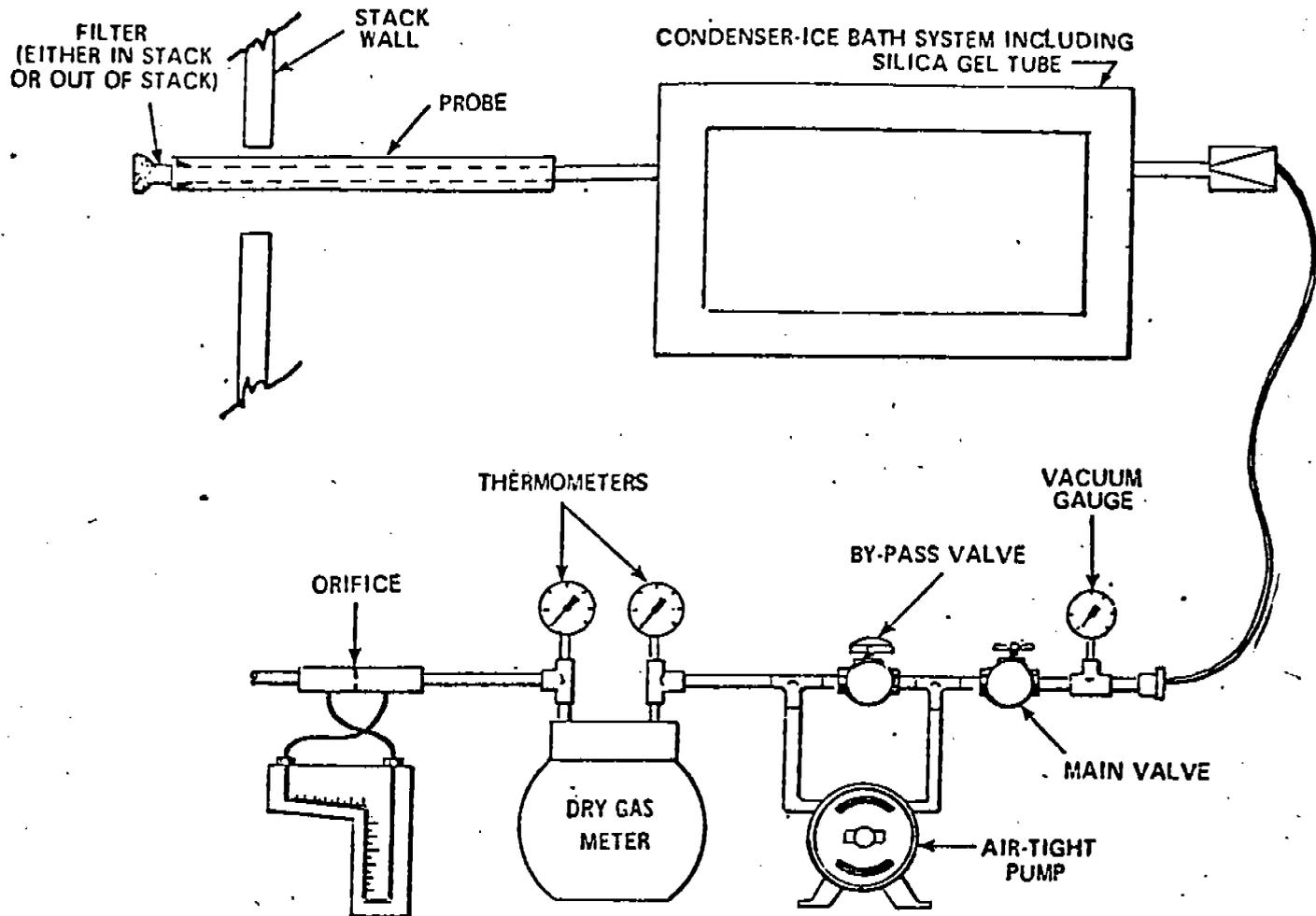


Figure 4-1. Moisture sampling train-reference method.

2.1.1 Probe. The probe is constructed of stainless steel or glass tubing, sufficiently heated to prevent water condensation, and is equipped with a filter, either in-stack (e.g., a plug of glass wool inserted into the end of the probe) or heated out-stack (e.g., as described in Method 5), to remove particulate matter.

When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.

2.1.2 Condenser. The condenser consists of four impingers connected in series with ground glass, leak-free fittings or any similarly leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 centimeter ($\frac{1}{2}$ inch) ID glass tube extending to about 1.3 cm ($\frac{1}{2}$ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator.

The first two impingers shall contain known volumes of water, the third shall be empty, and the fourth shall contain a known weight of 6- to 16-mesh indicating type silica gel, or equivalent desiccant. If the silica gel has been previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received. A thermometer, capable of measuring temperature to within 1°C (2°F), shall be placed at the outlet of the fourth impinger, for monitoring purposes.

Alternatively, any system may be used (subject to the approval of the Administrator) that cools the sample gas stream and allows measurement of both the water that has been condensed and the moisture leaving the condenser, each to within 1 ml or 1 g. Acceptable means are to measure the condensed water, either gravimetrically or volumetrically, and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures, or (2) passing

the sample gas stream through a tared silica gel (or equivalent desiccant) trap, with exit gases kept below 20°C (68°F), and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump, to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

2.1.3 Cooling System. An ice bath container and crushed ice (or equivalent) are used to aid in condensing moisture.

2.1.4 Metering System. This system includes a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter capable of measuring volume to within 2 percent, and related equipment as shown in Figure 4-1. Other metering systems, capable of maintaining a constant sampling rate and determining sample gas volume, may be used, subject to the approval of the Administrator.

2.1.5 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.6 Graduated Cylinder and/or Balance. These items are used to measure condensed water and moisture caught in the silica gel to within 1 ml or 0.5 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. These balances are suitable for use here.

2.2 Procedure. The following procedure is written for a condenser system (such as the impinger system de-

scribed in Section 2.1.2) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.

2.2.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used in all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Place known volumes of water in the first two impingers. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.

2.2.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 m³/min (0.75 cfm). When both moisture content and pollutant emission rate are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

2.2.3 Set up the sampling train as shown in Figure 4-1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of about 120°C (245°F), to prevent water condensation ahead of the condenser; allow time for the temperatures to stabilize. Place crushed ice in the ice bath container. It is recommended, but not required, that a leak check be done, as follows: Disconnect the probe from the first impinger or

RULES AND REGULATIONS

	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
DIFFERENCE		

Figure 4-3. Analytical data—reference method.

2.3.1 Nomenclature.

- B_w = Proportion of water vapor, by volume, in the gas stream.
 M_w = Molecular weight of water, 18.0 g/g-mole (15.0 lb/lb-mole).
 P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).
 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
 R = Ideal gas constant, 0.08206 (mm Hg) (m³)/(g-mole) (°K) for metric units and 21.85 (in. Hg) (ft³)/(lb-mole) (°R) for English units.
 T_m = Absolute temperature at meter, °K (°R).
 T_{std} = Standard absolute temperature, 293° K (528° R).
 V_m = Dry gas volume measured by dry gas meter, dem (dcf).
 ΔV_m = Incremental dry gas volume measured by dry gas meter at each traverse point, dem (dcf).
 $V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
 $V_{w(std)}$ = Volume of water vapor condensed corrected to standard conditions, scm (scf).
 $V_{w(std)}$ = Volume of water vapor collected in silica gel corrected to standard conditions, scm (scf).
 V_c = Final volume of condenser water, ml.
 V_i = Initial volume, if any, of condenser water, ml.
 W_f = Final weight of silica gel or silica gel plus impinger, g.
 W_i = Initial weight of silica gel or silica gel plus impinger, g.
 Y = Dry gas meter calibration factor.
 ρ_w = Density of water, 0.9982 g/ml (0.02201 lb/ml).

2.3.2 Volume of water vapor condensed.

$$V_{w(std)} = \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_w} = K_1 (V_f - V_i) \quad \text{Equation 4-1}$$

where:

- K_1 = 0.001335 m³/ml for metric units
 = 0.04707 ft³/ml for English units

2.3.3 Volume of water vapor collected in silica gel.

$$V_{w(std)} = \frac{(W_f - W_i) R T_{std}}{P_{std} M_w} = K_2 (W_f - W_i) \quad \text{Equation 4-2}$$

where:

- K_2 = 0.001335 m³/g for metric units
 = 0.04715 ft³/g for English units

2.3.4 Sample gas volume.

$$V_{m(std)} = V_m Y \frac{(P_m)(T_{std})}{(P_{std})(T_m)} = K_3 Y \frac{V_m P_m}{T_m} \quad \text{Equation 4-3}$$

where:

- K_3 = 0.356 °K/mm Hg for metric units
 = 17.64 °R/in. Hg for English units

NOTE—If the post-test leak rate (Section 2.2.6) exceeds the allowable rate, correct the value of V_m in Equation 4-3, as described in Section 6.3 of Method 6.

2.3.5 Moisture Content.

$$B_w = \frac{V_{w(std)} + V_{wz(std)}}{V_{w(std)} + V_{wz(std)} + V_m(std)} \quad \text{Equation 4-4}$$

NOTE—In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see Section 1.2), and another based upon the results of the impinger analysis. The lower of these two values of B_w shall be considered correct.

2.3.6 Verification of constant sampling rate. For each time increment, determine the ΔV_m . Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results and repeat the run.

3. Approximation Method

The approximation method described below is presented only as a suggested method (see Section 1.2).

3.1 Apparatus.

3.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter. A plug of glass wool, inserted into the end of the probe, is a satisfactory filter.

3.1.2 Impingers. Two midget impingers, each with 30 ml capacity, or equivalent.

3.1.3 Ice Bath. Container and ice, to aid in condensing moisture in impingers.

3.1.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

3.1.5 Valve. Needle valve, to regulate the sample gas flow rate.

3.1.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.

3.1.7 Volume meter. Dry gas meter, sufficiently accurate to measure the sample volume within 2%, and calibrated over the range of flow rates and conditions actually encountered during sampling.

3.1.8 Rate Meter. Rotameter, to measure the flow rate from 0 to 3 lpm (0 to 0.11 cfm).

3.1.9 Graduated Cylinder, 25 ml.

3.1.10 Barometer. Mercury, aneroid, or other barometer, as described in Section 2.1.5 above.

3.1.11 Vacuum Gauge. At least 760 mm Hg (30 in. Hg) gauge, to be used for the sampling leak check.

3.2 Procedure.

3.2.1 Place exactly 5 ml distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4-4. Leak check the train by placing a vacuum gauge at the inlet to the first impinger and drawing a vacuum of at least 250 mm Hg (10 in. Hg), plugging the outlet of the rotameter, and then turning off the pump. The vacuum shall remain constant for at least one minute. Carefully release the vacuum gauge before unplugging the rotameter end.

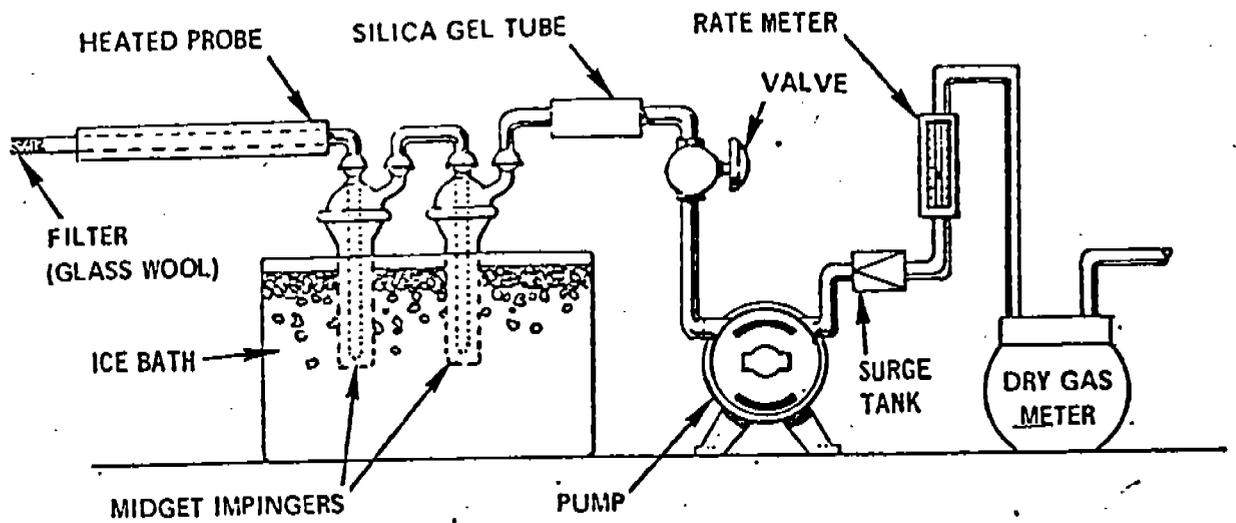


Figure 4-4. Moisture-sampling train - approximation method.

LOCATION _____ COMMENTS _____

TEST _____

DATE _____

OPERATOR _____

BAROMETRIC PRESSURE _____

CLOCK TIME	GAS VOLUME THROUGH METER, (Vm), m ³ (ft ³)	RATE METER SETTING m ³ /min. (ft ³ /min.)	METER TEMPERATURE, °C (°F)

Figure 4-5. Field moisture determination - approximation method.

3.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 lpm (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft³) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-5.

3.2.3 After collecting the sample, combine the contents of the two impingers and measure the volume to the nearest 0.5 ml.

3.3 Calculations. The calculation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content, for the purpose of determining isokinetic sampling rate settings.

3.3.1 Nomenclature:

B_{wa} = Approximate proportion, by volume, of water vapor in the gas stream leaving the second impinger, 0.025.

B_{ws} = Water vapor in the gas stream, proportion by volume.

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole)

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter.

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 (mm Hg) (m³)/g-mole) (°K) for metric units and 21.65 (in. Hg) (ft³/lb-mole) (°R) for English units.

T_m = Absolute temperature at meter, °K (°R)

T_{std} = Standard absolute temperature, 293° K (528° R)

V_f = Final volume of impinger contents, ml.

V_i = Initial volume of impinger contents, ml.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

3.3.2 Volume of water vapor collected.

$$V_{wc} = \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_w} = K_1 (V_f - V_i)$$

Equation 4-5

where:

K_1 = 0.001333 m³/ml for metric units
= 0.04707 ft³/ml for English units.

3.3.3 Gas volume.

$$V_{m(std)} = V_m \left(\frac{P_m}{P_{std}} \right) \left(\frac{T_{std}}{T_m} \right) = K_2 \frac{V_m P_m}{T_m}$$

Equation 4-6

where:

K_2 = 0.3555 °K/mm Hg for metric units
= 17.64 °R/in. Hg for English units

3.3.4 Approximate moisture content.

$$B_{wa} = \frac{V_{wc}}{V_{wc} + V_{m(std)}} + B_{ws} = \frac{V_{wc}}{V_{wc} + V_{m(std)}} + (0.025)$$

Equation 4-7

4. Calibration

4.1 For the reference method, calibrate equipment as specified in the following sections of Method 5: Section 5.3 (metering system); Section 5.5 (temperature gauges); and Section 5.7 (barometer). The recommended leak check of the metering system (Section 5.6 of Method 5) also applies to the reference method. For the approximation method, use the procedures outlined in Section 5.1.1 of Method 6 to calibrate the metering system, and the procedure of Method 5, Section 5.7 to calibrate the barometer.

5. Bibliography

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METHOD 5—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature in the range of 120±14° C (248±25° F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator, U.S. Environmental Protection Agency, for a particular application. The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 5-1. Complete construction details are given in APTD-0581 (Citation 2 in Section 7); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576 (Citation 3 in Section 7). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

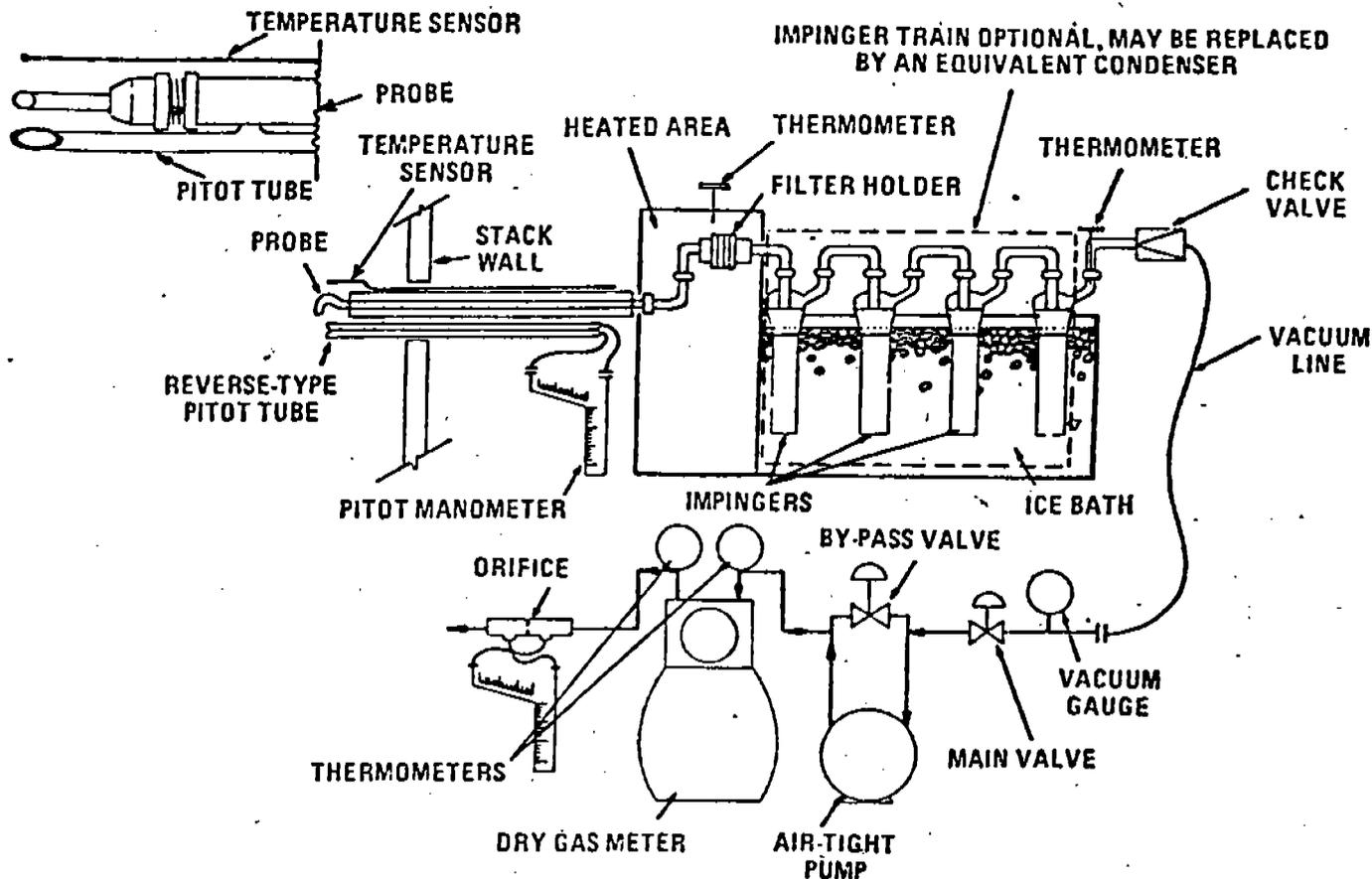


Figure 5.1. Particulate-sampling train.

2.1.1 Probe Nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper shall be $\leq 30^\circ$ and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing; other materials of construction may be used, subject to the approval of the Administrator.

A range of nozzle sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm ($1/8$ to $1/2$ in.)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm ($1/16$ in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of $120 \pm 14^\circ \text{C}$ ($248 \pm 25^\circ \text{F}$), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. (The tester may opt to operate the equipment at a temperature lower than that specified.) Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0351 and utilizing the calibration curves of APTD-0376 (or calibrated according to the procedure outlined in APTD-0376) will be considered acceptable.

Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 450°C (800°F) quartz liners shall be used for temperatures between 450 and 900°C (800 and $1,650^\circ \text{F}$). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate is 520°C (968°F), and for quartz it is $1,500^\circ \text{C}$ ($2,700^\circ \text{F}$).

Whenever practicable, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825, or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

2.1.3 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening

plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Section 4 of Method 2.

2.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

2.1.5 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

2.1.6 Filter Heating System. Any heating system capable of maintaining a temperature around the filter holder during sampling of $120 \pm 14^\circ \text{C}$ ($248 \pm 25^\circ \text{F}$), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0351 may be used.

2.1.7 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with 1.5 cm ($1/2$ in.) ID glass tube extending to about 1.5 cm ($1/2$ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 4.1.3), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A thermometer, capable of measur-

ing temperature to within 1°C (2°F) shall be placed at the outlet of the fourth impinger for monitoring purposes.

Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. Acceptable means are to measure the condensed water either gravimetrically or volumetrically and to measure the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20°C (68°F) and determining the weight gain.

If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

Note.—If a determination of the particulate matter collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individuals, States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

2.1.8 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volume to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0351 or APTD-0376 may be used provided that the specifications of this method are met.

2.1.9 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is

* Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.10 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type 5 pitot tube openings (see Method 2, Figure 2-7). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) of stainless steel, Nylon, Teflon, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dishes. For filter samples, glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

2.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

2.3 Analysis. For analysis, the following equipment is needed:

2.3.1 Glass Weighing Dishes.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 g.

2.3.5 Beakers. 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

3. Reagents

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (≤ 0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard method D 2956-71. Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2. Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Water. When analysis of the material caught in the impingers is required, distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

3.1.4 Crushed Ice.

3.1.5 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.2 Sample Recovery. Acetone—reagent grade, ≤ 0.001 percent residue, in glass bottles—is required. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks shall be run prior to field use and only acetone with low blank values (≤ 0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis. Two reagents are required for the analysis:

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

4. Procedure

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All the components shall be maintained and calibrated according to the procedure described in APTD-6576, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Desiccate the filters at 20±5.6° C (68±10° F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., <0.5 mg change from previous weighing; record results to the nearest 0.1 mc. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105° C (220° F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particulate sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of Method 2).

Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 min (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed liter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

When glass liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 200° C (400° F) and an asbestos string gasket when temperatures are higher. See APTD-6576 for

details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Set up the train as in Figure 5-1, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-6576) to avoid possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

Place crushed ice around the impingers.

4.1.4 Leak-Check Procedures.

4.1.4.1 Pretest Leak-Check. A pretest leak-check is recommended, but not required. If the tester opts to conduct the pretest leak-check, the following procedure shall be used.

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 350 mm Hg (15 in. Hg) vacuum.

NOTE.—A lower vacuum may be used, provided that it is not exceeded during the test.

If an asbestos string is used, do not connect the probe to the train during the leak-check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 350 mm Hg (15 in. Hg) vacuum (see Note immediately above). Then connect the probe to the train and leak-check at about 25 mm Hg (1 in. Hg) vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 350 mm Hg (15 in. Hg) vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.0057 m³/min (0.02 cfm), whichever is less, are unacceptable.

The following leak-check instructions for the sampling train described in APTD-6576 and APTD-6581 may be helpful. Start the pump with bypass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse direction of bypass valve; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check as shown below and start over.

When the leak-check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and silica gel from being entrained backward into the third impinger.

4.1.4.2 Leak-Checks During Sample Run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak-check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure outlined in Section 4.1.4.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.0057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

Immediately after component changes, leak-checks are optional; if such leak-checks are done, the procedure outlined in Section 4.1.4.1 above shall be used.

4.1.4.3 Post-test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.1.4.1, except that it shall be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.0057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 170±14° C (345±25° F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

For each run, record the data required on a data sheet such as the one shown in Figure 5-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted.

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fiting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow the Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter which is brushed from the probe. Run the brush through the probe three times or more until no visible particulate matter is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

It is recommended that two people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints have been wiped clean of silicone grease, clean the inside of the front half of the filter holder by rubbing the surfaces with a nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

Container No. 3. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container No. 3 in Section 4.3.

Impinger Water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid which is in the first three impingers to within ±1 ml by using a graduated cylinder or by weighing it to within ±0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see Note, Section 2.1.7).

If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

Whenever possible, containers should be shipped in such a way that they remain upright at all times.

4.3 Analysis. Record the data required on a sheet such as the one shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Plant _____

Date _____

Run No. _____

Filter No. _____

Amount liquid lost during transport _____

Acetone blank volume, ml _____

Acetone wash volume, ml _____

Acetone blank concentration, mg/mg (equation 5-4) _____

Acetone wash blank, mg (equation 5-5) _____

CONTAINER NUMBER	WEIGHT OF PARTICULATE COLLECTED, mg		
	FINAL WEIGHT	TARE WEIGHT	WEIGHT GAIN
1			
2			
TOTAL			
	Less acetone blank		
	Weight of particulate matter		

	VOLUME OF LIQUID WATER COLLECTED	
	IMPINGER VOLUME, ml	SILICA GEL WEIGHT, g
FINAL		
INITIAL		
LIQUID COLLECTED		
TOTAL VOLUME COLLECTED		g* ml

* CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER (1g/ml).

$$\frac{\text{INCREASE, g}}{1 \text{ g/ml}} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

Alternatively, the sample may be oven dried at 105° C (220° F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also opt to oven dry the sample at 105° C (220° F) for 2 to 3 hours, weigh the sample, and use this weight as a final weight.

Container No. 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Container No. 3. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

NOTE.—At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest

0.02 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

5.2 Pitot Tube. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Section 4 of Method 2.

5.3 Metering System. Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-6576. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values. Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.0007 m³/min (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed 0.0007 m³/min (0.02 cfm).

After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as outlined in APTD-6576.

Alternative procedures, e.g., using the orifice meter coefficients, may be used, subject to the approval of the Administrator.

NOTE.—If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

5.4 Probe Heater Calibration. The probe heating system shall be calibrated before its initial use in the field according to the procedure outlined in APTD-6576. Probes constructed according to APTD-6581 need not be calibrated if the calibration curves in APTD-6576 are used.

5.5 Temperature Gauges. Use the procedure in Section 4.8 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

5.6 Leak Check of Metering System Shown in Figure 5-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5-4): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

5.7 Barometer. Calibrate against a mercury barometer.

6. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

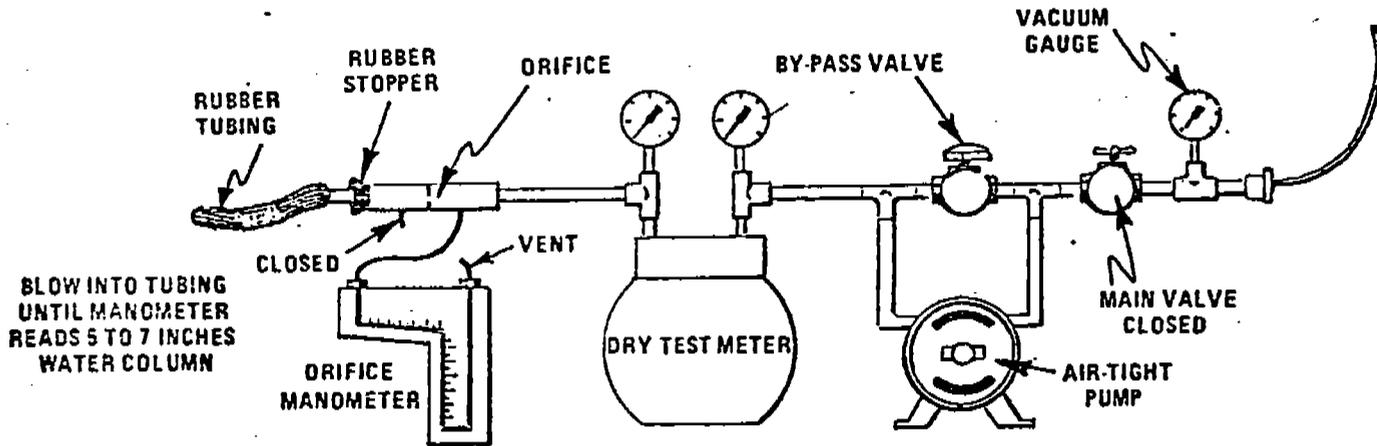


Figure 5-4. Leak check of meter box.

- 6.1 Nomenclature
- A_c = Cross-sectional area of nozzle, m² (ft²).
- B_w = Water vapor in the gas stream, proportion by volume.
- C_a = Acetone blank residue concentrations, mg/g.
- c_p = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
- I = Percent of isokinetic sampling.
- L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.
- L_i = Individual leakage rate observed during the leak check conducted prior to the "ith" component change (i=1, 2, 3, . . . n), m³/min (cfm).
- L_p = Leakage rate observed during the post-test leak check, m³/min (cfm).
- M_a = Total amount of particulate matter collected, mg.
- M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
- m_a = Mass of residue of acetone after evaporation, mg.
- P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).
- P_s = Absolute stack gas pressure, mm Hg (in. Hg).
- P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

- R = Ideal gas constant, 0.08206 mm Hg-m³/K-g-mole (21.85 in. Hg-ft³/R-lb-mole).
- T_a = Absolute average dry gas meter temperature (see Figure 5-2), °K (°R).
- T_s = Absolute average stack gas temperature (see Figure 5-2), °K (°R).
- T_{std} = Standard absolute temperature, 293° K (528° R).
- V_a = Volume of acetone blank, ml.
- V_{aw} = Volume of acetone used in wash, ml.
- V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.
- V_m = Volume of gas sample as measured by dry gas meter, dcm (dscf).
- V_{m(i+d)} = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
- V_{w(i+d)} = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
- V_s = Stack gas velocity, calculated by Method 2, Equation 2-9, using data obtained from Method 5, m/sec (ft/sec).
- W_a = Weight of residue in acetone wash, mg.
- Y = Dry gas meter calibration factor.
- ΔH = Average pressure differential across the orifice meter (see Figure 5-2), mm H₂O (in. H₂O).
- ρ_a = Density of acetone, mg/ml (see label on bottle).
- ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).
- τ = Total sampling time, min.

- t₁ = Sampling time interval, from the beginning of a run until the first component change, min.
- t₂ = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.
- t_n = Sampling time interval, from the final (nth) component change until the end of the sampling run, min.
- 13.6 = Specific gravity of mercury.
- 60 = Sec/min.
- 100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg or 68° F, 29.92 in. Hg) by using Equation 5-1.

$$V_{m(i+d)} = V_m Y \left(\frac{T_{std}}{T_m} \right) \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}} \right] = K_1 V_m Y \frac{P_{bar} + (\Delta H/13.6)}{T_m}$$

Equation 5-1

where:
 $m_1 = 0.2835 \text{ g/mm Hg}$ for metric units
 $= 17.64 \text{ g/in. Hg}$ for English units

NOTE.—Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds L_1 . If L_1 or L_2 exceeds L_0 , Equation 5-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 5-1 with the expression:

$$V_m = (L_1 - L_0)\theta$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 5-1 by the expression:

$$\left[V_m - (L_1 - L_0)\theta_1 - \sum_{i=2}^n (L_i - L_0)\theta_i - (L_2 - L_0)\theta_2 \right]$$

and substitute only for those leakage rates (L_1 or L_2) which exceed L_0 .

6.4 Volume of water vapor.

Equation 5-2

$$V_{w(sld)} = V_{1s} \left(\frac{p_w}{M_w} \right) \left(\frac{RT_{sld}}{P_{sld}} \right) = K_2 V_{1s}$$

where:
 $K_2 = 0.001333 \text{ m}^3/\text{ml}$ for metric units
 $= 0.04707 \text{ ft}^3/\text{ml}$ for English units.

6.5 Moisture Content.

$$B_{w,s} = \frac{V_{w(sld)}}{V_m(sld) + V_{w(sld)}}$$

Equation 5-3

$$I = \frac{100 T_m [K_1 V_{1s} + (V_m/T_m) (P_{bar} + \Delta H/13.6)]}{60 \theta v_s P_s A_s}$$

Equation 5-7

where:
 $K_1 = 0.03454 \text{ mm Hg} - \text{m}^3/\text{ml} - ^\circ\text{K}$ for metric units.
 $= 0.002609 \text{ in. Hg} - \text{ft}^3/\text{ml} - ^\circ\text{R}$ for English units.

6.11.2 Calculation From Intermediate Values.

$$I = \frac{T_m V_m(sld) P_{sld} 100}{T_{sld} \theta A_s P_s 60 (1 - B_{w,s})}$$

$$= K_3 \frac{T_m V_m(sld)}{P_s V_s A_s \theta (1 - B_{w,s})}$$

Equation 5-8

where:
 $K_3 = 4.320$ for metric units
 $= 0.06450$ for English units.

6.12 Acceptable Results. If 90 percent $< I < 110$ percent, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or if I is less than 90 percent, the Administrator may opt to accept the results. Use Citation 4 to make judgments. Otherwise, reject the results and repeat the test.

7. Bibliography

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4. Smith, W. S., R. T. Shipchra, and W. F. Todd. A Method of Interpreting Stack Sampling Data. Paper Presented at the 63d Annual Meeting of the Air Pollution Control Association, St. Louis, Mo. June 14-19, 1970.
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7. Shipchra, R. T. Adjustments to the EPA Nomenclature for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News 5:4-11. October, 1974.

NOTE.—In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values of $B_{w,s}$ shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the Note of Section 1.2 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-2 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is $\pm 1^\circ\text{C}$ ($^\circ\text{F}$).

6.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a}$$

Equation 5-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_{av} \rho_a$$

Equation 5-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 5-3). NOTE.—Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$c_p = (0.001 \text{ g/mg}) (m_p/V_m(sld))$$

Equation 5-6

6.10 Conversion Factors:

From	To	Multiply by
scf	m ³	0.02832
g/ft ³	g/m ³	15.43
g/ft ³	lb/ft ³	2.205 × 10 ⁻³
g/ft ³	g/m ³	25.31

6.11 Isokinetic Variation.

6.11.1 Calculation From Raw Data.

8. Volaro, R. F. A Survey of Commercially Available Instrumentation for the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. November, 1976 (unpublished paper).

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METHOD 6—DETERMINATION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES

1. Principle and Application

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The sulfuric acid mist (including sulfur trioxide) and the sulfur dioxide are separated. The sulfur dioxide fraction is measured by the barium-thorium titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources. The minimum detectable limit of the method has been determined to be 3.4 milligrams (mg) of SO₂/m³ (2.12 × 10⁻⁴ lb/ft³). Although no upper limit has been established, tests have shown that concentrations as high as 80,000 mg/m³ of SO₂ can be collected efficiently in two midsize impingers, each containing 15 milliliters of 3 percent hydrogen peroxide, at a rate of 1.0 lpm for 20 minutes. Based on theoretical calculations, the upper concentration limit in a 20-liter sample is about 53,300 mg/m³.

Possible interferences are free ammonia, water-soluble cations, and fluorides. The cations and fluorides are removed by glass wool filters and an isopropanol bubbler, and hence do not affect the SO₂ analysis. When samples are being taken from a gas stream with high concentrations of very fine metallic fumes (such as in inlets to control devices), a high-efficiency glass fiber filter must be used in place of the glass wool plug (i.e., the one in the probe) to remove the cation interferences.

Free ammonia interferes by reacting with SO₂ to form particulate sulfate and by reacting with the indicator. If free ammonia is present (this can be determined by knowledge of the process and noticing white particulate matter in the probe and isopropanol bubbler), alternative methods, subject to the approval of the Administrator, U.S. Environmental Protection Agency, are required.

2. Apparatus