

## Draft Guidance for Acceptable Procedures for Archiving PM<sub>2.5</sub> Samples

### 1. Introduction:

This guidance is a draft, assembled from comments by PM<sub>2.5</sub> researchers with many years of experience in the particulate field, and is intended to provide guidance for long-term storage of SLAMS FRM filter samples that have already been analyzed for mass. Little information is available in the peer-reviewed literature on this subject. The purpose of this guidance is to provide an approach for the long-term storage that will maintain sample integrity after its initial mass analysis, such that a future analysis will provide the same concentration value as the initial one, or that the sample will be available for a different analysis in the future.

The sample preservation steps for the preservation of PM<sub>2.5</sub> mass and the different chemical species collected by the PM<sub>2.5</sub> FRM are given below in the recommendation section. However, a single, one-size-fits-all approach to sample storage may not be appropriate or successful, depending on the desired analysis.

### 2. Recommendations:

#### a. Routine PM<sub>2.5</sub> SLAMS samples by State and local agencies.

- (1) Store/archive in vapor-tight petri dishes or petri slide dishes. [Currently, it is believed that means Millipore instead of Gelman, but use the criteria of tightness-if-fit, stated in the previous sentence, to decide.] (Chow, 1995)
- (2) As much as is practical, keep the filter in the same petri container from before collection and especially from post-sampling conditioning, through long-term storage.
- (3) Place the filter into the petri container under low RH conditions. This will minimize concerns about water freezing during refrigerated or freezing storage conditions.
- (4) Do preliminary QC checks to ensure that significant weight change is not occurring over time. Checks should include at least two blanks and at least four previously weighed, loaded filters each quarter, as atmospheric composition changes during the year the composition of the collected sample changes and the effect of that change on sample integrity during storage needs to be considered. One loading should be at the high end, another at the low end, and the other two in the middle of typically experienced loadings. Initial frequency might be one weighing per week for the first month, then one per month for the first quarter, and then quarterly, if no significant change is found. Since re-weighing the same filter may result in losses or gains due to handling, possibly due to multiple temperature changes, do not re-weigh the same filter each time, over time. Instead, after

two re-weighings from the archive, select another pair of blanks and of light and heavily loaded exposed filters. Select from the same batch if the batches are large or from different batches if the batches are small. If your checks determine significant change, take steps to minimize change by preventing vapor exchange. To start, use the acceptance values for your laboratory's performance of the PM<sub>2.5</sub> FRM: lab ( $\pm 15 \mu\text{g}$ ) and field ( $\pm 30 \mu\text{g}$ ) blank as a first estimate on upper bounds of a control chart; your laboratory's practical Minimum Detection Limit; or a % of the mass. One suggestion was 15%. Examples include sealing in petri containers with Teflon tape and/or wrapping in aluminum foil. The more layers, the greater the barrier to change (vapor exchange). Continue, document, and track the QA weights, decreasing frequency as data indicate.

- (5) Store/archive in frost-free refrigerator ( $< 4\text{EC}$ ).
- (6) Storage/archiving at  $< 4\text{EC}$  is adequate. If a freezer is used, it should also be frost-free to minimize problems from moisture effects, such as condensation or crystallization. However, several experienced PM<sub>2.5</sub> research scientists recommend against freezing due to concerns that the samples will be changed even by the small amount of water content in the slides.
- (7) Filters should be stored in the dark. Wrap the vapor sealed container of one or more petri dishes in aluminum foil.
- (8) Filters that need to be re-analyzed should be equilibrated (= conditioned) back to room temperature under low RH conditions ( $< 40\% \text{ RH}$ ), BEFORE opening the petri dish or taking off any vapor sealing tape or other material functioning in that capability (that is, acting as a vapor barrier). Since re-weighing should be done in the conditioning room where weighing also occurs, and this room is kept between 30% and 40% RH, or lower, for the PM<sub>2.5</sub> FRM, this location is recommended for this step.

Note, the above approach is likely appropriate for mass, organic material and inorganic compounds by bulk analysis methods, but does not include biological material, especially ones needed for active biological samples. Basically, sample storage in the short- and long-term should be determined based on the chemical species and analysis (es) of interest, and method of maintaining sample integrity for that or those species, rather than just storing based on one approach.

- b. Routine PM<sub>2.5</sub> PEP samples by the EPA PEP labs.
  - (1) Keep the same for now; see PEP lab SOP's on AMTIC.
  - (2) Should be the same as for 1 above.

- (3) Consider continuing cold (< 4EC) storage for the entire 3 years.
- c. Semi-routing PM<sub>2.5</sub> EPA speciation samples by State and local agencies.
- (1) Same as for 1 above for now, except filters for organic speciation. Filter samples for organic speciation should be maintained at 15EC or below (Cass, 1991). Usually samples intended for organic or elemental carbon analysis are collected on quartz fiber filters. They may be used in combination with denuders, which collect strong acidic or basic gases, to keep these gases away from the quartz fiber filters. Also, ion extracts (typically nitrate, sulfate, chloride, ammonium, sodium, and potassium) from nylon filter sampling and extracts from the denuders are frozen to maintain stability before analysis.
  - (2) Conduct QC checks of filter storage; combine and evaluate with speciation data.
  - (3) Modify requirements as appropriate.
- d. Routine PM<sub>2.5</sub> EPA speciation samples by EPA speciation contract laboratories.
- (1) Same as for 3 above for now.
  - (2) Conduct QC checks of filter storage; combine and evaluate with speciation data.
  - (3) Modify requirements as appropriate.

For guidance on alternative methods of PM<sub>2.5</sub> collection or analysis for specific chemical species, see the EPA OAQPS TTN/AMTIC PM<sub>2.5</sub> Monitoring Information Page at [www.epa.gov/ttn/amtic/amticpm.html](http://www.epa.gov/ttn/amtic/amticpm.html). Once at that page, select the Speciation Information Page. On that page, select “Speciation File Area,” at url location [www.epa.gov/ttn/amtic/pmspec.html](http://www.epa.gov/ttn/amtic/pmspec.html). Of the list of guidance documents on that page, the 1-26-99 document “Guideline on Speciated Particulate Monitoring” probably has the most information and associated references containing considerations for handling specific chemical samples.

Section 4.2 of the Guideline addresses common analyses for specific chemicals collected, the typically used filter media, pretreatment methods, and presampling and preanalysis storage requirements. Also of special interest, Section 5 describes measurement artifacts (see also Actelik and Ormand, 1998) and interferences.

If checks of stored QC blank and/or loaded filters indicate a need for corrective action, likely storage conditions to try modifying include examining the effect of relative humidity during pre- and post-storage equilibration, and/or during storage (less than 100% vs. less than 40%, etc.);

storage temperature (e.g., -15 through -80EC, £ 4EC, - 20EC); glass petri dish versus plastic petri slide (significance of potential plasticizer effects); use of various wrapping tapes (e.g., aluminum foil, zip-lock bags). The quality control checks and procedure changes should look at the potential for gain or loss of mass or selected constituent and the integrity of the filter and sample (e.g., ice crystals, torn filters).

3. References:

- a. Achteлик, G. H. and Ormand, J. (1998). Effects of environmental conditions on particulate nitrate stability during post sampling phase. *In PM<sub>2.5</sub> - A Fine Particle Standard*, Chow, J. and Koutrakis, P., Eds., Air and Waste Management Association, Pittsburgh, PA, pp. 365-374.
- b. Cass, G. (1991). Sources of organic Aerosols. *ES&T*, 25: 1112-1125.
- c. Chow, J. (1995). Measurement methods to determine compliance with ambient air quality standards for suspended particles. *JAWMA*, 45: 320-382.

d. Acknowledgment:

Drs. Ricky Tropp, Judy Chow, John Watson, DRI; Paul Solomon, EPA; Dr. George Allen, Harvard University, School of Public Health.

Comments on this guidance are very welcome and should be based on and cite relevant experience and reference published reports and/or peer-reviewed professional journal articles that document as much as possible of these experiences. Call Mark Shanis at 919/541-1323, fax to 919/541-1903, or email to [shanis.mark@epa.gov](mailto:shanis.mark@epa.gov).

If there is enough communicated interest, a work group and conference call might be set up to facilitate information exchange and consensus building.