

Preparation of Lead Filter Audit Filters from NIST SRM and Teflon Filters

Standard Operating Procedure (SOP)

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1.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) is intended as a guide for the preparation of lead spiked teflon filters for use as laboratory audit filter samples using National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) by an experienced laboratory chemist. This SOP is not intended to address the extraction or analysis of audit filter samples. The method is intended to be used in the development of Pb Analysis Audits for the ambient air monitoring program as described in 40 CFR Part 58 Appendix A Section 3.3.4.2. These filters can only be used for total extraction methods of analysis.

2.0 SUMMARY OF METHOD

This method describes the preparation of 46.2mm teflon filters spiked with varying amounts of lead from NIST SRM 3128¹ using calibrated mechanical pipettes. Filters are placed in holders, wet with an alcohol solution, spiked with a working standard prepared from NIST SRM 3128 using a mechanical pipette, dried in a HEPA-filtered laminar flow hood, and packaged. A subset of the filters prepared is extracted and analyzed to ensure the correct spiking level.

3.0 DEFINITIONS

Pb – Elemental or ionic lead

NIST – National Institute of Standards and Technology

D.I. water - Deionized water

RTI – RTI International

SOP – Standard Operating Procedure

SRM – NIST Standard Reference Material

USEPA – U.S. Environmental Protection Agency

v/v – volume to volume ratio

4.0 INTERFERENCES

4.1 Glassware, plasticware, and other sample processing hardware have the potential to contaminate the audit filters. All labware and hardware used must be soaked or rinsed in 1% (v/v) nitric acid, rinsed with deionized water, and completely dried prior to use.

5.0 HEALTH AND SAFETY CAUTIONS

5.1 This method does not address all the possible safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and compliance

with all OSHA regulations. Material Safety Data Sheets (MSDS) for all chemicals used should be readily available to all personnel involved with the procedure.

5.2 The NIST SRM 3128 ampoules contain a lead solution in 10% HNO₃. Nitric acid is a strong, corrosive, oxidizing agent that requires protection of the eyes, skin, and clothing. Items to be worn during use of this reagent include:

- Safety goggles (or safety glasses with side shields)
- Acid resistant rubber gloves
- A protective garment such as a laboratory apron. Nitric acid spilled on clothing will destroy the fabric and result in a hole; contact with the skin underneath will result in a burn.

It is also essential that an eye wash fountain or eye wash bottle be available during performance of this method. An eye wash bottle has a spout that covers the eye. If acid or any other corrosive gets into the eye, the water in this bottle is squirted onto the eye to wash out the harmful material. Eye washing should be performed with large amounts of water immediately after exposure. Medical help should be sought immediately after washing. If nitric acid is spilled onto the skin, wash immediately with large amounts of water. Medical attention is not required unless the burn appears to be significant. Even after washing and drying, the nitric acid may leave the skin slightly brown in color, this will heal and fade with time.

5.3 Lead (Pb) salts and lead solutions are toxic. Great care must be taken to ensure that samples and standards are handled properly; wash hands thoroughly after handling.

5.4 Care must be taken when opening the glass ampoules. The chemist must follow the instructions contained in the NIST certificate of analysis, INSTRUCTIONS FOR USE section, that accompany the SRM.

6.0 EQUIPMENT AND SUPPLIES

6.1 Materials

- Plastic filter holder.
- Pipette, Rainin EDP2, 10-100 µL, with disposable tips, or equivalent.
- Pipette, Rainin EDP2, 1000 µL, with disposable tips, or equivalent.
- Plastic tweezers; VWR Catalog No. 89026-420, or equivalent.
- Laboratory marker.
- Blank labels or labeling tape, VWR Catalog No. 36425-045, or equivalent.
- Volumetric flask, 100mL, VWR Catalog No. 89001-898, or equivalent.
- Teflon filters, unexposed, Measurement Technology Laboratory, or equivalent.

- Millipore Element deionized water system, or equivalent, capable of generating Type I water ($>17.9 \text{ M}\Omega\text{-cm}$).
- Mettler XP205 Balance or equivalent with readability of at least 0.01mg, detectability of at least $1.6\text{mg} + 1.2 \times 10^{-5} R_{gr}$, and repeatability of at least $0.008\text{mg} + 6 \times 10^{-8} R_{gr}$ where R_{gr} = gross weight.

7.0 REAGENTS AND STANDARDS

- 7.1 Reagent or trace metals grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.
- 7.2 Concentrated nitric acid, 67-70%, SCP Science Catalog No. 250-037-177, or equivalent.
- 7.3 Deionized water – All references to deionized water in the method refer to Type I deionized water with a resistivity $>17.9 \text{ M}\Omega\text{-cm}$.
- 7.4 NIST SRM 3128 Working Standard. The chemist must exactly follow the NIST directions for “Preparation of Working Standard Solution by Volume” as detailed in the NIST certificate. The chemist must prepare the solution on a weight/volume basis with the concentration of the working standard close to a nominal value of $1000 \mu\text{g/mL}$. Based on the current NIST certified value of 9.987mg/g , a 10-fold dilution is required. The exact solution concentration must be determined following the instructions in the NIST “Preparation of Working Standards by Volume” instructions on the certificate that accompanies the SRM.
- 7.5 Isopropyl Alcohol, B&J ACS, HPLC Grade, or equivalent, 2-fold dilution with deionized water.

8.0 FILTER PREPARATION

All plasticware and glassware used in the preparation procedure must be rinsed or soaked in 1% HNO_3 (v/v) and then rinsed with deionized water and dried prior to use. Record all preparation steps in a laboratory notebook or appropriate electronic laboratory records system.

- 8.1 The mechanical pipette to be used for spiking the filters must be verified at the volumes to be used for spiking. The analyst must use deionized water and a calibrated balance readable to at least a 4 decimal places. NOTE: Mechanical pipettes should be set to the desired volume and then activated through one draw and dispense cycle before any liquids are pipetted. A small disposable polypropylene beaker or other container must be placed on the balance and tared. The analyst must

pipette the selected volume of deionized water into the container and record the weight, tare the balance, and repeat until five replicate weights have been recorded. The average of the five replicates is calculated by dividing the sum of the weights by 5. The relative percent error is calculated by Equation 1 below.

Equation 1. Relative Percent Error

$$\text{Relative \% Error} = ((M - T)/T) \times 100$$

Where M = average of five replicate weights

T = target weight

The relative percent error must be less than 1 at each volume to be used for spiking.

- 8.2 Using plastic tweezers carefully select one filter at a time and place the filter in a clamp so that only the support ring makes contact with the clamp as shown in Figure 1.
- 8.3 The Teflon filter surface must be wet with 0.25mL of 50% IPA solution. Using a mechanical pipette, dispense 0.25mL of the 50% IPA solution in the center of the filter. It is critical that the lead solution is spiked within a few minutes after wetting the filter with IPA solution.
- 8.4 The amount of NIST working standard prepared in Section 7.4 to be used for spiking must be calculated. Equation 2 below shows the calculation for determining the amount of spike to add to achieve the desired percentage of the NAAQS standard.

Equation 2.

$$\left| \begin{array}{c|c|c} T \mu\text{g} & 24 \text{ m}^3 & \text{mL} \\ \hline \text{m}^3 & \text{filter} & C \mu\text{g} \end{array} \right| = \text{ ______ mL}$$

Where

T = the concentration in $\mu\text{g}/\text{m}^3$ (desired percentage in decimal form X $0.15 \mu\text{g}/\text{m}^3$)

$24\text{m}^3/\text{filter}$ = theoretical 24 hour air volume of PM10 low volume sampler

C = the concentration of the NIST working standard prepared in Section 7.4

For 70% of the NAAQS standard ($0.15 \mu\text{g}/\text{m}^3 * 0.7 = 0.105 \mu\text{g}/\text{m}^3$) on a 46mm Teflon filter using a 1024 $\mu\text{g}/\text{mL}$ working standard, the calculation would be:

$$\left| \begin{array}{c|c|c} 0.105 \mu\text{g} & 24 \text{ m}^3 & \text{mL} \\ \hline \text{m}^3 & & 1024 \mu\text{g} \end{array} \right| = 0.0025\text{mL}$$

The calculation of the exact $\mu\text{g}/\text{filter}$ based on 0.0025mL of working standard is shown in Equation 3 below.

Equation 3.

$$\frac{\begin{array}{c} \text{A} \\ 0.0025 \text{ mL} \end{array}}{\text{mL}} \times \frac{\begin{array}{c} \text{B} \\ 1024 \text{ } \mu\text{g} \end{array}}{\text{mL}} \times \frac{\text{1 Filter}}{\text{filter}} = \frac{\begin{array}{c} \text{C} \\ 2.56 \text{ } \mu\text{g} \end{array}}{\text{filter}}$$

Where:

A = the mL of NIST working standard added

B = the concentration of the NIST working standard prepared in Section 7.4

C = the final concentration in $\mu\text{g}/\text{filter}$

NOTE- The final concentration in μg is the reporting unit that is required to be reported to AQS.

- 8.5 Set the mechanical pipette to the desired volume and activate it through one draw/dispense cycle. Place a tip on the pipette and draw the desired volume of NIST working standard prepared in Section 7.4. NOTE: The analyst must visually inspect the pipette tip after drawing spiking solution to ensure that there are no droplets on the exterior of the tip. The analyst should also note the liquid level inside the tip to compare to all subsequent draws.
- 8.6 Hold the pipette over the center of the filter and activate the dispense cycle. The pipette tip should be just above the filter, but not touching the filter. The analysts must visually inspect the filter after spiking. There must not be spiking solution beading on the filter surface. NOTE: The orientation of the tip to the audit filter is critical to ensure that all the spiking solution drawn is deposited on the filter. The analyst must visually inspect the pipette tip after deposition to ensure that all solution was deposited and no droplet remains in the tip. If any liquid remains in the pipette tip, the pipette tip and filter should be discarded and the spiking repeated on a new filter.
- 8.7 The steps in 8.3, 8.5, and 8.6 are repeated until all filters have been spiked.
- 8.8 The audit filters must be allowed to dry for a minimum of 8 hours in a HEPA filtered laminar flow hood.
- 8.9 Once the audit filters have dried for at least 8 hours, the individual filters are placed in plastic petri slides that have been screened for the presence of lead.
- 8.10 Label the audit filters so that the concentrations can be identified by the laboratory preparing the filters but not by outside laboratories.

9.0 QUALITY CONTROL

- 9.1 A minimum of seven audit filters at each concentration prepared must be selected at random from all the audit filters prepared for extraction and analysis. The actual number of filters will be dependent on the total number prepared and the desired

confidence level. The filters selected must have been taken through the entire preparation process including packaging and labeling.

- 9.2 Extract and analyze the selected filters by a method appropriate for the analysis of lead on PM10 filters. This SOP is specific to the preparation of the audit filters and not intended to cover the extraction and analysis of audit filters.
- 9.3 The analysis results must be within 5% bias of the expected result. The expected result is calculated by Equation 3 as shown in Section 8.4. If the average of the seven filters is outside $\pm 5\%$ bias, the lot must be rejected and the entire preparation process repeated.

10.0 METHOD PERFORMANCE

Information in this section is an example of typical performance results achieved by following this SOP. Actual performance must be demonstrated by each individual laboratory and instrument.

- 10.1 Recovery tests with audit filters spiked with NIST SRMs were performed using the ultrasonic/ nitric and hydrochloric acid filter extraction method and measurement of the dissolved lead with ICP-MS. Table 1 shows recoveries for two spiking levels. The recoveries for the audit filters are all within $\pm 5\%$ bias for each filter and the average.

11.0 POLLUTION PREVENTION

- 11.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. The sources of pollution generated with this procedure are waste acid extracts and lead-containing solutions.
- 11.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th St. N.W., Washington, D.C., 20036, www.acs.org.

12.0 WASTE MANAGEMENT

- 12.1 Laboratory waste management practices must be conducted consistent with all applicable rules and regulations. Laboratories are urged to protect air, water, and land by minimizing all releases from hood and bench operations, complying with the letter and spirit of any sewer and discharge permits and regulations, and by complying with

all solid and hazardous waste regulation. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society listed in Section 14.2.

- 12.2 Waste nitric acid, hydrochloric acid, and solutions containing these reagents and/or Pb must be placed in labeled bottles and delivered to a commercial firm that specializes in removal of hazardous waste.

13.0 REFERENCES

- ¹ NIST, *Certificate of Analysis: Standard Reference Materials 3128, Lead Standard Solution, Nominal 10 mg/g Lead*, National Institute of Standards and Technology, Gaithersburg, MD, 2005.

14.0 TABLES AND FIGURES

Table 1. Recoveries of Lead from NIST SRM Spiked Audit Filters.

Low Audit Filter (2.5 µg/filter)	µg/filter	% Bias
1		
2		
3		
4		
5		
6		
7		
Average		
Standard Deviation		
%RSD		
High Audit Filter (9.0 µg/filter)	µg/filter	% Bias
1		
2		
3		
4		
5		
6		
7		
Average		
Standard Deviation		
%RSD		

Figure 1. Filters in Clamp

