

California Environmental Protection Agency



SOP MLD062

**STANDARD OPERATING PROCEDURE FOR
FILTER AND CANISTER PREPARATION
FOR PM_{2.5} SPECIATION SAMPLES**

Northern Laboratory Branch
Monitoring and Laboratory Division

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1 Introduction

In 1997, the U.S. Environmental Protection Agency created new federal air quality standards for PM_{2.5} and ozone, and proposed new requirements to reduce the regional haze that impairs visibility. The PM_{2.5} standards complement existing federal and state standards that target the full range of inhalable particulate matter (PM₁₀). Efforts to characterize PM_{2.5} and comply with the federal standards will further progress toward California's own PM₁₀ standards.

The goal of the PM_{2.5} monitoring program is to provide ambient data that support the nation's air quality programs. These data include mass measurements and chemically resolved, or speciated, data. Mass measurements are used principally for comparison to the PM_{2.5} National Ambient Air Quality Standards (NAAQS). These comparisons identify areas that do or do not meet the PM_{2.5} NAAQS, thereby allowing areas to be designated as attainment or nonattainment. Speciated data support the development of emission mitigation strategies intended to reduce ambient PM_{2.5} levels. The data are used to evaluate emissions inventory and air quality models, analyze source attribution, and track the success of emission control programs.

2 Summary of Method

Method MLD062 describes the preparation of the sampling canisters used for the collection of PM_{2.5} speciation samples, shipment to the field collection sites, receipt and disassembly, and login / distribution of samples for analysis within the laboratory. Each sample shipment consists of three canisters, one containing a preweighed teflon filter for mass and metals analysis, one containing a nylon filter for ion analysis, and one containing a quartz filter for organic carbon / elemental carbon (OC/EC) analysis.

3 Interferences and Limitations

- 3.1 Contaminants in the reagent water, reagents, glassware, filters, or other sample processing apparatus could lead to detectable concentrations of any of the analytes of interest. Blanks are run with each set of samples to monitor these possible sources of contamination. Gloves are worn when handling all canisters and components during the cleaning, assembly, and disassembly processes to avoid contamination.

4 Equipment

This SOP assumes familiarity with the operation of the muffle furnace, the vacuum oven, and the dishwasher. For detailed instructions in the operation of this equipment, refer to the operation manuals.

4.1 Equipment

1. Muffle furnace, maximum temperature at least 900 °C
2. Vacuum oven, capable of reaching 60 °C, with drying trays
3. Dishwasher, laboratory grade, using deionized water
4. Desiccator
5. Met One Instruments SASS™ Canister assemblies with denuder sleeve, denuders, filter cassettes, and filter spacer
6. Ice chest, 50 quart
7. Storage bin with dividers and snap on lid
8. Freeze packs
9. Foam packing pieces
10. Forceps
11. Plastic bags

5 Materials and Chemicals

5.1 Materials:

1. Nylon Filters, Whatman #7410-004, 47 mm diameter, 1.0 µm pore size
2. Quartz Filters, Whatman #1851-047, 47 mm diameter, Grade QM-A
3. Teflon Filters, Whatman # 7592-004, 46.2 mm diameter, 2 µm
4. Plastic centrifuge tubes with caps, 50 mL size
5. Beaker, 1 L size
6. Gloves, class 100
7. Plastic petrislides, 47 mm
8. Petri dishes, 47 mm
9. Porcelain dish
10. Non-sudsing dishwasher soap

5.2 Chemicals: All chemicals are at least spectrophotometric grade.

1. Sodium carbonate (Na_2CO_3)
2. Nanopure ASTM Type 1 deionized water (>16 MΩ-cm)

5.3 Nylon filter washing solution: 0.015 M sodium carbonate prepared by dissolving 1.59 g of the salt and diluting to one liter with nanopure deionized water.

6 Preparation of Filters

- 6.1 Teflon filters are preweighed according to SOP MLD055 and are delivered to the canister assembly lab with accompanying data sheet and preassigned bar code.
- 6.2 Nylon filters absorb nitric acid over time and may also contain significant amounts of sodium. As such, they require pretreatment before they are ready to be assembled in canisters and sent out for sample collection.
 - 6.2.1 Add 500 mL of 0.015 M sodium carbonate to a 1 L beaker. Add new inspected nylon filters to this solution one at a time, edge first so that they sink below the surface of the solution. Cover the beaker and soak for 4 hours.
 - 6.2.2 After 4 hours soaking, carefully pour off the sodium carbonate and add 100 mL of nanopure deionized water. Mix by swirling, allow the filters to settle, and carefully pour off the water. Repeat twice.
 - 6.2.3 Add 500 mL of nanopure deionized water, mix by swirling, and cover. Sonicate the beaker for 60 minutes and then soak overnight. The next morning, carefully pour off the water, add another 500 mL nanopure deionized water, mix by swirling, cover, sonicate for 60 minutes and let soak all day.
 - 6.2.4 In the afternoon, carefully pour off the water, add another 500 mL nanopure deionized water, mix by swirling, cover, and let soak overnight. Pour off the water and add 500 mL nanopure deionized water. At least 2% of the filters must be checked for ion content. Remove the appropriate number of filters from the beaker and analyze by SOP MLD064. All ions must be present at less than 1.0 μg / filter. If the ion content of any of the analyzed filters exceeds this level, sonicate the remaining batch of filters and soak overnight. Carefully pour off the water, add 500 mL nanopure deionized water and reanalyze.
 - 6.2.5 After analysis has confirmed that no ions are present at greater than 1.0 μg /filter, carefully remove the filters using forceps and place them on the vacuum oven drying racks.
 - 6.2.6 Place the drying racks in the vacuum oven at 60 °C and apply vacuum. Leave in the oven until the filters are dry. Opening the oven and wiping condensed water from the inside of the front window after a few minutes may speed up the drying process. Total drying time is usually about 30 minutes.
 - 6.2.7 When the filters are dry, store them in new clean petri dishes in the desiccator until needed.

- 6.3 New quartz filters may contain organic carbon and therefore require pretreatment before they are ready to be assembled in canisters and sent out for sample collection.
 - 6.3.1 Set the muffle furnace at 900 °C and allow it to come to temperature.
 - 6.3.2 Place several inspected quartz filters into a porcelain dish and place the dish into the furnace. After the furnace returns to 900 °C let the filters bake for four hours at temperature. Turn off the furnace and allow to cool overnight.
 - 6.3.3 The next morning when the furnace is cool, remove the porcelain dish from the furnace. Remove the filters from the dish with forceps, place them in plastic petri dishes and store them in the desiccator until use.

7 Assembly of Canisters and Shipment to Sites

- 7.1 Gather an adequate supply of clean canister assemblies, filter cassettes, filters, plastic bags, foam packing and storage bins. Always wear gloves when handling clean opened canisters and canister components.
- 7.2 Lay a canister base on the mounting block making sure the O ring is in place. Place an empty filter cassette into the base and a filter spacer on top of it, making sure the spacer has O rings seated on both sides. Load a filter of the appropriate type for this channel into a filter cassette. Secure the top ring of the filter cassette and place the assembled cassette on top of the filter spacer. For teflon or quartz filters, place an empty denuder ring, fitted with O rings on both ends, on top of the filter cassette. For nylon filters, place a denuder ring containing a denuder, fitted with O rings on both ends, on top of the filter cassette. Record the denuder number on the custody / field data form. Carefully place the canister cover over the base, align the screw holes and secure with screws and washers. Fit caplugs into the holes of the canister base and cover. After canisters containing a teflon, nylon, and quartz filter have been assembled, the canister assembly process is complete.
- 7.3 Enter all the remaining data onto the custody / field data form, including canister numbers, bin number, site name, and scheduled sampling date. Field blanks are scheduled every 10 sampling events and use the field blank custody / field data forms. Enter the barcode number onto the sample account log under the appropriate site and date. Seal the assembled canisters in plastic bags, and pack them in the bins, securing them with foam inserts. Seal the custody / field data form along with a “if this ice chest is lost” form into a plastic bag and lay on top of the canisters. For field blanks, include a copy of the “field blank instruction” form. Place room temperature freeze packs in the bin’s empty spaces and place the lid on the bin.
- 7.4 Load the bin into an ice chest with foam inserts and room temperature freeze packs. Label the ice chest with the site name on top of the lid. Deliver the ice chest to the shipping area for shipment to the site.

8 Receipt of Canisters, Disassembly, and Filter Distribution

- 8.1 When ice chests are returned to the laboratory, the temperature upon receipt must be determined. Open the ice chest, and with the bin still loaded with freeze packs, insert a thermometer probe into the center of the bin and close the lid of the ice chest. When the temperature reading has stabilized, record the result on the custody / field data sheet along with the date and time the sample was received. Store the bin in a refrigerator at 4 °C until it is disassembled.
- 8.2 Verify that the bin and canister ID numbers match those on the custody / field data sheet. If these numbers do not match, reconcile any discrepancies before proceeding. Place a check next to the barcode number on the sample account log for this site and date to indicate that the sample has been received.
- 8.3 To disassemble a canister, remove the caplugs from the base and place the canister on the mounting block. Using the hex wrench, loosen and remove the screws and carefully remove the canister cover. Remove the denuder ring or denuder and the filter cassettes from the canister base.
- 8.4 Separate the rings of the filter cassette by sliding the cassette along the opening tool. Carefully remove the filter and place it in the appropriate container. The teflon filter is returned to the bar-coded petrislide that it originally came from. The nylon filter is placed into a 50 mL centrifuge tube with the exposed side of the filter facing the center of the tube. The quartz filter is placed into a petri dish. The centrifuge tube and the petri dish receive bar-coded labels matching the petrislide barcode.
- 8.5 Place the labeled petrislide containing the teflon filter into the freezer compartment of the refrigerator in the sample processing lab. After sample log-in (see Section [9](#) for Sample Log-in information) has been completed, place the custody / field data sheet in the labeled box on the lab bench in the sample processing lab. The filters and data sheets will be collected by the PM_{2.5} balance room and analyzed by SOP MLD055.
- 8.6 After mass analysis has been completed by the PM_{2.5} balance room, the data sheets and filters are returned to the ion chromatography lab with the filters stored in the refrigerator at 4°C. When samples from a two month period are collected, the samples are organized by site and sampling date and delivered to the X-ray Fluorescence lab for analysis by SOP MLD034.
- 8.7 Place the labeled 50 mL centrifuge tube containing the nylon filter into the tube rack in the refrigerator in the Ion Chromatography lab for analysis by SOP MLD064.
- 8.8 Place the labeled petri dish containing the quartz filter into the storage container in the refrigerator in the OC/EC lab for analysis by SOP MLD065.

9 Sample Log-in

- 9.1 Log into SQL-LIMS and after choosing the appropriate site for the sample, scan the barcode number and record the assigned LIMS number onto the custody / field data sheet. Enter all of the remaining sample information from the data sheet. If any required sample information is missing from the data sheet, contact the site operator to obtain the necessary data.
- 9.2 After all the samples from this batch have been logged in, place the data sheets in the box labeled "PM_{2.5} speciation data sheets for mass analysis" in the sample processing lab.
- 9.3 Run the log-in summary report and inspect all the entries to verify that the data was entered correctly. After this has been verified and any errors corrected, initial the summary and file it in the appropriate folder.

10 Canister Cleaning

- 10.1 Canister bases and covers, filter denuder sleeves, and filter spacers are washed in the dishwasher by placing them in wash racks on the dishwasher shelves. Filter cassettes (assembled with screens) and caplugs are loaded into washing bags and laid on the top shelf of the dishwasher.
- 10.2 Add detergent, set for minimum wash and no dry and start the washer. After the wash cycle is completed, remove the items and disassemble the filter cassettes. Rinse all items three times with nanopure deionized water and place in a rack to dry overnight. All clean items are only handled while wearing gloves.
- 10.3 Return the cleaned canister pieces to the sample receipt lab and assemble the canisters with base, cover, filter spacer, and for the channel one and three canisters (green and orange dots), a denuder spacer. The channel two canister (red dot) will have a denuder added during canister and filter assembly (see Section 7 for Assembly of Canister and Shipment to Sites information). Store the assembled canisters in their assigned drawers until needed.
- 10.4 Assemble the filter cassettes with their support screens and store them in plastic bags in the sample processing lab until needed.

11 References

1. Quality Assurance Guidance Document, Final, Quality Assurance Project Plan, PM_{2.5} Speciation Trends Network Field Sampling, EPA-4154/R-01-001.