

# STANDARD OPERATING PROCEDURE

## Speciation Sampling Canister Processing

July 7, 2003

Version 1.0

DEQ03-LAB-0044-SOP



State of Oregon  
Department of  
Environmental  
Quality

**Laboratory Division**  
1712 SW 11th Avenue  
Portland, OR 97201  
Phone: (503) 229-5983  
Fax: (503) 229-6924  
[www.deq.state.or.us](http://www.deq.state.or.us)



Prepared by: Ben Jones \_\_\_\_\_ Date: July 7, 2003 \_\_\_\_\_

Reviewed by: \_\_\_\_\_ Date: \_\_\_\_\_  
Technical Director

Approved by: \_\_\_\_\_ Date: \_\_\_\_\_  
Quality Assurance Officer

January 2002



This page left intentionally blank.

---

## Table of Contents

Table of Contents .....	3
1. Test Method .....	4
2. Applicable Matrices .....	4
3. Detection Limits .....	4
4. Scope and Application .....	4
5. Summary .....	4
6. Definitions .....	4
7. Interferences .....	5
8. Safety .....	5
9. Equipment and Supplies .....	5
10. Reagents .....	6
11. Standards .....	6
12. Sample Collection, Preservation, Shipment, and Storage .....	6
13. Calibration and Standardization .....	7
14. Quality Control .....	7
15. Procedure .....	7
15.1. Filter preparation: .....	7
15.2. Denuder preparation: .....	7
15.3. Cassette loading: .....	8
15.4. Sampling Canister Loading: .....	8
15.5. Sampling Canister Unloading: .....	8
15.6. Additional steps required for Quality Control elements in related SOPS .....	9
16. Calculations .....	9
17. Method Performance .....	9
18. Maintenance .....	9
19. Pollution Prevention .....	9
20. Waste Management .....	9
21. References .....	9

## 1. Test Method

Speciation Sampling Canister Processing

## 2. Applicable Matrices

This method applies to the processing of MetOne SASS Speciation canisters used to collect ambient air particulate on 47mm diameter Teflon, Quartz, and Nylon filters.

## 3. Detection Limits

No applicable.

## 4. Scope and Application

This method describes the cleaning, loading, and unloading of the MetOne SASS speciation sampler canisters. At this time, these canisters are used only in the collecting of ambient air particulate matter for the PM<sub>2.5</sub> Speciation program. Sampling and analysis procedures are found in other SOPs.

The results from the Speciation program are used in modeling, SIP development, and air toxics assessment. The canisters that DEQ prepares and analyze are not used at sites that are part of the National Trends Network. We have elected to have the work for the National Trends Network performed by the EPA contract lab, in the interest of consistency.

## 5. Summary

Filters are prepared as appropriate for their material type and the analytes for which they are used.

- Teflon filters are pre-sampling weighed, and the analytes for the particulate collected on the Teflon media include both the mass and the XRF determined elements.
- Nylon filters are washed with DI water, and the analytes for the particulate collected on the Nylon media are the IC determine analytes: the extractable anions and cations.
- Quartz filters are fired at 700°C, and the analytes for the particulate collected on quartz media are the carbon fractions such as elemental carbon and organic carbon.

Each canister is loaded with one type of filter. In reassembling the canisters with their filters, the canisters containing the Nylon filters include a MgO denuder upstream of the filter to prevent absorption of gases, such as nitrogen oxides, on the filter or its particulate.

## 6. Definitions

**Batch:** environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

**Analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices.

**Blank:** a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

**Lab Blanks:** These are filters that have been prepared (conditioned and weighed, washed, or fired) as appropriate for the filter type, and then stored in labeled Petri dishes in the freezer until analyzed.

**Field Blanks:** These are filters that are treated the same as a normal sample (including installation on the samplers in the field) except that no air is sampled through them. (Field blanks, as defined in this SOP, are referred to as “transfer blanks” in LIMS.)

**Holding Times (Maximum Allowable Holding Times):** the maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

**Laboratory Information Management System (LIMS):** a comprehensive computerized database system that a laboratory uses for sample tracking and data management, from sample receipt to reporting and archiving.

**Particulate matter:** A generic term for a broad class of chemically and physically diverse substances that exist in the air as discrete particles (liquid droplets or solids) over a wide range of sizes.

## 7. Interferences

Contamination of the filters is our primary concern in terms of the canister preparation and disassembly. Because so many analytes are being determined, any detergents or solvents may cause more problems than they solve. Maintaining clean equipment and spaces reduces the need to clean with much more than filtered compressed air or DI water. The following precautions are followed so as to collect uncontaminated samples:

Clean, solid-surface workspaces are used during all assembly and disassembly processes.

The filters, when not in the canisters, are kept in closed containers, as much as possible.

The canisters are tightly closed and the ends are plugged whenever they are not installed on the samplers. This includes times when the canisters are empty, as well as when they contain filters.

Each canister and its internal parts are dedicated to a specific filter type.

Filters are only touched with non-serrated, stainless steel forceps.

Extra precautions are taken to clean filter cassettes and screens after use with the Quartz filters, as loose fibers from the quartz cling to contact surfaces.

## 8. Safety

For general laboratory safety procedures, consult the DEQ Laboratory Safety Manual (Chemical Hygiene Plan).

Refer to the Job Hazard Assessment for the related procedure “Particulate Matter Weighing” and observe the safety precautions specified there.

The only additional hazard associated with this SOP is the use of the muffle furnace. The need to avoid touching hot surface or materials when using the muffle furnace is the obvious precaution required.

Material Safety Data Sheets (MSDS) are available in the laboratory library.

## 9. Equipment and Supplies

- Canisters, MetOne SASS sampler
- Cassette opening tool.
- Desiccator, glass, with vacuum port.

Revision: 1.0

- Cassettes, filter, consisting of three parts: a top, bottom, and screen, as supplied by R & P. These cassettes meet the requirements as stated in section 7.3.5 of Appendix L to 40 CFR Part 50, except they have beveled edges as required for the R&P Partisol samplers.
- Filter labels, vinyl, pre-numbered, that fit in the groove on the edge of the cassettes.
- Forceps, non-magnetic, stainless steel, non-serrated.
- Freezer.
- In-line filter installed on the line attached to the building compressed air, Balson Filter Tube, Grade BQ.
- Kimwipes.
- Muffle furnace
- Nylon filters, 47 mm, 1um pore size, from Nylasorb filters from Pall Corporation.
- Petri dishes, polystyrene, 50x9 mm, Falcon.
- Protective boxes for shipping and temporary storage of filters while they are in the filters cassettes, 2 5/8"square by 5/8" high molded, hinged clear styrene
- Quartz filters, 47mm, QMA, from Whatman.
- Quartz filters, 8x10 inch, as used for HV samplers
- Refrigerator maintained at <4°C.
- Teflon filters, 46.2 mm, as supplied by EPA. These filters meet the requirements as stated in section 6.0 of Appendix L to 40 CFR Part 50. These have a nominal pore size of 2 um (based on water filtering) but collect greater than 99.7% of the 0.3 um particles based on air filtering.
- Ultrasonic bath. Sonicore Model 211 TH, or similar.

## 10. Reagents

Not applicable

## 11. Standards

Not applicable

## 12. Sample Collection, Preservation, Shipment, and Storage

Samples are retrieved from the samplers within 48 hours of the end of sampling. Samples are collected from the samplers by the field staff, at which time they are plugged with the plastic end caps, are placed in a plastic bag with twist wire closings, and are stored in either a cooler or an on-site refrigerator. The filters are held in their canisters until they are removed by the laboratory staff.

The field staff records on the field sheets any observed abnormalities of the canisters, the sampler, or the nearby environment.

During shipping or transport, the samples are kept at <4°C. Upon return to the laboratory, the samples are placed in a refrigerator maintained at ≤ 4°C, until removal from the canisters. After removal from the canisters, the nylon and quartz filters are store in a freezer (< -20°C) and the Teflon are stored in the refrigerator (< 4°C). During storage, all filters types are kept in separate Petri dishes.

There are no specific holding times for the speciation sampling filters. The holding times used for the FRM samples (see DEQ SOP—"Gravimetric Analysis of Particulate Collected with R&P Partisol Samplers and MetOne SASS Samplers" are the target holding times for the speciation Teflon filter samples. Because the nylon and quartz filters are handled in parallel with the Teflon filters, they have the same target holding times up to the point that they are removed from the canisters and placed in storage (until analysis is started). It is our intent to analyze filters within 90 days of sampling, but filters are not downgraded if this target time is not met.

Revision: 1.0

### 13. Calibration and Standardization

Not applicable.

### 14. Quality Control

Not applicable in this SOP, although the analytical SOPs associated with these samples all have QC sections with criteria and control actions. See also Section 15.6 below.

### 15. Procedure

#### 15.1. Filter preparation:

**Teflon filters:** Whatman 46.2mm diameter filters as supplied by EPA are inspected, equilibrated, and weighed as per the DEQ SOP "Gravimetric Analysis of Particulate Collected with R&P Partisol Samplers and MetOne SASS Samplers". These are loaded into clean R&P cassettes, labeled, and then are stored in individual polystyrene boxes until being loaded in METOne sampling canisters.

**Quartz filters:** Whatman 47mm QMA filters. The quartz filters are fired at 700° C for 2 hours. During firing, the filters should be lying on a clean, flat surface, such as a quartz HV filter. This will prevent the filter from becoming distorted during the firing process. After firing, the filters are cooled to approximately 300° C while still in the muffle furnace, and then cooled to room temperature in an evacuated desiccator. The time period of cooling is kept to a minimum so that the filters do not absorb gases (organics) from the laboratory air. As soon as possible after the filters have cooled to room temperature (normally the next morning) filter should be placed in tightly closed Falcon Petri dishes, at approximately 10 per dish, where they are stored until being loaded into a METOne sampling canister. The filter lot and the date of firing are recorded on top of each Petri dish, as well as in a log book.

**Nylon filters:** Pall 47 mm Nylasorb filters with a 1um pore size. These are washed in DI water in batches of 100. To wash, place all filters—without the separators that are included in the packaging—in a clean 1 liter poly. Rinse 5 times with DI water. (Note: In all steps requiring rinsing, this means filling the container completely with DI water, stirring, and then draining completely.) Then, with fresh DI water, tumble the 1 liter poly for at least 1 hour, using the TCLP tumbler. Pour off the water, refill and tumble for another hour. After 2<sup>nd</sup> tumbling, transfer the filters to a clean 500 ml beaker and rinse with DI water another 10 times. The filters are then dried in an evacuated desiccator and then stored in tightly closed Falcon Petri dishes, at approximately 10 per dish, until loaded into a METOne sampling canister. The filter lot and the date of washing are recorded on top of each Petri dish, as well as in a log book.

#### 15.2. Denuder preparation:

MgO denuders, used upstream on the nylon filters, are purchased from MetOne. They can be used for at least 60 sampling events with the expected air quality found in Oregon. The number of times that each denuder is used is kept track of in 2 ways: The denuder that is used with each filter is recorded, and a label is placed on each denuder to record an "X" for each use. The denuders will be replaced when they have been used 60 times, or they will be replaced if they become wet as occasionally occurs during wet weather. The denuders are kept in seal sampling canisters whenever they are not used to keep them from becoming saturated from the nitrogen oxides that may be present in the lab air.

### **15.3. Cassette loading:**

The same stock of cassettes is used both for the R&P samplers and the MetOne SASS canisters.

Clean blue cassettes are loaded with their filters just prior to loading the cassettes in the sampling canisters (except for the Teflon filters, which are loaded into the cassettes at the time of taring). A label with the filter ID is then placed on the edge of each cassette. (White cassettes were discovered to contaminate the filters with organics, and therefore the white cassettes are not used for speciation sampling, especially on quartz filters.)

Between uses, the blue rings of the cassettes are washed on cycle 7 of the dishwasher, a cycle that includes a 60° C detergent wash that is followed by both a tap water and DI water rinse. The stainless steel screens are soaked in a dilute hot detergent bath and then rinsed thoroughly with tap water and DI water.

### **15.4. Sampling Canister Loading:**

Sample canisters are loaded with cassettes that have been loaded with filters and a color coded label with the Filter ID is applied to the outside of each canister. If the canister includes a denuder, a mark is placed on the denuder to keep track of how many times it has been used. Once the cassette and internal parts are assembled and the bolts are tightened, the canisters are placed in a clean plastic bag and sealed with a twisty tie.

The date of canister loading, the filter ID, and the filter lot are recorded in a record book.

Each canister is dedicated to one filter type, and the internal parts remain sealed within the canisters when not in use. (Because the canisters remained sealed at all times, they are not washed unless they become contaminated by misuse or an odd sampling event.)

Note: When new canisters are placed into service, the O-rings at the ports on each end receive a thin layer of silicone vacuum grease to prevent damage to the O-ring during installation of the cyclone.

### **15.5. Sampling Canister Unloading:**

When canisters are returned from the field, they are stored in a <4C refrigerator until removal of the filter.

When the canisters are disassembled, they are allowed to sit at room temperature for ~ 1 hour to warm to the point that they do not collect excessive condensation. The canister is then removed from the plastic bag and disassembled. The filter cassettes are removed and placed in clean polystyrene boxes, and the canister is reassembled and tightly closed for storage until the next use.

During disassembly, any unusual condition are noted (e.g. water in the canister, missing part, damaged filter) in the log book with the filter IDs and dates of canister assembly. Also, the correspondence between the external filter ID and the filter ID on the cassette is confirmed.

The cassettes with the filters are then disassembled (unless the filter is a Teflon filter, in which case they are placed in the refrigerator to be processed in the same manner as the FRM loaded filters) in a clean environment. The filters are placed in Petri dishes, to which the filter ID labels have been applied. These Petri dishes are immediately placed in a Freezer and stored until analysis.

The cassettes (both the rings and the metal screens) that hold the quartz filters become contaminated with the quartz filters, so after removing the filters, these parts are partly cleaned prior to placing them in the tray that holds the other dirty cassettes.

---

### ***15.6. Additional steps required for Quality Control elements in related SOPS.***

During canister assembly, 10% of the filters are set aside as lab blanks (except for the Teflon filters, which have 5% of the filters set aside as lab blanks when they are tared.)

Sets of filters (1 each Teflon, Nylon, and Quartz) are sent to each site at approximately a bimonthly frequency to be used as transfer blanks. These are stored at the site, installed on instrument, uninstalled, and returned to the lab in the same manner as canisters that have been used for sampling.

An extra canister, containing rotating filter types, is sent along with a normal set of 3 canisters. This fourth canister is installed on the 4<sup>th</sup> channel of the instrument and is sampled along with the other three. It is designated as a replicate sample. If canisters are available, these field duplicates will be collected at a frequency of one of each filter type per site per 2 months.

## **16. Calculations**

Records of field activity are recorded on field sheets. In addition, electronic records are downloaded from the instruments. Both sets of information are maintained in a database. This information is used in tracking, calculating and reporting the results.

## **17. Method Performance**

Not applicable. See related SOPS.

## **18. Maintenance**

Cassettes are cleaned in same manner as described in the DEQ SOP: "Gravimetric Analysis of Particulate Collected with R&P Partisol Samplers and MetOne SASS Samplers".

See the Section 7 (Interferences) for additional cleaning issues.

See the Section 15.2 for denuder replacement schedule.

## **19. Pollution Prevention**

There is no specific pollution prevention associated with this method.

## **20. Waste Management**

There is no specific waste management associated with this method.

## **21. References**

MetOne Instruments, "Field Operation Manual, Model SASS and SuperSASS PM2.5 Ambient Chemical Speciation Samplers", January 10, 2001.

Research Triangle Institute, "Quality Assurance Project Plan Chemical Speciation of PM2.5 Filter Samples, RTI/7565/00-01S, September 8, 1999.