TABLE OF CONTENTS

LIST OF TABLES

REFERENCES

Schematic C76-AQ-2469A, Double cutter for preparing masks.

1.0 PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) describes the process for procurement and acceptance testing of filters for use in the IMPROVE aerosol sampling network. Aerosol collection filters are purchased from suppliers guaranteeing purity levels, and are acceptance tested by the Air Quality Group for consistency and lack of contamination.

Four filter substrates are used for aerosol sampling in the IMPROVE network; stretched Teflon mesh, nylon, quartz, and quartz impregnated with glycerol and potassium carbonate. The table below lists the modules in which each substrate is used, and the analytical measurements obtained from each substrate.

Table 1 Summary of IMPROVE Aerosol Sampler Data Collection Parameters

Module	*Size Region*	*Filter*	*Analytical Measurement*
A	0-2.5 µm	Teflon	mass, coefficient of optical absorption, elements (H, Na-Pb)
B	0-2.5 µm	nylon with denuder	nitrate, sulfate, chloride
C	0-2.5 µm	quartz	organic and elemental carbon
D	0-10 µm	Teflon	PM_{10} mass, some with elements

2.0 RESPONSIBILITIES

2.1 Project Manager

The project manager shall:

- Verify planned continuation of current sites.
- Summarize expected filter usage for the next year.
- Review filter test data to determine whether the filters are acceptable.

2.2 Research Technician

The research technician shall:

- Initiate and oversee acceptance testing of filters.
- Process filter test data
- Report the results of the tests to the project manager

3.0 REQUIRED EQUIPMENT AND MATERIALS

3.1 Filter and Filter Testing Supplies

The equipment required for filter acceptance testing are as follows:

- Annual supply of stretched Teflon membrane filters having the same lot #.
- Nylasorb™ filter medium
- Filter cassettes
- Leak check device.
- Four side-by-side $PM_{2.5}$ IMPROVE protocol sampler modules.
- Facilities for gravimetric and B_{abs} analysis of collected aerosol samples.

4.0 METHODS

The procedures for filter testing and acceptance are described in the following subsections:

4.1 Filter Procurement
4.2 Mask Procurement
4.3 Filter Acceptance Testing

4.1 Filter Procurement

Annual network filter requirements are estimated at the start of each financial year by the project manager. Estimates include filters for network site sampling, quality control and quality assurance, and planned special studies.

Purchase orders for filters are generated by the project manager and forwarded to the supplies vendors or contractors. Filter testing for Teflon and nylon filters is done upon receipt of the order, prior to accepting the new filter lots for network use. Quartz and impregnated quartz filters are prepared for use by the subcontractors responsible for analysis of the filters, including quality assurance testing.

4.1.1 Teflon Filter Procurement

Teflon (Gelman Teflo™) filters are purchased by UC Davis in a single lot for an entire year. A single major order is initiated within existing procurement guidelines approximately six months prior to the requirement to use the lot. The purchase request specifies "All Teflon filters must be from the same lot." Upon receipt from the Teflon filters vendor or manufacturer, the containers are sequentially numbered to allow for future internal identification. Approximately one percent of the new filters are selected throughout the lot for acceptance testing.

The filters used in the IMPROVE network are 25mm stretched Teflon mesh filters with pore size 3.0μm, and a rigid olefin ring support.

4.1.2 Nylon Filter Procurement

Gelman Nylasorb™ 1.0μm pore size nylon filter material is purchased by UC Davis in 8.5" by 11" sheets separated by thin sheets of inert spacer paper. Each nylon filter sheet will provide eighty to ninety 25mm filters. Nylon filter sheets are purchased by UC Davis in a single lot for an entire year. A single major order is initiated within existing procurement guidelines approximately six months prior to the requirement to use the lot. The purchase request specifies "All sheets must be from the same lot."

Filters for use in the IMPROVE network are prepared in batches of ninety to one hundred from the nylon substrate sheets using a sterilized 25mm punch. The procedure for filter cutting is as follows:

1. Obtain a thick pad of clean, pure Teflon material from the laboratory supply retained for this purpose. This pad, approximately 1/2-inch thick, provides a cutting surface that will not dull or change the geometry of the cutter punch faces.
2. Place the Teflon material on the base of the mandrel press. Open the mandrel press enough to accommodate the 25mm cutter punch assembly when resting on the Teflon.

3. Retrieve 1 sheet of nylon filter material, with the inert spacer paper, from its sealed container in the laboratory refrigerator and place it on the Teflon pad such that the cutting punch is at the at the corner of the sheet.
4. Gently, but firmly, apply pressure to the assembly with the press arm until a distinct cutting sound is heard and felt. Open the press and observe that a 25 mm filter has been produced.
5. Remove the completed filter and spacer and stack them, with a spacer between each filter, in the current Nylasorb™ filter stock box from the refrigerator.
6. Move the punch to cut another filter, making sure to waste as little filter substrate as possible.
7. When no more filters may be cut from the sheet (roughly 90 filters), seal the filters in their container and return them to the refrigerator. Wrap the cutter punch with tape and return it to the protective foam box. Return the Teflon pad to its storage, and throw away the remainder of the nylon filter sheet.

4.1.3 Quartz Filter Procurement

Quartz filters are provided to the Air Quality Group by the carbon contractor responsible for analyzing the associated data. The filters are included in the analysis costs charged to the network. All quality assurance testing of filters is done prior to shipment to the Air Quality Group according to the specifications listed in the carbon contractor standard operating procedures. Summarized procedures are provided here.

1. Carbon Contractor: Purchase 25 mm Pallflex 2500 QAT-UP quartz filters.

2. Carbon Contractor: Pre-fire the quartz filters at 900°C for at least four hours following procedures developed by the contractor. The contractor procedures include testing a portion of the pre-fired filters for contamination.

3. Carbon Contractor: Ship the carbon filters to UC Davis in a cooled container.

4. UC Davis: Receive the pre-fired quartz filters and retain them in a clean, cool, dry environment until required in the loading sequence.

4.1.4 Impregnated Quartz Filter Procurement

Impregnated filters for measuring gaseous SO_2 as SO_4 are both supplied and analyzed by an external contractor. The filters are included in the analysis costs charged to the network. All quality assurance testing of filters is done prior to shipment to the Air Quality Group according to the specifications listed in the SO_2 contractor standard operating procedures. Summarized procedures are provided here.

1. SO_2 Contractor: Purchase 25 mm Pallflex QAT-UP filters.

2. SO_2 Contractor: Impregnate the filters with a 10 percent potassium carbonate and four percent glycerol solution. Dry the impregnated filters in a vacuum oven. The contractor procedures include testing a portion of the impregnated filters for contamination.

3. SO_2 Contractor: Package the impregnated filters in sub lots of 100 and ship to UC Davis.

4. UC Davis: Receive the impregnated filters and retain them in a clean, cool, dry environment until required in the loading sequence.

4.2 Collection Mask Procurement

The collection mask fits underneath the fine Teflon filter in the cassette and reduces the area of collection. The primary purpose is to improve the sensitivity of the XRF, PIXE and PESA analyses by concentrating the particles. The mask also reduces the mass artifact by isolating the filter from the O-ring. The decrease in the area of collection is limited by filter clogging: in regions of high concentrations the flow rate can be reduced below the acceptable range.

The collection masks are prepared from the inert paper spacers that Nuclepore Corporation provides between Nuclepore™ 47 mm 8 µm polycarbonate filters. The spacers have a very light coating of Apiezon-L grease. Many years of experience have shown that this paper will not transfer mass to the filters. The spacers are retained in their original factory containers and labeled and sealed until required as stock for preparing the collection masks.

The masks are prepared using specially machined double-action cutter punches. The cutter punches simultaneously cut the 25 mm outer diameter and the desired inner collection diameter. Both punch and cutter are concentric, centering the collection area in the 25 mm mask. It is imperative that the cutter punch be handled gently and the heat-treated and ground edges be protected. The edges are very sharp and can cause injury. If the cutter punch is dropped or not correctly used, damage to the cutter punch will result and it will have to be re machined.

The procedures for preparing the collection masks are as follows.

1. Obtain a sealed and marked container of 100 Nuclepore™ spacers from the laboratory supply.

2. Obtain the correct punch for the desired collection area, generally 2.2 cm^2. For schematics to construct a double cutter for preparing 1.1cm^2 and 2.2cm^2 masks, see drawing C76-AQ-2469A.

3. Obtain a thick pad of clean, pure Teflon material from the laboratory supply retained for this purpose. This pad, approximately 1/2-inch thick, provides a cutting surface that will not dull or change the geometry of the cutter punch faces.

4. Place the Teflon material on the base of the mandrel press. Open the mandrel press enough to accommodate the cutter punch assembly when resting on the Teflon. Initially, place no more than 5 spacer papers on the Teflon pad and center them under the cutter punch. Gently, but firmly, apply pressure to the assembly with the press arm until a distinct cutting sound is heard and felt. Open the press and observe that 25 mm masks with the desired hole have been produced. Remove the completed masks and put them in a clean container. Discard the excess spacer material.

5. Verify that the mask geometry is correct. Using a vernier inside/outside caliper from the laboratory supervisor, measure the inside diameter of the fabricated masks. make several measurements and determine the mean. For a 2.2 cm^2 collection area, the diameter should be 16.73 ± 0.01 mm. (The collection area of the actual particles on the filters has been found to equal this measured area.)

6. With practice and experience, up to 10 masks can be produced at a time. If incomplete cutting occurs, reduce the number of pieces of stock in the press back to 5.

7. When all 100 spacers in the original supply have been converted to masks, seal the masks in their container. Wrap the cutter punch with tape and return it to the protective foam box. Return the Teflon pad to its storage.

4.3 Filter Acceptance Testing

The IMPROVE aerosol samplers rely on consistent, high quality filter media. Consistency throughout the lots of filters is important both to ensure optimal flow rate, and provide stable artifact values. The quality of the filters refers to their artifact levels and lack of manufacturing flaws. High artifact levels, especially if not consistent, may lead to erroneous recorded concentrations. Manufacturing flaws may lead to leakage, or may simply slow the filter handling processes. To prevent surprises, and to ensure the filters are appropriate for use, extensive testing of each lot of filters is undertaken prior to use of that lot in the network. The following four sections describe acceptance testing procedures for the four filter media used in the IMPROVE aerosol sampling network.

4.3.1 Teflon Filter Acceptance Testing

Consistency in mass, areal density, and composition are the primary requirements for Teflon filters being used in the IMPROVE network. Roughly one percent of the new filters are selected throughout the lot for acceptance testing. A maximum of one filter is taken from each box. When certified, the entire lot is accepted for use in network operations and payment to the vendor is authorized.

The following tests are done by research technicians:

1. The randomly selected filters are visually inspected for unevenly stretched Teflon, holes, and poorly attached or damaged olefin rings.

   Unevenly stretched Teflon manifests itself as color variation on the filter. The less stretched portions are thicker, and appear brighter white than the thinner, more tightly stretched sections. Unevenly stretched surfaces are inevitable during production, but the number of visually uneven filters should be kept to a minimum of under 5%. Uneven stretching leads to local variations in pressure drop across the filter, and therefore local variations in areal loading. Fortunately, the analysis procedures compensates for these small differences in areal loading.

   Holes in the stretched Teflon mesh are unacceptable. No visual holes should appear on the filters. If more than 1% of the filters have manufacturing defects, the lot is considered unacceptable. No filters with holes are used for network operations. Generally, damaged filters are removed from the stock, as they are encountered, and stored in a bin for return to the manufacturer.

   The semi-rigid olefin ring constitutes the support structure for the Teflon membrane. The ring is heated and melted onto stretched Teflon mesh to form a filter. Poorly attached olefin rings occur when the heating is inadequate to melt the entire surface of the ring so that the Teflon is attached loosely, if at all. This detachment can allow air flow around instead of through the filter resulting in invalid data. Similarly, for weakly bonded Teflon and olefin rings, the sample may be collected, but become non-analyzable due to detachment and subsequent deformation of the stretched Teflon. Detachment and weak bonding can be visually discerned and are another manufacturer defect. If more than 1% of the filters in the lot show these defects, the lot should be returned.

   Teflon filters are screened for two other problems, namely warped or damaged olefin rings. If these occur in more than 1% of the filters, acceptance of the filter lot is refused. Warped olefin rings are created by excess heating during manufacture. These filters do not lie flat on a surface and are difficult to mount into cassettes. Damaged olefin rings often have uneven strips of olefin hanging off them or large dents that allow air to leak into the filter cassettes. Either defect, in quantities over 1% of the filters in the lot is sufficient to invalidate the entire lot.

2. Ten filters are weighed to determine if the weights are within usual Teflon filter ranges (25 to 40 mg). The weighing procedure is described in section 201. If the weights fall outside the range, the filters are still acceptable, but the tare weights

on the balances and the programs for data processing must be slightly changed to process the lot.

3. The ten weighed filters are used to test the consistency of the areal density of the stretched Teflon substrate. A 2.2cm^2 area is cut from each filter, following the procedures in Section 4.3 Collection mask procurement, then weighed. The average areal-density (area of the filter punch = 2.2cm^2 divided by the mass of the Teflon punch) and standard deviation are calculated and compared with current standards.

   The average areal density of the stretched Teflon should be within ±20% of 0.39mg/cm^2 . The standard deviation between the tested filters should be no greater than 30% of the average areal density, though larger deviations are acceptable.

4. Half of the filters selected for quality assurance procedures are analyzed by the LIPM, XRF, PESA and PIXE systems for elemental artifacts. The procedures for analysis are described in SOP 226**.** The spectrum obtained during each analysis should be a Bremsstrahlung spectrum showing no elemental peaks.

   If there are elemental peaks in the spectra, greater than twice the minimum detectable limit, the filters are considered contaminated. In this case, the project manager reviews the data, contacts the supplier, forwards the data to the technical division, and requests a new lot of filters.

5. The remaining half of the filters are tested for organic artifact by collecting four sets of side by side samples in an array of four to six collocated samplers. Sampling procedures are described in section 201. The filters are analyzed by XRF, PESA and PIXE following the standard analysis procedures listed in SOP 226.

   The elemental data are plotted for each set of side by side samples. The mean concentrations and standard deviations of the elemental data are calculated. The precision of the side by side samples, 100*(mean standard deviation / mean concentration), is calculated. The values for the 1994 acceptance tests are as follows:

Table 2 1994 Teflon Filter Quality Assurance Tests for Elemental Artifact

species	H	Na	Mg	Al	Si	P	S	Cl	K
mean precision	9	20	21	20	16	8	10	10	15
species	Cu	Ti	V	Cr	Mn	Fe	Ni	Cu	Zn
mean precision	13	27	39	26	41	13	28	41	20
species	As	Pb	Se	Br	Rb	Sr	Zr	Mo	OMH
mean precision	31	18	19	19	17	16	15	14	14

Outliers in OMH, points more than three standard deviations from the mean, are the primary concern. OMH is a derived measurement that assumes all sulfur is ammonium sulfate, and that none of the hydrogen measured is from nitrate (OMH = 13.75*(H - 0.25*S) where H is hydrogen concentration and S is the sulfur concentration). If outliers are observed, the samples should be examined with a microscope and scanning electron microscope to determine whether surface contamination is visible. Filters having organic artifact generally contain irregular patches of transparent material. This material, unidentified but associated with the manufacturing process, is difficult to see on unused filters. However, once aerosol sampling occurs, the transparent patches are easily spotted as no air flow, and thus little aerosol deposition, occurs in these regions.

The Quality Assurance Manager determines whether the new lot is acceptable as free of artifact. If there are no outlier points (points more than three standard deviations from the mean), and the elemental concentrations obtained compare well with previous measurements at the testing site, the filters are considered acceptable.

Teflon filter supplies are bulk stored in their original containers in a climate controlled environment until required for use. Lot documentation is maintained to record the application of the filters.

4.3.2 Nylon Filter Acceptance Testing

Consistency in pressure drop and composition are the primary requirements for nylon filters being used in the IMPROVE network. Gelman Nylasorb™ nylon filter material is purchased by UC Davis in 8.5" by 11" sheets separated by thin sheets of inert spacer paper. Each nylon filter sheet will provide eighty to ninety 25mm filters. One filter is punched out from each of thirty randomly selected filter sheets for acceptance testing. When certified, the entire lot is authorized for use in network operations.

The following tests are done by research technicians:

1. Randomly select 30 sheets from the lot being tested. Punch one 25 mm filter from each sheet following the procedures listed in section 4.1.2, Nylon filter procurement.

2. Measure the pressure drop across 20 randomly selected filters to verify the filters in the lot will have uniform thickness and thus similar flow characteristics.
   - Carefully load each filter into a cassette, being sure all parts are assembled properly.
   - Mount the cassettes on the leak check device in the air quality group lab.
   - Turn on the pump using the switch on the power strip near the leak check device.
   - Place the flow blocking device, a silicone bulb, over the open end of a cassette.
   - Open the valve below the cassette being checked by pressing down on the lever to allow air flow.
   - Record the pressure drop across the filter displayed on the small gauge in "Hg.

- Close the valve by lifting the lever to it's resting position and prepare to test the next cassette.
- If the readings for a cassette seem anomalously large (i.e. more than three standard deviations from the mean value of the other readings), take the cassette apart and re-assemble it, verifying that no cause for leaks is apparent.
- If the readings remain anomalously large, remove the filter from its cassette and install it in a new cassette.
- If the readings still remain anomalously large, the filter media is probably to blame and that sheet of filter material should be thrown out to avoid accidental use in the network.

3. Determine the expected flow variations due to the variations in pressure drop within the filter lot. This test simulates the change in flow rate that should be expected when the new filter lot is installed in samplers that were calibrated using filters from the current filter lot.
   a. Use the 20 filters mounted in cassettes in section 2 for this testing.
   b. Mount three filters from the current lot into cassettes.
   c. Install the three cassettes with filters from the current lot in an IMPROVE module in the testing area in the shop.
   d. Turn on the pump and the sampler by turning the white breaker switch in the base of the module to on, and the pump override switch on the face plate of the module to manual.
   e. Check the pressure drop of the three filters by, one at a time, pushing on the toggle switches on the face plate corresponding to the cassette being tested.
   f. Record the pressure drop data, then release the toggle switch and select the cassette having the mid range value as the primary cassette.
   g. Following the procedures of TI 176C , adjust the flow rate for the IMPROVE module to 23 liters per minute (lpm) using the primary cassette.
   h. Using the primary cassette, audit the sampler, according to the procedures in section TI 176C , and record the calibration equation.
   i. Remove the cassettes containing the current filter lot and install the cassettes containing the lot being tested.
   j. Check the pressure drop and magnehelic readings for each of the filters being tested by, one at a time, pushing on the toggle switches on the face plate corresponding to the cassette being tested.
   k. Record the pressure drop data, then release the toggle switch.
   l. Repeat the procedure until data for all 20 cassettes containing filters from the lot being tested has been recorded.
   m. Using the calibration equation derived from the audit in step h, calculate the flow rate from the magnehelic data recorded for each cassette.
   n. Calculate the average flow rate for the lot being tested
   o. If the average flow rate for the lot being tested varies by more than 2% from the average flow rate of the current lot, then all the IMPROVE samplers having a module B must be re calibrated once the new lot filters are installed.

This information must be passed to the project manager for approval and scheduling.

4. The ten remaining filters are sent for analysis by the ion contractor to verify that there are no abnormal artifacts.
   a. Place each selected filter in a petri dish and label the dishes "test 1" through "test 10"
   b. Package the filters in a petri dish box and ship them overnight to the ion contractor.
   c. Receive the data and determine the average concentrations and standard deviations for each species.
   d. Compare the results for the lot being tested with the results from the testing of the lot currently in use.
   e. Compare the results for the lot being tested with the results from current field blank analysis.
   f. Forward the results and comparisons to the project manager. If the measured concentrations for the two lots are within 5%, the tested blanks are lower than the analyzed field blanks, and the standard deviation is low, the filters are accepted for use.

4.3.3 Quartz Filter Acceptance Testing

Quartz filters for fine carbon aerosols are purchased, pre-fired, and analyzed by an external contractor. Specific requirements and procedures are included in Appendix C. Summarized actions are described here:

1. Carbon Contractor: Purchase 25 mm Pallflex 2500 QAT-UP quartz filters.

2. Carbon Contractor: Pre-fire the quartz filters at 900°C for at least four hours following procedures developed by the contractor.

3. Carbon Contractor: Visually inspect filters for holes or uneven texture over a light table. Remove rejects to a separate box.

4. Carbon Contractor: Select two filters for acceptance testing, mounting them in petri slides and labeling the slides with the lot number, and subjecting them to standard analysis procedures.

5. Carbon Contractor: Determine whether the lot passes the acceptance test (less than 1.5 µg organic carbon, 0.5 µg elemental carbon, and 2.0 µg total carbon). If the filters do not pass the acceptance test, the entire lot is discarded. If the filters pass, the lot is accepted for use.

6. Carbon Contractor: Ship the carbon filters to UC Davis in a cooled container.

4.3.4 Impregnated Quartz Filter Acceptance Testing

Impregnated filters for measuring gaseous SO_2 as SO_4 are both supplied and analyzed by an external contractor. Specific requirements and procedures are included in Appendix C. Summarized actions are described here.

1. SO_2 Contractor: Purchase 25 mm Pallflex QAT-UP filters.

2. SO_2 Contractor: Soak the filters for five minutes in a solution of 10 percent potassium carbonate and four percent glycerol in distilled de-ionized water. Remove from solution, and allow to drip free.

3. SO_2 Contractor: Dry the impregnated filters in a vacuum oven at 60 to 70° C under 15 to 20 " Hg vacuum for at least 15 minutes.

4. SO_2 Contractor: Inspect each filter for holes, wrinkles, torn edges, foreign particles, excessive wetness or dryness, uniform thickness, uniform appearance and color, stiffness, and shape (must be perfectly circular). Any filters not meeting the requirements are removed from the batch and thrown away.

5. SO_2 Contractor: Analyze 1% of the filters with ion chromatography for background sulfate levels and potassium ion concentration. If the concentrations are not within acceptable limits for sulfate or potassium ion concentrations, the lot will not be certified for use and will be thrown out.

6. SO_2 Contractor: Package the impregnated filters in sub lots of 100 and ship to UC Davis.

7. UC Davis: Receive the impregnated filters and retain them in a clean, cool, dry environment until required in the loading sequence.