

**Compendium of Methods
for the Determination of
Inorganic Compounds
in Ambient Air**

Compendium Method IO-3.1

**SELECTION, PREPARATION
AND EXTRACTION OF
FILTER MATERIAL**

Center for Environmental Research Information
Office of Research and Development
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Method IO-3.1

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Method IO-3.1
Selection, Preparation and Extraction of
Filter Material

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Chapter IO-3
CHEMICAL SPECIES ANALYSIS
OF
FILTER-COLLECTED SUSPENDED PARTICULATE MATTER (SPM)

Method IO-3.1
SELECTION, PREPARATION AND EXTRACTION OF
FILTER MATERIAL

1. Scope

1.1 This methodology consists of (1) filter media selection, (2) numbering and pre-field tare weighing of filters, (3) post-field final weighing of filters, (4) microwave or hot acid extraction, and (5) analysis for metal analysis by ICP, FAA, ICP/MS or GFAA.

1.2 Pre-field filters are conditioned in a room of constant humidity and temperature and are gravimetrically tared. After air samples have been collected, the filters are returned to the laboratory and conditioned as before and weighed. The final filter weight minus the tare weight is calculated. The procedure for the weighing of filters is based on 40 CFR 50, Appendix B, entitled "*Reference Method for the Determination of Suspended Matter in the Atmosphere (High-Volume Method)*."

1.3 After the post-field filter final weights have been obtained, the filter is subsampled by cutting a filter strip consisting of one-ninth of the overall filter and digested using a microwave or hot acid extraction technique; these extracts are then analyzed by one of many analytical techniques. The results are multiplied by a factor of 9 to obtain the actual total μg of each metal found on the entire 8" x 10" filter. Based upon the analysis of a blank filter, background metal concentration may be subtracted from the total metal concentration to get a net value. Therefore, the analytical results represent the total μg found on the 8" x 10" filter but do not represent the volume of air sampled.

1.4 Sectioning the filter for extraction is based on 40CFR50, Appendix B entitled "*Determination of Lead in Suspended Particle Matter Collected From Ambient Air*." The procedure for the microwave extraction is based on a method developed by EPA entitled *Microwave Extraction of Glass-Fiber Filters*, as identified in Section 2.2. This procedure has been modified for extracting quartz fiber filters.

2. Applicable Documents

2.1 ASTM Documents

- D4096 *Application of the High Volume Sample Method for Collection and Mass Determination of Airborne Particle Matter.*
- D1356 *Definition of Terms Related to Atmospheric Sampling and Analysis.*
- D1357 *Practice for Planning the Sampling of the Ambient Atmosphere.*
- D2986 *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test.*

2.2 Other Documents

- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: A Field Guide for Environmental Quality Assurance*, EPA-600/R-94/038a.
- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition)*, EPA-600/R-94/038b.
- *Reference Method for the Determination of Particulate Matter in the Atmosphere*, Code of Federal Regulations (40 CFR 50, Appendix J).
- *Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)*, Code of Federal Regulations (40 CFR 50, Appendix B).
- *Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air*, Federal Register 43 (194): 46258-46261.
- U. S. Environmental Protection Agency, *Microwave Extraction of Glass Fiber Filters*, Method Research and Development Division, RTP, NC 1989.

3. Apparatus

3.1 Equipment For Gravimetric Analysis

3.1.1 Controlled Temperature. Temperature between 15 and 30EC with less than ± 2 EC variation during equilibration period.

3.1.2 Controlled Humidity. Less than 50% relative humidity, constant within $\pm 5\%$

3.1.3 Analytical Balance. Sensitive to 0.1 mg, with weighing chamber designed to accept an unfolded 20.3 x 25.4 cm (8" x 10") filter.

3.1.4 Area Light Source. Similar to X-ray film viewer to backlight filters for visual inspection.

3.1.5 Numbering Device. Capable of printing identification numbers on the filters before they are placed in the filter conditioning environment if not numbered by the supplier.

3.1.6 Hygrothermograph. Capable of recording temperature and relative humidity in the weighing room.

3.2 Microwave Digestion Apparatus and Materials

3.2.1 Microwave Digestive System and Capping Station. With programmable power settings up to 600 watts, best source.

[Note: Commercial kitchen or home-use microwave should NOT be used for digesting samples. The oven cavity must be corrosion resistant and well ventilated. All electronics must be protected against corrosion for safe operation.]

3.2.2 PFA Teflon® Digestion Vessels. Capable of withstanding pressures of up to 120 psi. Pressure vessels capable of controlled pressure relief at pressures exceeding 120 psi (60-120 mL capacity), best source.

3.2.3 Teflon® PFA Overflow Vessel. Double ported (60-120 mL capacity), best source.

3.2.4 Rotating Table. Uniform exposure of samples within the oven.

3.2.5 Volumetric Glassware. 50-100 mL capacity (Class A borosilicate).

3.2.6 Bottles, Linear Polyethylene or Polypropylene with Leakproof Caps, for Storing Samples. Teflon® bottles for storing multielement standards (500 mL, 125 mL, and 30 mL), best source.

- 3.2.7 Centrifuge Tubes.** Oak Ridge polysulfone tubes with screw caps of polypropylene, 30 mL.
- 3.2.8 Nylon or Teflon® 0.45 µm Syringe Filters.** Acrodisc® No. 4438 or equivalent and syringes for rapid nonmetals contributing filtering.
- 3.2.9 Sterile Polypropylene Tubes with Screw Caps of Polypropylene, 15 mL Capacity.** Best source.
- 3.2.10 Pipette.** Automatic dispensing with an accuracy of setting 0.1 mL or better and repeatability of 20 FL, Grumman Automatic Dispensing Pipette, Model ADP-30DT or equivalent.
- 3.2.11 Particle Mask.** 3M, No. 8500, to be worn while cutting and handling glass-fiber filters.
- 3.2.12 Template.** Aid in sectioning the glass fiber filter. Federal Register 43 (194): 46258-46261.
- 3.2.13 Pizza Cutter, Thin Wheel.** Clean razor blade (< 1 mm).
- 3.2.14 Vortex Mixer.** VWR2 variable speed or equivalent.
- 3.2.15 Hydrochloric Acid.** Baker Instra-Analyzed, concentrated (sp.gr.1.19) or equivalent, for preparing samples.
- 3.2.16 Nitric Acid.** Baker Instra-Analyzed, concentrated (sp.gr.1.41) or equivalent, for preparing samples.
- 3.2.17 ASTM Type I Water.** ASTM D193.
- 3.2.18 Extraction Solution (5.55% HNO₃/16.75% HCl).** Prepare by adding in ~ 500 mL of Type I DI water, 55.5 mL of concentrated HNO₃, and 167.5 mL of concentrated HCl, dilute to one liter with Type I DI water.

3.3 Hot Acid Extraction Apparatus and Materials

3.3.1 Thermolyne Model 2200 Hot-Plate or Equivalent.

[Note: Temperature of the extracts may be monitored by the use of a beaker containing a thermometer and similar reagents as the samples.]

- 3.3.2 Volumetric Glassware.** 50-100 mL capacity (Class A borosilicate).
- 3.3.3 Bottles, Linear Polyethylene or Polypropylene with Leakproof Caps, for Storing Samples.** Teflon® bottles for storing multielement standards (500 mL, 125 mL, and 30 mL).
- 3.3.4 Centrifuge Tubes.** Polypropylene or Oak Ridge polysulfone tubes with screw caps of polypropylene, 30 mL (Nalgene 3119-0050/3115-0030 or equivalent).
- 3.3.5 Nylon or Teflon® 0.45 µm Syringe Filters.** Acrodisc® No. 4438 or equivalent and syringes for rapid nonmetals contributing filtering.
- 3.3.6 Sterile Polypropylene Tubes with Screw Caps of Polypropylene, 15 mL capacity.** Falcon Model No. 2099 or equivalent.
- 3.3.7 Pipette.** Automatic dispensing with an accuracy of setting 0.1 mL or better and repeatability of 20 FL. (Grumman Automatic Dispensing Pipette, Model ADP-30DT or equivalent).
- 3.3.8 Particle Mask.** 3M, No. 8500. To be worn while cutting and handling glass-fiber filters.
- 3.3.9 Vortex Mixer.** VWR2 variable speed or equivalent.
- 3.3.10 Hydrochloric Acid.** Baker Instra-Analyzed, concentrated (36.5%-38%/12.3 M) or equivalent, for preparing samples.
- 3.3.11 Nitric Acid.** Baker Instra-Analyzed, concentrated (70% 16M) or equivalent, for preparing samples.
- 3.3.12 ASTM Type I Water.** ASTM D193.

4. Filter Medium Selection

4.1 Introduction

4.1.1 In general, the filter medium depends on the purpose of the test. For any given standard test method, the appropriate medium will be specified. However, it is important to be aware of certain filter characteristics that can affect selection and use.

4.1.2 Selecting a filtration substrate for time-integrated SPM monitoring must be made with some knowledge of the expected characteristics and a pre-determined analytical protocol. For any given standard test method, the appropriate medium will normally be specified.

4.1.3 In high-volume sampling, four types of filter material to capture SPM are commonly used. They include cellulose fiber, quartz/glass fiber, mixed fiber, and membrane filter types. Selecting a filter depends upon variables such as background metal content, artifact formation, and affinity for moisture. The basic characteristics of the types of filter material used in high volume are outlined in Table 1 sampling. Useful filter properties are described in Table 2. Several characteristics are important in the selection of filter media. They are:

- **Particle Sampling Efficiency.** Filters should remove more than 99% of SPM drawn through them, regardless of particle size or flow rates.
- **Mechanical Stability.** Filters should be strong enough to minimize leaks during sampling and wear during handling.
- **Chemical Stability.** Filters should not chemically react with the trapped SPM.
- **Temperature Stability.** Filters should retain their porosity and structure during sampling.
- **Blank Correction.** Filters should not contain high concentrations of target compound analytes.

Quartz fiber filter medium is most widely used for determining mass loading. Weight stability with respect to moisture is an attractive feature. Quartz fiber filters provide high efficiency and collect airborne particles of practically every size and description. Typical characteristics of quartz fiber filters are (1) a fiber content of high purity quartz, (2) a binder of below 5% (zero for binderless types), (3) a thickness of approximately 0.5 mm, (4) a surface with no pinholes, and (5) an allowance of no more than 0.05% of smoke particles to pass through the filter at a pressure of 100mm of water with a flow rate of 8.53 m/min (28 ft/min), as determined by ASTM-D2986, *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test*.

Particulate matter collected on quartz fiber filters can be analyzed for many constituents. If chemical analysis is anticipated, binderless filters should be used. Glass is a commercial product generally containing test-contaminating materials; therefore, appropriate background corrections should be made. Background concentration of various metals associated with different grades of quartz fiber filters are documented in Table 3.

4.1.4 Silica fiber filters are used when it may be required or desirable to use a mineral fiber filter, which may later be extracted by strong reagents. These fibers are usually made by leaching glass fibers with strong mineral acids followed by washing with deionized water. The fibers are rather weak but can be formed into filter sheets using little or no binder. These filters have been recently developed and are commercially available.

4.1.5 For some purposes, airborne particles may be collected on cellulose fiber filters. Cellulose low-ash filters are especially useful when the filter is to be destroyed by ignition or chemical digestion. However, these filters have higher flow resistance (lower sampling rate) and have been reported to have much poorer

collection efficiency than the glass fiber media. Furthermore, cellulose is very sensitive to moisture conditions, and even with very careful conditioning before and after sampling, accurately weighing the collected particles is difficult. The filter should be enclosed in a lightweight metal can with a tight lid and weighed.

4.1.6 As documented in the 40 CFR Part 58, Appendices A and B, identify the filter specifications when used as part of the *Federal Reference Method for Particulate Matter in Ambient Air*. These specifications include (1) a quartz-fiber, nonhygroscopic filter, (2) a size of approximately 8" x 10", (3) an exposure area of approximately 63 in.², (4) a 99% collection efficiency as measured by ASTM-2986 (DOP test) for particles 0.3 μm diameter, (5) a pressure drop range of 42-54 mm Hg at a flow rate of 1.5 m³/min through the nominal exposed area, (6) a pH of 6 to 10, and (7) a maximum weight integrity of 2.4 mg.

4.2 Visual Filter Inspection

4.2.1 After purchased, all filters must be visually inspected for defects, and defective filters must be rejected if any are found. Batches of filters containing numerous defects should be returned to the supplier.

4.2.2 The following are specific defects to look for:

4.2.2.1 Pinhole. A small hole appearing as a distinct and obvious bright point of light when examined over a light table or screen, or as a dark spot when viewed over a black surface.

4.2.2.2 Loose material. Any extra loose material or dirt particles on the filter that must be brushed off before the filter is weighed.

4.2.2.3 Discoloration. Any obvious visible discoloration that might be evidence of a contaminant.

4.2.2.4 Filter nonuniformity. Any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity across the face of the filter.

4.2.2.5 Other. A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

4.2.3 Visually inspect each filter in front of an area light and observe for any specific defects listed above.

4.2.4 Use a renumbering stamp to code the filter on its noncollection side with a 7-digit code before tare weighing. The noncollection side of the filter is designated by the manufacturer printed number and by a mesh texture. The number code might be as follows:

Example: Filter Number Code = **9622001**

First 2 digits = yr, such as **96** for 1996

Third digit = project, such as **2**

Fourth digit = filter type, such as **2** for 8" x 10" quartz fiber, Whatman QMA type

Last 3 digits = filter number, such as **001**

5. Gravimetric Determination

5.1 Introduction

5.1.1 The filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain. The total volume of air sampled corrected to EPA standard conditions (25°C, 760 mm Hg) is determined from the measured flow rate and the sampling time. The concentration of TSP matter in the ambient air is computed as the mass of collected particles divided by the volume of air sampled (corrected to standard conditions) and expressed in μg/std m³ (see Inorganic Compendium Method IO-2.4). For samples collected at temperatures and pressures significantly different than standard conditions, the corrected

concentrations may differ substantially from actual concentrations Fg/m^3 , particularly at high elevations. The actual particulate matter concentration can be calculated from the corrected concentration using the actual temperature and pressure during the sampling period.

5.1.2 Verify that the weighing room conditions are within the limits. Filter equilibrium and weighing should be performed under controlled atmospheric conditions--a temperature of 25 ± 10 EC and a relative humidity $< 50\%$ (normally $50 \pm 5\%$ humidity).

5.1.3 Use the results from the motorized psychrometer to verify the temperature and relative humidity indicated by the hygrothermograph. Record the psychrometer values on the strip chart, along with the date, time, and your initials.

[Note: For traceability purposes, document your initials and full name in the front of the weighing room notebook.]

5.1.4 Record the room equilibration data on the Weighing Room Atmospheric Condition Form (see Table 4).

5.2 High Volume Filter Weighing Procedure

5.2.1 Filter Handling Procedure.

5.2.1.1 Filters should only be handled with finger cots or vinyl (nonpowdered) gloves. This procedure applies to filter handling in the field as well as in the weigh room.

5.2.1.2 Avoid using metal tweezers since the filters later will be used for metals analysis. When handling filter with gloved fingers or with any type of tweezers, avoid touching the sampled area.

5.2.2 Initial Weighing of High Volume Filter.

5.2.2.1 Upon receipt of new high volume filters (8" x 10" quartz fiber), take them to the climate controlled room, remove the paper and plastic envelope (wearing clean plastic gloves), place each on edge in a clean metal file rack, and cover with clean white paper towels.

5.2.2.2 Allow the filters to equilibrate in the metal file rack in the weighing room atmosphere for at least 24 h. Humidity and temperature must be within Federal Reference method specification, (i.e., $< 50\%$ and 15-35EC, respectively).

5.2.2.3 Zero the high volume balance before weighing.

5.2.2.4 Manually calibrate the balance. However, checks against two working NIST traceable weights (Class S) standards should be conducted before the daily weighing. If the difference between the traceable weights is more than 0.5 mg, do not use the balance until it has been repaired.

5.2.2.5 Record the results on the Weighing Balance Check Form (see Table 5).

5.2.2.6 Weigh each filter and record filter numbers and tare weights on the Filter Weighing Form (see Table 6).

5.2.2.7 Return the weighed filters to the plastic and paper envelopes.

5.2.2.8 Weigh filters in lots of approximately 100, if possible. After every tenth weighing, recheck the zero of the balance. The balance response should be ± 1 mg from 0. All differences should be corrected. Any difference exceeding 1 mg requires reweighing the previous ten filters. Any filter weight outside the normal range of 3.5-5.0 g requires immediate investigation. In addition, after every tenth filter weighing, the analyst should review at least one of the working standards. Once again, if this measurement disagrees from the verified value by more than 0.5 mg, reweigh the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both

working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 0.5 mg, then all weighings from the previous acceptable check must be repeated.

5.2.2.9 A second analyst should reweigh 10% of the filters. If the difference between the weights is less than 1.0 mg, the results are acceptable.

5.2.2.10 If the difference is greater than this limit, wait another 24 h and reweigh them.

5.2.2.11 If the results are still outside acceptable limits, wait another 24 h and reweigh them again. Then report the last reweigh values as the pre-field tare weights.

5.2.3 Final Weighing of High Volume Filter.

5.2.3.1 Exposed filters should be logged into the laboratory computer and received in individual manila folders, with computer printed identification labels affixed. No exposed filter should be touched until this label is affixed.

5.2.3.2 Condition all filters in the manner specified by the Federal Reference Method, as documented in Sections 5.1.2 and 5.2.2.

5.2.3.3 Weigh all filters according to the Tare Weighing Procedure in Section 5.2.2. Record final weights on the Filter Weighing Form (see Table 6).

5.2.3.4 For filters not to be analyzed, put an asterisk in the space preceding the four-letter code. Leave this space blank for samples to be analyzed. Sign and date the forms.

5.2.3.5 Archive asterisked high volume filters.

5.2.3.6 Have a second analyst reweigh 10% of the filters and verify that the weights have not changed.

- If the difference between the weights is less than 2.0 mg, the results are acceptable. Use the results from the first weighing.
- If the difference is greater than this limit, reweigh 100% of that lot and use the last reweigh weight.

5.2.3.7 Calculate and report the particulate matter concentrations as:

where:

$$\text{SPM} = \frac{(W_f - W_i) \times 10^6}{V_{\text{std}}}$$

SPM = mass concentration of suspended particulate matter (TSP or PM₁₀), µg/std m³.

W_i = initial weight of clean filter, g.

W_f = final weight of exposed filter, g.

V_{std} = air volume sampled, converted to standard conditions (25°C and 760 mm Hg), std m³.

10⁶ = conversion of g to µg.

5.3 Dichotomous and Partisol® Filter Weighing Procedure

5.3.1 Initial Weighing of Dichotomous or Partisol® Filters.

5.3.1.1 Fabric filters, 37-mm or 47 mm (as appropriate for the samples) in diameter, with a circumferential plastic reinforcing ring are usually supplied in small boxes. Open the boxes in the climate-controlled room under conditions suitable for high volume weighing. Cover with a clean paper towel and allow to equilibrate for 24 h.

5.3.1.2 Weigh filters on a Mettler microbalance; each balance is identified by a balance number.

5.3.1.3 Assign each balance a block of 7-digit sample numbers to be used sequentially. Assign a sample number to each filter when it is tared.

[Note: Inaccuracies in this aspect of the procedure will cause irremedial sample loss.]

5.3.1.4 Turn on the microbalance and allow it to warmup for at least 15 min. If the balance is used daily, leave it on at all times.

5.3.1.5 Manually calibrate the microbalance with two working NIST traceable weight (Class S) standards (for example, a 100-mg standard and a 200-mg standard). If the difference between the traceable weights is more than 3 µg, reweigh the working standards. Record the Results on the Weighing Balance Check Form (see Table 7).

5.3.1.6 If the values still disagree, troubleshoot and take appropriate corrective action, which may include (1) recertifying the working standards against the laboratory primary standards, and/or (2) having a service technician repair the microbalance.

5.3.1.7 Using clean nonserrated tweezers that will not damage the filter, remove the filter from the Lexan jig or filter cassette and place it on the weighing pan. Turn the release lever to "1" and dial in tare weights until a reading between 0.000 and 7.000 is obtained. Allow the reading to stabilize (which may require 2 to 4 min). Record the reading and the dialed-in tare weight on the Filter Weighing Form (see Table 8). Return the release lever to "0" and remove the filter from the weighing pan.

[Note: Do not use metal tweezers.]

5.3.1.8 Place a white label on a clean 50-mm diameter plastic petri dish (tight fitting lid type).

5.3.1.9 Assign a sample number to each filter (from those assigned to that balance), taking extreme care to avoid duplication or missed numbers.

5.3.1.10 Record the assigned sample number on the petri dish label, leaving sufficient room for one more letter to be written following the number. Do not record the balance number on this label.

5.3.1.11 Record the balance number, the assigned sample number, the dialed-in tare weight, and the digital-displayed tare weight on the sample form. Number each sheet of the form sequentially in the upper right-hand corner. Write "Tare Weight, Dichot or Partisol® Filters" on the top of each sheet. When bound, these forms may serve as the laboratory notebook.

5.3.1.12 Place the weighed filter in its numbered petri dish for future use.

5.3.1.13 After every tenth filter weighing, the analyst should check the "zero" and reweigh at least one of the working standard. Record the measurement on the Weighing Balance Check Form. Once again, if this measurement disagrees from the verified value by more than 3 µg, reweigh the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 3 µg then all weighings from the previous acceptable check must be repeated.

5.3.1.14 At the end of the weighing session, at least 10% of the filters should be reweighed by a second analyst. Record the replicate measurement on the Filter Weighing Form (see Table 6). If the replicate measurement disagrees from the original measurement by more than 15 µg, reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

5.3.1.15 Return the filter to the filter-handling container, replace the lid, and return it to conditioning chamber to protect it from contamination prior to sampling.

5.3.2 Final Weighing of Dichotomous or Partisol® Filter.

5.3.2.1 Filters should be returned from the field with a computer printed label affixed to the petri dish. The label should contain a five-character identification code that is different from the original sample number, a balance ID, the balance tare, and other information. All filters should be accompanied by extra labels. Some will have the words "To Be Analyzed" on the labels. The filter in each petri dish should rest in a Lexan jig or filter cassette.

5.3.2.2 Weigh each filter on the balance on which its tare weight was obtained. In the climate-controlled room, group the filters according to recorded balance numbers. Open the petri dishes, making certain that lids are placed under the bottoms and that no mixup occurs. Cover with a clean white paper towel and allow to equilibrate.

5.3.2.3 Repeat Section 5.3.1.4 to 5.3.1.6 of the filter tare weighing procedure.

5.3.2.4 Using clean, nonserrated tweezers that will not damage the filter, remove the filter from the Lexan jig or filter cassette and place it on the weighing pan. Dial in the tare weight recorded on the information label and turn the release lever to "1." Allow the reading to stabilize (which may require 2 to 4 min). Record the reading and the dialed-in tare weight. Return the release lever to "0" and remove the filter from the weighing pan.

5.3.2.5 After every tenth filter weighing, the analyst should check the "zero" and reweigh at least one of the working standards. Record the measurement on the Weighing Balance Check Form. Once again, if this measurement disagrees from the verified value by more than 3 ug, review the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 3 µg, then all weighings from the previous acceptable check must be repeated.

5.3.2.6 At the end of the post weighing session, at least 10% of the filters should be reweighed by a second analyst. Record the replicate measurement on the Filter Weighing Form (see Table 8). If the replicate measurement disagrees from the post measurement by more than 15 µg, reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, and/or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

5.3.2.7 If the dichotomous filter is not to be analyzed, use the tweezers to place it in a small glass envelope to which one of the extra labels has been affixed. Place an asterisk before the five-character code on the form. Deliver these filters to the filter bank for archiving.

5.3.2.8 If the filter is to be analyzed, use tweezers to carefully put it back into the petri dish. Place the petri dish **carefully** in a box.

5.3.2.9 Place a label on a sheet of 8 ½" x 11" paper for NAA, XRF, or other analysis as appropriate. Indicate the page number and balance number on each list. Keep the samples in the box in an order corresponding with the lists.

5.3.2.10 Without jostling the box, deliver it, the two lists, and the original Field Test Data Sheets with two copies of each to the sample custodian who will initial the original forms and return them upon receipt.

5.3.2.11 Calculate and report the particulate matter concentration for both fine and coarse samples utilizing the following equation:

$$PM = \frac{(W_f - W_i) \times 10^6}{V_{std}}$$

where:

PM = mass concentration of particulate matter (TSP, fine or coarse fraction), $\mu\text{g}/\text{std m}^3$.

W_i = average initial weight of clean filter, g.

W_f = average final weight of exposed filter, g.

V_{std} = air volume sampled, converted to standard conditions, std m^3 (see Inorganic Compendium Method IO-2.4).

10^6 = conversion of g to μg .

5.4 Transport of Filters

5.4.1 After collecting samples, transport the filters to the laboratory, taking care to minimize contamination and loss of the sample. Glass fiber filters should be transported or shipped in a shipping envelope. Cover the exposed surface of the membrane filters with an unexposed filter and seal the filter in plastic filter holders.

5.4.2 Assign numbers to the filters and log them into the data record form, ensuring that any necessary sampling information is included (Untreated filter samples may be stored indefinitely.)

5.4.3 Provide one blank sample with every 10 actual samples. No air is drawn through the blank filter, but it is subjected to the same handling and shipping manipulations as the actual samples.

6. Extraction of Glass Fiber Filters in Preparation for Metal Analysis

6.1 Introduction

This section describes both a microwave-extraction procedure and a hot-acid extraction procedure to extract inorganics from the particulate quartz glass-fiber filter. Following extraction, target analytes are analyzed by ICP, ICP/MS, FAA, or GFAA.

6.1.1 Ambient air quartz fiber filters should be received folded in half lengthwise with the particulate material inward and enclosed in protective envelopes. Store these protective envelopes approximately 15E-30EC until analysis.

6.1.2 The maximum sample holding times is usually 180 days. Analyze the samples within 180 days, even if these times are less than the maximum data submission times allowed.

6.2 Microwave Extraction Procedure

6.2.1 Filter Cutting Procedure.

6.2.1.1 Cut a 1" x 8" strip from the 8" x 10" filter using a template (see Figure 1) and cutting tool (see Figure 2) as described in the Federal Reference Method for lead. Use a laboratory microwave extraction system to extract the metals with a hydrochloric/nitric acid solution. After cooling, mix the digestate and use

Acrodisc® syringe filters to remove any insoluble material. Microwave extraction is used to prepare samples for ICP, ICP/MS, FAA, or GFAA.

6.2.1.2 Prior to use, acid wash the plexiglass filter template, the polysulfone centrifuge tubes and caps, and all other laboratory equipment that will come into contact with the filter samples to prevent contamination.

6.2.1.3 Using vinyl gloves, place the acid-cleaned filter template and cover inside a balance hood for cutting quartz fiber filters.

6.2.1.4 Wipe plexiglass template base, cover, and cutting blade with a clean, dry Kimwipe® to prevent sample cross-contamination.

6.2.1.5 Unfold the 8" x 10" quartz filter to be sectioned and carefully place sampled side up (numbered side down) within the plexiglass template filter margins.

6.2.1.6 Carefully (without disturbing sampled area of filter) place the grooved cover, notch side down, within the margins of the base template. Use a clear cutting blade to cut a 1" x 8" strip.

6.2.1.7 Using gloved fingers, accordion-fold or tightly roll the filter strip and transfer on edge to an acid cleaned polysulfone® tube, labeled with wax pencil. DO NOT use barcodes or tape in microwave.

6.2.1.8 Clean filter template between samples with dry Kimwipes®. (Gloves should be changed after 50 filters to minimize cross-contamination.)

6.2.1.9 Duplicate sample frequency is normally 1 per 20 field samples (see Table 9). Prepare a sample filter duplicate by moving the template cover to a second portion of the field collected filter. Cut an additional filter strip by moving the template cover to a second section of the filter and repeat Sections 6.2.1.6 through 6.2.1.8 above using a separate polysulfone tube.

6.2.1.10 Select a field collected filter for matrix spiking. In addition to the filter strip cut for determining metals, section a second portion of the filter, and fortify (spike) with target metals.

6.2.1.11 Prepare matrix spike samples at a frequency of 1 per 20 field samples or a minimum of 1 per extraction day (see Table 9). Move the template to a second section of the filter and repeat Sections 6.2.1.6 through 6.2.1.8, using a separate polysulfone tube and spike as shown in Table 9.

6.2.2 Microwave Calibration Procedure. Calibration of the microwave unit is a critical step prior to its use. In order that absolute power settings may be interchanged from one microwave unit to another, the actual delivered power must be determined, which allows the analyst to relate power in Watts to the partial power setting of the unit (% Power).

Calibration of a laboratory microwave unit (see Figure 3) depends on the type of electronic system used by the manufacturer. If the unit has a precise and accurate linear relationship between the output power and the scale used in controlling the microwave unit, the calibration can be a three-point calibration in the range of 50% to 100% power. If the unit does not prove linear (± 10 W) using the three-point technique, a multiple-point calibration is necessary. A bracketed calibration range of the digesting power to be used is recommended for determining the calibration points. If the unit power calibration needs multiple-point calibration, the point where the linearity begins must be identified. For example, a calibration at 100, 99, 98, 97, 95, 90, 80, 70, 60, and 50% power settings can be applied and the data plotted. The nonlinear portion of the calibration curve can be excluded or restricted. Each percent is equivalent to approximately 5.5-6.5 W and becomes the smallest unit of power that can be controlled. If 20-40 W are contained from 99-100%, that portion of the microwave calibration is not controllable by 3-7 times that of the linear portion of the control scale and will prevent duplication of precise power conditions specified in that portion of the power scale.

6.2.3 Microwave Power Evaluation. The equation in the following section evaluates the power available for heating in a microwave cavity. The variables are determined by measuring the temperature rise in 1 kilogram of water exposed to electromagnetic radiation for a fixed period of time. The following procedure is used for evaluating each calibration point, represented as % power output for each microwave.

6.2.3.1 Measure and record a 1 kilogram (1,000 g ± 0.1 g) sample of room temperature (23E± 2EC) distilled water in a thick-walled microwave transparent (Teflon®) beaker for each calibration point.

6.2.3.2 Measure and record the initial temperature of the water, (T_i), to within 0.1EC. The starting temperature should be between 22 and 26EC.

6.2.3.3 Place the Teflon® beaker in microwave and irradiate at full power (100% point) for 2 min (120 s). Each calibration point (i.e., 100%, 50% or multi-points) requires a separate clean beaker containing water at room temperature.

6.2.3.4 Remove beaker from the microwave and measure and record the maximum final temperature (T_f) to 0.1EC, within 30 s of the end of irradiation. This process should be done while stirring continuously (an electronic stirrer using a large stir bar works best).

Calculate the microwave power according to the following formula:

$$\text{Power} = \frac{K \times C_p \times M \times \Delta T}{t}$$

$$\frac{K \times C_p \times M}{t} = 34.87$$

$$\text{Power} = 34.87 \times \Delta T$$

where:

Power = The apparent power absorbed by the sample, watts (W = joule-s⁻²).

K = The conversion factor for thermochemical calories-s⁻¹ to W = 4.184.

C_p = The heat capacity, thermal capacity, or specific heat (cal-g⁻¹-EC⁻¹ = 1.0 for water).

M = The mass of the sample, grams.

ΔT = T_f - T_i, EC.

t = Time, s.

6.2.3.5 Derive an equation for the linear portion of the calibration range and determine the equivalent value in watts of the arbitrary setting scale. Use the actual power in watts to determine the appropriate setting of the particular microwave unit being used. Each microwave unit will have its own (% power) setting that corresponds to the actual power (in wattage) delivered to the samples.

6.2.3.6 An initial multipoint power evaluation should be performed for each microwave unit. If linear, the calibration should be checked on a regular basis, using the 3-point calibration verification routine. A single point verification may be appropriate when utilizing a single power output for digestion. If any part of the power source to the microwave has been serviced or altered, the entire calibration must be reevaluated.

6.2.4 Cleaning Procedure for PFA Vessels. All digestion vessels must be acid cleaned and rinsed with reagent water prior to use to prevent contamination.

6.2.4.1 Wash each PFA vessel with deionized detergent and rinse with reagent water.

6.2.4.2 Add 10 mL concentrated HNO₃ to each of 12 vessels, cap, and place in microwave.

6.2.4.3 Heat vessels at 100% power in microwave for 10 min as recommended by CEM (microwave manufacturer). Rinse the vessels with copious amounts of deionized, distilled water prior to use for any

analyses. If only 6 vessels are to be cleaned, 70% power may be utilized, which corresponds to approximately 5% per vessel.

6.2.5 Digestion Procedure for Microwave Extraction For Ambient Filter Samples.

[Note: Nitric and hydrochloric acid fumes are toxic. Prepare in a well-ventilated fume hood. Mixing results in an exothermic reaction. Stir slowly.]

6.2.5.1 Using vinyl gloves or plastic forceps, retrieve the filter strip from Section 6.2.1 and place on its edge in a labeled centrifuge tube. Using the plastic forceps, crush the filter strip down into the lower portion of the centrifuge tube to ensure acid volume will cover entire filter.

[Note: A breathing mask and vinyl gloves are required for safety of personnel handling dry glass-fiber filters. The breathing mask prevents the inhalation of minute glass shards and particulate material. The gloves protect the skin from the same materials and also prevent contamination of the sample by skin secretions. A recommended alternate to the use of a breathing mask would be performing cutting and transfer operations involving sample filters in a laminar flow hood, if available.]

[Note: More than one strip from a filter should be extracted to ensure adequate sample volume for sample and QC sample analysis. Blank filter samples should be extracted and analyzed, and digestion blanks should be run to ensure low levels of metals in the reagents used.]

6.2.5.2 Using a preset calibrated automatic dispensing pipette or Class A glass pipette, add 10.0 mL of the extraction solution to each of the centrifuge tubes. The acid should cover the strip completely. The sequence of adding the filter strip and acid to the centrifuge tube may be reversed, if more convenient, without affecting the results. Place the centrifuge tube in a Teflon® PFA vessel containing 31 mL of deionized water. Continue this process for a total of 12 samples to maximize microwave capacity.

6.2.5.3 Place the PFA vessel caps with the pressure release valves on the vessels hand-tight and tighten using the capping station to a constant torque of 12 ft-lb. Weigh and record the capped vessel assembly to the nearest 0.01 g. Place the vessels in the microwave carousel. Connect each sample vessel to the overflow vessel using the Teflon® PFA connecting tubes (see Figure 3).

6.2.5.4 Place the carousel containing the 12 vessels onto the turntable of the microwave unit. Any vessels containing 10 mL of acid solution for analytical blank purposes are counted as sample vessels. Irradiate the sample vessels at 486 W (power output) for 23 min. (Based on the calibration of the microwave as previously described). If fewer than 12 samples are to be digested, adjust the microwave system by reducing the power so that equivalent digesting power is delivered to the smaller sample batch. Generally, each vessel represents approximately 5% power. Therefore, a reduction in W would be reduced by 30% if only 6 vessels are digested. This reduction is only approximate, and each microwave unit will produce a different level of power output.

6.2.5.5 At the end of the microwave program, allow the pressure to dissipate (venting may be utilized with caution), then remove the carousel containing the vessels and cool in tap water for 10 min. Weigh the capped vessels assembly to the nearest 0.01 g and compare to the initial weight to verify no loss of sample. The initial and final weights should compare within 0.1 g. If the initial and final weights do not agree within 0.01 g, the appropriate action must be taken which may include rejecting the digested sample. Using the capping station uncapped the microwave vessels, remove the labeled centrifuge tubes containing samples and discard the water in the PFA vessels.

6.2.5.6 Using a calibrated automatic dispensing pipette or a Class A glass pipette, add 10 mL of deionized distilled water to each centrifuge tube. Cap the centrifuge tube tightly and vortex (mix) the contents thoroughly for 2-3 minutes to complete extraction. Using a nylon or teflon syringe pull-up a volume

of sample from the centrifuge tube, place Acrodisc filter on syringe and dispense into a pre-labeled sterile 15 mL centrifuge tube. Continue until centrifuge tube contains 10 mL of filtered digestate.

6.2.5.7 The final extraction volume is 20 mL based upon the above procedure. The final extraction solution concentration is 3% HNO₃/8% HCl. The filtered sample is now ready for analysis. Store for subsequent analysis by one or more of the Inorganic Compendium methods.

6.3 Hot Acid Extraction Procedure

6.3.1 Introduction. A hot extraction procedure to solubilize metals from the glass-fiber filter for subsequent analysis by ICP, ICP/MS, FAA, or GFAA is described in this method. An acid extraction solution is used to extract the metals from the quartz filter on a hot-plate.

6.3.2 Summary of Method.

6.3.2.1 Use the hot-acid extraction procedure as an alternate when microwave technology is not available.

6.3.2.2 Cut a 1" x 8" strip from the 8" x 10" filter as described in Federal Reference Method for lead. The inorganics are extracted from the filter strip by a HCl/HNO₃ acid solution using a hot acid extraction procedure. After cooling, pour the digestate rinses to a volumetric flask and dilute to volume. Filter to remove any insoluble material.

6.3.3 Hot Acid Extraction Procedure.

6.3.3.1 Using vinyl gloves or plastic forceps, retrieve the strip from Section 6.2.1 and place it in a labeled 150 mL Griffin beaker. Place the filter strip down into the lower portion of the beaker to ensure acid volume will cover entire filter.

[Note: More than one strip from a filter should be extracted to ensure adequate sample volume for sample and QC sample analysis. Blank filter samples should be extracted and analyzed and digestion blanks should be run to ensure low levels of metals in the reagents used.]

6.3.3.2 Using a preset calibrated automatic dispensing pipette or Class A glass pipette, add 10 mL of extracting acid (see Section 6.2.5.2) for analysis.

[Note: The acid should cover the strip completely.]

6.3.3.3 Place beaker on the hot-plate, contained in a fume hood, and reflux gently while covered with a watch glass for 30 min. Do not allow sample to dry. Remove the beakers from the hot-plate and allow to cool.

[Caution: Nitric acid fumes are toxic.]

6.3.3.4 Rinse the beaker walls and wash with D.I. water. Add approximately 10 mL reagent water to the remaining filter material in the beaker and allow to stand for at least 30 min. This critical step must not be deleted; it allows the acid to diffuse from the filter into the rinse. Transfer the extraction fluid in the beaker to a 20 mL volumetric flask or other graduated vessel. Rinse the beaker and any remaining solid material with Type I water and add the rinses to the flask. Some solids from the filter may be transferred to the flask with the rinses; this is acceptable. Dilute to the mark with Type I water and shake.

6.3.3.5 Using a nylon or Teflon[®] syringe, pull-up a volume of sample from the centrifuge tube, place disc filter on syringe, and dispense into a pre-labeled sterile 15 mL centrifuge tube. Continue until centrifuge tube contains 10 mL of filtered digestate.

6.3.3.6 The final extraction volume is 20 mL based upon the above procedure. The final extraction solution concentration is 3 % HNO₃/8% HCl. The filtered sample is now ready for analysis. Store for subsequent analysis by one or more of the Inorganic Compendium methods.

TABLE 1. CHARACTERISTICS OF FILTER MEDIUM

Cellulose Fiber (Cellulose Pulp)
<ul style="list-style-type: none"> • Low ash • Maximum temperature of 150EC • High affinity for water • Enhances artifact formation for $\text{SO}_4^{=}$ and NO_3^- • Good for x-ray/neutron activation analysis • Low metal content
Quartz Fiber (Quartz spun with/without organic binder)
<ul style="list-style-type: none"> • Maximum temperature up to 540EC • High collection efficiency • Non-hydroscopic • Good for corrosive atmospheres • Very fragile however • Difficult to ash; good with extraction
Synthetic Fiber (Teflon® and Nylon®)
<ul style="list-style-type: none"> • Collection efficiency > 99% for 0.01 μm particles • Low artifact formation • Low impurities • Excellent for X-ray analysis • Excellent for determining total mass due to non-hydroscopic nature • Nylon fiber good for HNO_3 collection
Membrane Fiber (Dry gel of cellulose esters)
<ul style="list-style-type: none"> • Fragile; requires support pad during sampling • High pressure drop • Low residue when ashed

TABLE 2. SUMMARY OF USEFUL PHYSICAL PROPERTIES OF VARIOUS FILTER MEDIUMS

Filter and Filter Composition	Density, mg/cm ²	pH	Filter Efficiency %
Teflon® (Membrane) (CF ₂) _n (2 µm Pore Size)	0.5	Neutral	99.95
Cellulose (Whatman 41) (C ₆ H ₁₀ O ₅) _n	8.7	Neutral (Reacts with HNO ₃)	58% at 0.3 µm
Glass Fiber (Whatman GF/C)	5.16	Basic pH - 9	99.0
"Quartz" Gelman Microquartz	6.51	pH - 7	98.5
Polycarbonate (Nuclepore) C ₁₅ H ₁₄ + CO ₃ (0.3 µm Pore Size)	0.8	Neutral	93.9
Cellulose Acetate/Nitrate Millipore (C ₉ H ₁₃ O ₇) _n (1.21 µm Pore Size)	5.0	Neutral (Reacts with HNO ₃)	99.6

TABLE 3. EXAMPLE OF TYPICAL BACKGROUND METAL CONCENTRATION (µg/FILTER) OF VARIOUS GRADES AND TYPES OF FILTERS

Metal	1	4I	42	542	Filter 17ch	Grades GF/A	EPM 2K	934QH	QMA	QMB
Al	1	2	2	1	--	4,300	170	2,950	4	--
Ag	--	--	--	--	--	< 1	< 1	--	< 1	--
As	< 0.02	< 0.02	< 0.02	< 0.02	--	< 6	< 6	--	< 6	--
B	1	< 2	2	2	--	6,100	42	--	42	--
Ba	< 1	< 1	< 1	< 1	--	8,500	50	< 1	10	--
Be	--	--	--	--	--	0.2	< 1	0.1	< 1	< 1
Bi	--	--	--	--	--	0.3	< 4	0.8	< 4	7
Ca	185	13	13	9	29	2,500	540	7,000	85	--
Cd	--	--	--	--	--	1	< 1	0.8	0.2	< 1
Co	--	--	--	--	--	11	< 1	5.5	1.1	< 1
Cr	0.3	0.3	0.3	0.7	--	0.2	4.5	3.0	1.6	0.7
Cu	1.2	0.1	0.4	0.2	0.6	56.0	1	7	3.4	3.4
Fe	5.0	6.0	6.0	3.0	5.0	100	15	265	23	29
Mg	< 0.005	--	< 0.005	< 0.005	--	--	--	--	--	--
K	4.0	1.5	1.5	0.6	7	775	573	125	--	--
Mg	7	1.8	1.8	1.0	6	1,090	238	2,800	--	--
Mn	0.1	< 0.05	< 0.05	< 0.05	--	8.0	< 1	2	0.5	< 1
Mo	--	--	--	--	--	2.0	< 2	4	< 2	< 1
N	28	--	12	260	--	--	--	--	--	--
Na	160	40	37	8	30	1,830	1,020	1,100	280	--
Ni	--	--	--	--	--	6.0	--	2.5	3.4	2.2
Pb	0.4	0.2	0.2	0.1	1	10	2.5	2.0	2.3	8.3
S	17	< 5	< 5	< 5	--	26	--	--	--	--
Sb	< 0.02	< 0.02	< 0.02	< 0.02	--	< 4	< 4	--	< 4	--
Si	15	< 2	< 2	< 2	--	--	--	--	--	--
Ti	--	--	--	--	--	0.1	< 1	< 30	< 1	< 4
V	--	--	--	--	--	0.1	--	10	--	< 4
Sn	2.5	0.6	0.6	0.3	--	5,500	15	34	10	13

TABLE 9. EXAMPLE QUALITY CONTROL SAMPLES

Type	Frequency	Contains 1"x8" filter strip	Comments ^a
Method Blank	1 per 20 samples	No	Containing reagents only, to evaluate background contributions from reagents.
Filter Lot Blank	1 per filter lot change	Yes	Analyzed prior to use of new filter lot.
Filter Duplicate	1 per 20 samples	Yes	This is a second 1"x8" filter strip cut from a single field sample.
Matrix Spike ^b	1 per 20 samples	Yes	See individual methods.
LCS ^c	1 per extraction day	Yes	See individual methods.

^aThese multimetal stock standards can be acquired from Spex Industries, Inc., Inorganic Ventures Inc., or equivalent.

^bThe matrix spike (MS) is a 1"x8". strip cut from a field sample filter and spiked at a target level.

^cThe Lab Control Sample (LCS) is a blank filter spiked with the same concentration of metals as the matrix spike.

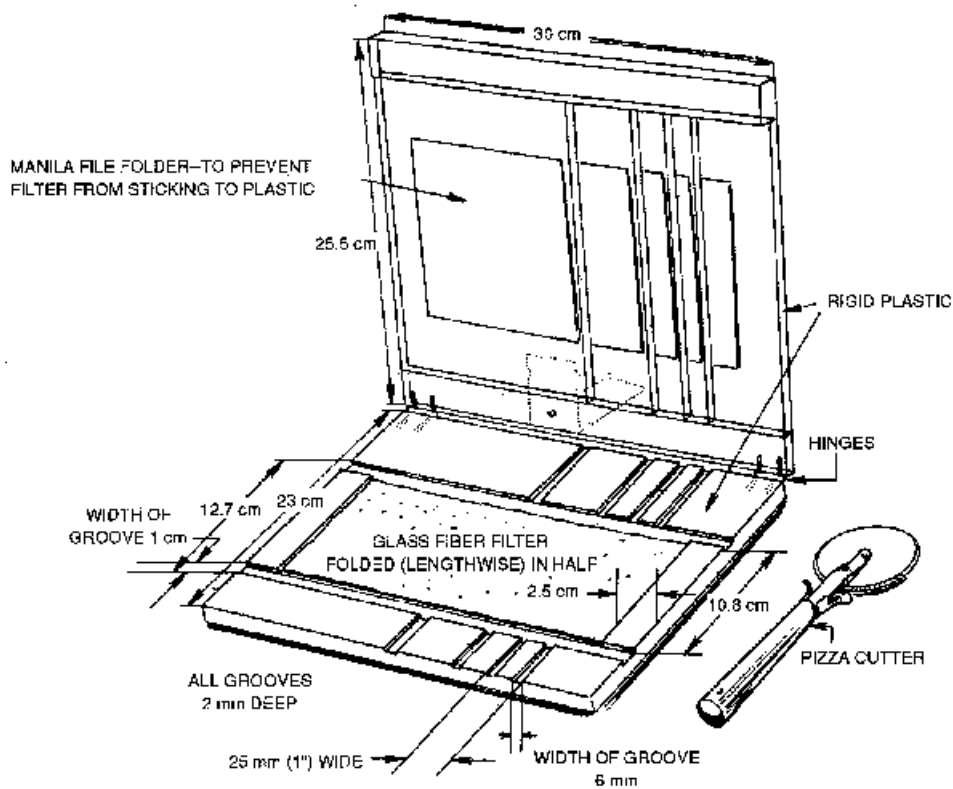


Figure 1. Example of templates for cutting filters.

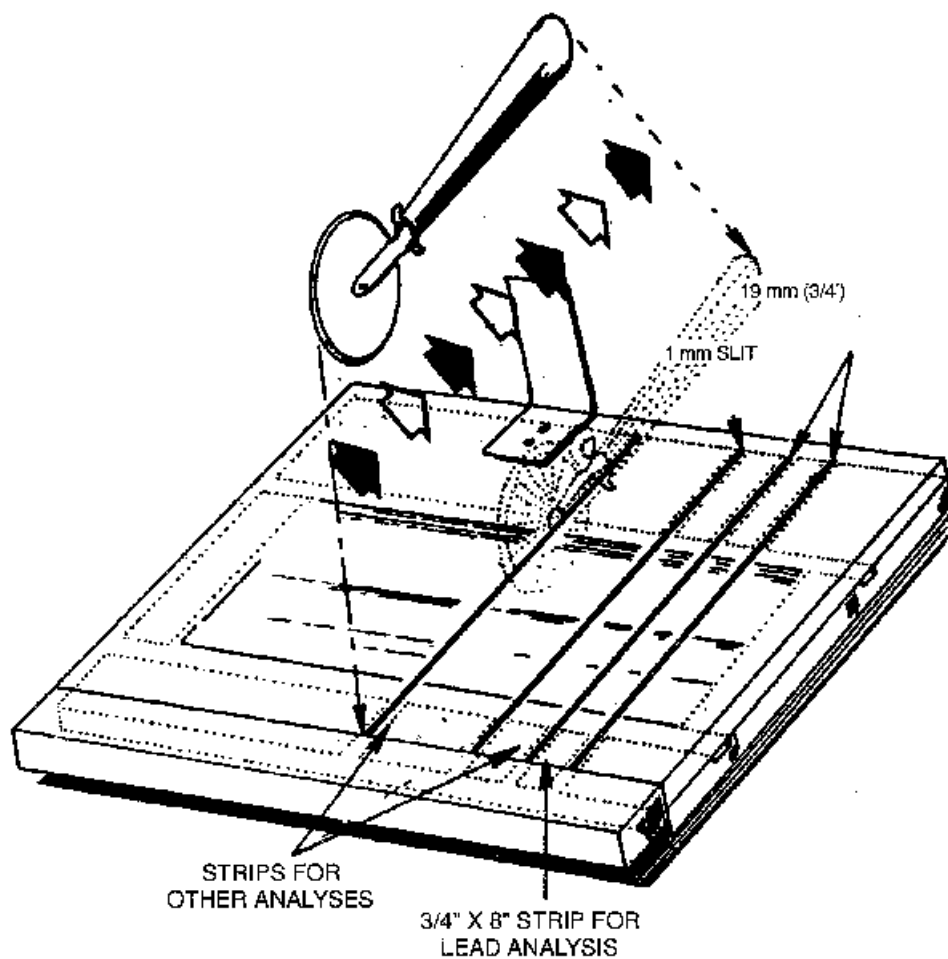


Figure 2. Diagram of filter cutting procedure.

MICROWAVE EXTRACTION

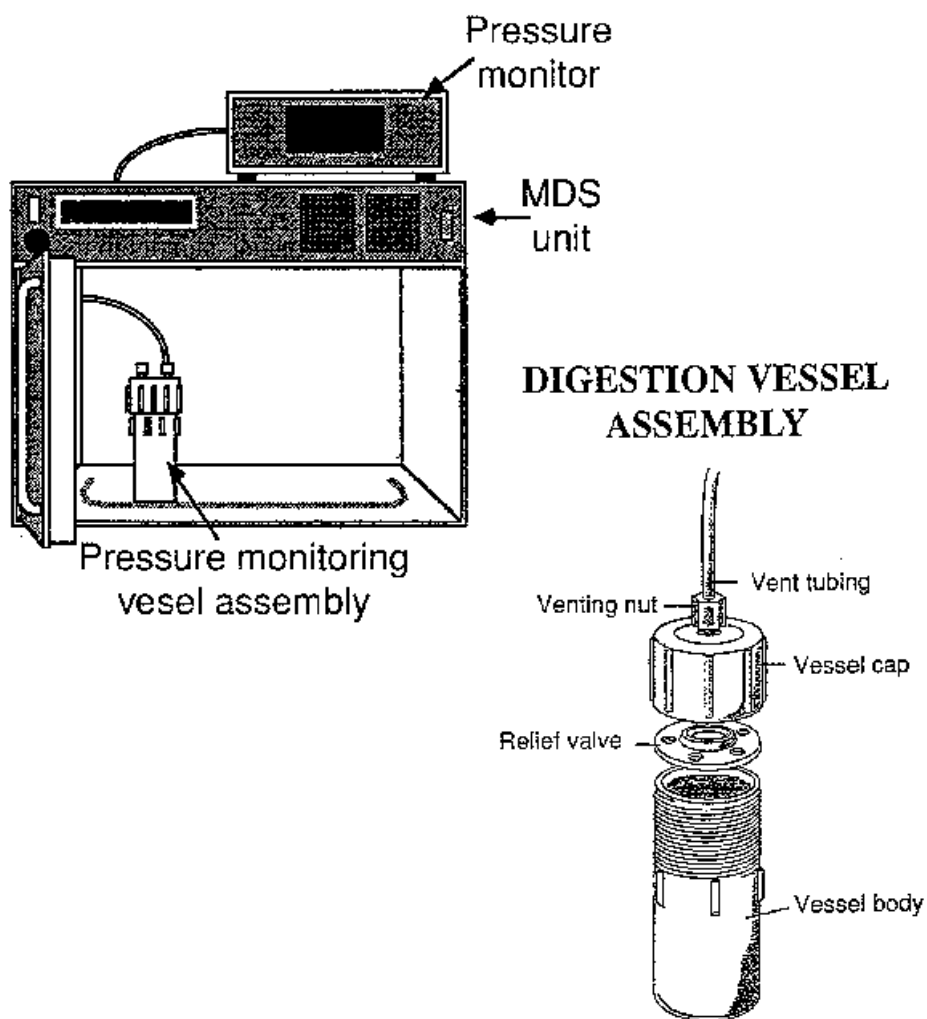


Figure 3. Example of microwave digestion system.