Chapter IP-8

DETERMINATION OF ORGANOCHLORINE PESTICIDES IN INDOOR AIR

1. Scope

This document describes a method for sampling and analysis of a variety of organochlorine pesticides in indoor air. The procedure is based on the adsorption of chemicals from indoor air on polyurethane foam (PUF) using a low volume sampler. The low volume PUF sampling procedure is applicable to multicomponent atmospheres containing organochlorine pesticide concentrations from 0.01 to 50 $\mu g/m^3$ over 4- to 24-hour sampling periods. The detection limit will depend on the nature of the analyte and the length of the sampling period. The analysis methodology described in this document is currently employed by laboratories using EPA Method 608. The sampling methodology has been formulated to meet the needs of pesticide sampling in indoor air. The sampling methodology involves a low volume (1 to 5 L/minute) sampler to collect vapors on a sorbent cartridge containing PUF. Airborne particles may also be collected, but the sampling efficiency is not known. Pesticides are extracted from the sorbent cartridge with 5% diethyl ether in hexane and determined by gas-liquid chromatography coupled with an electron capture detector (ECD). For some organochlorine pesticides, high performance liquid chromatography (HPLC) coupled with an ultraviolet (UV) detector or electrochemical detector may be preferable. This method describes the use of an electron capture detector.

2. Significance

2.1 Pesticide usage and environmental distribution are common to rural and urban areas of the United States. The application of pesticides can cause adverse health effects to humans by contaminating soil, water, air, plants, and animal life.

2.2 Many pesticides exhibit bioaccumulative, chronic health effects; therefore, monitoring the presence of these compounds in ambient air is of great importance.

2.3 Use of portable, low volume PUF sampling system allows the user flexibility in locating the apparatus. The user can place the apparatus in a stationary or mobile location. The portable sampling apparatus may be positioned in a vertical or horizontal stationary location (if necessary, accompanied with supporting structure). Mobile positioning of the system can be accomplished by attaching the apparatus to a person to test air in the individual's breathing zone. Moreover, the PUF cartridge used in this method provides for successful collection of most pesticides.

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1.2 The low volume PUF sampling procedure is applicable to multicomponent atmospheres containing organochlorine pesticide concentrations from 0.01 to 50 $\mu g/m^3$ over 4 to 24 hour sampling periods. The detection limit will depend on the nature of the analyte and the length of the sampling period.

1.3 Specific compounds for which the method has been employed are listed in Table 1. The analysis methodology described in this document is currently employed by laboratories using EPA Method 608. The sampling methodology has been formulated to meet the needs of pesticide sampling in indoor air.

2. Applicable Documents

2.1 ASTM Standards

D1356 Definitions of Terms Related to Atmospheric Sampling and Analysis
D1605-60 Standard Recommended Practices for Sampling Atmospheres for Analysis of Gases and Vapors
D4861-88 Standard Practice for Sampling and Analysis of Pesticides and Polychlorinated Biphenyls in Indoor Atmospheres
E260 Recommended Practice for General Gas Chromatography Procedures
E355 Practice for Gas Chromatography Terms and Relationships

2.2 Other Documents

U.S. EPA Technical Assistance Documents (1)
Indoor/Ambient Air Studies (2-9)
Existing Procedures (10-11)

3. Summary of Method

3.1 A low volume (1 to 5 L/min) sampler is used to collect vapors on a sorbent cartridge containing PUF. Airborne particles may also be collected, but the sampling efficiency is not known.

3.2 Pesticides are extracted from the sorbent cartridge with 5% diethyl ether in hexane and determined by gas-liquid chromatography coupled with an electron capture detector (ECD). Note: For some organochlorine pesticides, high performance liquid chromatography (HPLC) coupled with an ultraviolet (UV) detector or electrochemical detector may be preferable. This method describes the use of an electron capture detector.

3.3 Interferences resulting from analytes having similar retention times during gas-liquid chromatography are resolved by improving the resolution or separation, such as by changing

the chromatographic column or operating parameters, or by fractionating the sample by column chromatography.

3.4 The sampling procedure is also applicable to other pesticides which may be determined by gas-liquid chromatography coupled with a nitrogen-phosphorus detector (NPD), flame photometric detector (FPD), Hall electrolytic conductivity detector (HECD), or a mass spectrometer (MS).

4. Significance

4.1 This procedure is intended to be used primarily for non-occupational exposure monitoring in domiciles, public access buildings and offices.

4.2 A broad spectrum of pesticides are commonly used in and around the house and for insect control in public and commercial buildings. Other semi-volatile organic chemicals, such as PCBs, are also often present in indoor air, particularly in large office buildings. This procedure will promote needed accuracy and precision in the determination of many airborne chemicals which may prove to present unacceptable long-term health risks or contribute to short-term episodes, such as "sick building syndrome."

4.3 Use of a portable, low volume PUF sampling system allows the user flexibility in locating the apparatus. The user can place the apparatus in a stationary or mobile location. The portable sampling apparatus may be positioned in a vertical or horizontal stationary location (if necessary, accompanied with supporting structure). Mobile positioning of the system can be accomplished by attaching the apparatus to a person to test air in the individual's breathing zone. Moreover, the PUF cartridge used in this method provides for successful collection of most pesticides. Figure 1(a) illustrates PUF sampling system in a fixed location and Figure 1(b) shows the sampling system attached to an individual.

5. Definitions

Definitions used in this document and in user-prepared Standard Operating Procedures (SOPs) should be consistent with ASTM D1356, D1605-60, and E355. All abbreviations and symbols are defined within this document at point of use. Additional definitions and abbreviations are provided in Appendices A-1 and B-2 of this Compendium.

5.1 Sampling efficiency (SE) - ability of the sampling medium to trap vapors of interest. %SE is the percentage of the analyte of interest collected and retained by the sampling medium when it is introduced as a vapor in air or nitrogen into the air sampler and the sampler is operated under normal conditions for a period of time equal to or greater than that required for the intended use.

5.2 Retention efficiency (RE) - ability of sampling medium to retain a compound added (spiked) to it in liquid solution.

5.2.1 Static retention efficiency - ability of the sampling medium to retain the solution spike when the sampling cartridge is stored under clean, quiescent conditions for the duration of the test period.

5.2.2 Dynamic retention efficiency - ability of the sampling medium to retain the solution spike when air or nitrogen is drawn through the sampling cartridge under normal operating conditions for the duration of the test period. The dynamic RE is normally equal to or less than the SE.

5.3 Retention time (RT) - time to elute a specific chemical from a chromatographic column. For a specific carrier gas flow rate, RT is measured from the time the chemical is injected into the gas stream until it appears at the detector.

5.4 Relative retention time (RRT) - a ratio of RTs for two chemicals for the same chromatographic column and carrier gas flow rate, where the denominator represents a reference chemical.

6. Interferences

6.1 Any gas or liquid chromatographic separation of complex mixtures of organic chemicals is subject to serious interference problems due to coelution of two or more compounds. The use of capillary or narrow bore columns with superior resolution and/or two or more columns of different polarity will frequently eliminate these problems.

6.2 The electron capture detector responds to a wide variety of organic compounds. It is likely that such compounds will be encountered as interferences during GC-ECD analysis. The NPD, FPD, and HECD detectors are element specific, but are still subject to interferences. UV detectors for HPLC are nearly universal, and the electrochemical detector may also respond to a variety of chemicals. Mass spectrometric analyses will generally provide positive identification of specific compounds.

6.3 Certain organochlorine pesticides (e.g., chlordane) are complex mixtures of individual compounds that can make difficult accurate quantification of a particular formulation in a multiple component mixture. Polychlorinated biphenyls (PCBs) may interfere with the determination of pesticides.

6.4 Contamination of glassware and sampling apparatus with traces of pesticides can be a major source of error, particularly at lower analyte concentrations. Careful attention to cleaning and handling procedures is required during all steps of sampling and analysis to minimize this source of error.

6.5 The general approaches listed below should be followed to minimize interferences.

6.5.1 Polar compounds, including certain pesticides (e.g., organophosphorus and carbamate classes), can be removed by column chromatography on alumina. This sample clean-up will permit analysis of most organochlorine pesticides.

6.5.2 PCBs may be separated from other organochlorine pesticides by column chromatography on silicic acid.

6.5.3 Many pesticides can be fractionated into groups by column chromatography on Florisil (Floridin Corp.).

7. Apparatus

7.1 Sample Collection

7.1.1 Sampling pump - (DuPont Alpha-1 Air Sampler, E.I. DuPont de Nemours & Co., Inc., Wilmington, DE, 19898, or equivalent). The pump should be quiet and unobtrusive and provide a constant flow (< ±5%).

7.1.2 Sampling cartridge shown in Figure 2 - constructed from a 20 mm (i.d.) x 10 cm borosilicate glass tube drawn down to a 7 mm (o.d.) open connection for attachment to the pump via vinyl tubing. The cartridge can be fabricated inexpensively from glass by Kontes (P.O. Box 729, Vineland, NJ, 08360), or equivalent.

7.1.3 Sorbent, polyurethane foam (PUF) - cut into a cylinder, 22 mm in diameter and 7.6 cm long, fitted under slight compression inside the cartridge. The PUF should be of the polyether type, density of 0.022 g/cm^3. This type of foam is used for furniture upholstery, pillows, and mattresses; it may be obtained from Olympic Products Co. (Greensboro, NC), or equivalent source. The PUF cylinders (plugs) should be slightly larger in diameter than the internal diameter of the cartridge. They may be cut by one of the following means:

- High-speed cutting tool, such as a motorized cork borer. Distilled water should be used to lubricate the cutting tool.
- Hot wire cutter. Care should be exercised to prevent thermal degradation of the foam.
- Scissors, while plugs are compressed between the 22 mm circular templates.

Alternatively, pre-extracted PUF plugs and glass cartridges may be obtained commercially (Supelco, Inc., Supelco Park, Bellefonte, PA, 16823, No. 2-0557, or equivalent).

7.2 Sample Analysis

7.2.1 Gas chromatograph (GC) with an electron capture detector (ECD) and either an isothermally controlled or temperature programmed heating oven. The analytical system should be complete with all required accessories including syringes, analytical columns, gases, detector, and strip chart recorder. A data system is recommended for measuring peak heights. Consult EPA Method 608 for additional specifications.

7.2.2 Gas Chromatographic Columns

7.2.2.1 The following 4 or 2 mm (i.d.) x 183 cm borosilicate glass GC columns may be used packed with

- 1.5% SP-2250 (Supelco, Inc.)/1.95% SP-2401 (Supelco, Inc.) on 100/120 mesh Supelcoport (Supelco, Inc.)
- 4% SE-30 (General Electric, 50 Fordham Rd., Wilmington, MA, 01887, or equivalent)/6% OV-210 (Ohio Valley Specialty Chemical, 115 Industry Rd., Marietta, OH, 45750, or equivalent) on 100/200 mesh Gas Chrom Q (Alltec Assoc., Applied Science Labs, 2051 Waukegan Rd, Deerfield, IL, 60015, or equivalent)
- 3% OV-101 (Ohio Valley Specialty Chemical) on UltraBond (Ultra Scientific, 1 Main St., Hope, RI, 02831, or equivalent)
- 3% OV-1 (Ohio Valley Specialty Chemical) on 80/100 mesh Chromosorb WHP (Manville, Filtration, and Materials, P.O. Box 5108, Denver, CO, 80271, or equivalent)

7.2.2.2 Capillary GC columns, such as 0.25 mm (i.d.) x 30 m DB-5 (J&W Scientific, 3871 Security Park Dr., Rancho Cordova, CA, 95670, or equivalent) with 0.25 μm film thickness may be used.

7.2.2.3 HPLC columns, such as 4.6 mm x 25 cm Zorbax SIL (DuPont Co., Concord Plaza, Wilmington, DE, 19898, or equivalent) or μ-Bondapak C-18 (Millipore Corp., 80 Ashby Rd., Bedford, MA, 01730, or equivalent) can be used.

7.2.2.4 Other columns may also give acceptable results.

7.2.3. Microsyringes - 5 μL volume or other appropriate sizes.

8. Reagents and Materials

Note: For a detailed listing of various other items required for extract preparation, cleanup, and analysis, consult U.S. EPA Method 608 which is provided in Appendix A of Method TO-4 in the Compendium.

8.1 Round bottom flasks - 500 mL, best source.

8.2 Soxhlet extractors - 300 mL, with reflux condensers, best source.

8.3 Kuderna-Danish concentrator apparatus - 500 mL, with Snyder columns, best source.

8.4 Graduated concentrator tubes - 10 mL, Kontes, P.O. Box 729, Vineland, NJ, 08360, Cat. No. K-570050, size 1025, or equivalent.

8.5 Graduated concentrator tubes - 1 mL, Kontes, Vineland, NJ, Cat. No. K-570050, size 0124, or equivalent.

8.6 TFE fluorocarbon tape - 1/2 in, best source.

8.7 Filter tubes - size 40 mm (i.d.) x 80 mm, Corning Glass Works, Science Products, Houghton Park, AB-1, Corning, NY, 14831, Cat. No. 9480, or equivalent.

8.8 Serum vials - 1 mL and 5 mL, fitted with caps lined with TFE fluorocarbon, best source.

8.9 Pasteur pipettes - 9 in, best source.

8.10 Glass wool - fired at 500°C, best source.

8.11 Boiling chips - fired at 500°C, best source.

8.12 Forceps - stainless steel, 12 in, best source.

8.13 Gloves - latex or polyvinyl acetate, best source.

8.14 Steam bath, best source.

8.15 Heating mantle, - 500 mL, best source.

8.16 Analytical evaporator, nitrogen blow-down (N-Evap®, Organomation Assoc., P.O. Box 159, South Berlin, MA, 01549, or equivalent).

8.17 Acetone - pesticide quality, best source.

8.18 n-Hexane - pesticide quality, best source.

8.19 Diethyl ether preserved with 2% ethanol - Mallinckrodt, Inc., Science Products Division, P.O. Box 5840, St. Louis, MO, 63134, Cat. No. 0850, or equivalent.

8.20 Sodium sulfate - anhydrous, analytical grade, best source.

8.21 Alumina - activity grade IV, 100/200 mesh, best source.

8.22 Glass chromatographic column - 2 mm i.d. x 15 cm long, best source.

8.23 Soxhlet extraction system, including Soxhlet extractors (500 and 300 mL), variable voltage transformers, and cooling water source, best source.

8.24 Vacuum oven connected to water aspirator, best source.

8.25 Die - use to cut PUF adsorbent, best source.

8.26 Ice chest, best source.

8.27 Silicic acid - pesticide quality, best source.

8.28 Octachloronaphthalene (OCN) - research grade, Ultra Scientific, Inc., 1 Main St., Hope, RI, 02831, or equivalent.

9. Assembly and Calibration of Sampling System

9.1 Description of Sampling Apparatus

9.1.1 The entire sampling system is diagrammed in Figure 1. This apparatus was developed to operate at a rate of 1-5 L/minute and is used by U.S. EPA for low volume sampling of indoor air. The method writeup presents the use of this device.

9.1.2 The sampling module in Figure 2 consists of a glass sampling cartridge in which the PUF plug is retained.

9.2 Calibration of Sampling System

9.2.1 Air flow through the sampling system is calibrated by the assembly shown in Figure 3. The air sampler must be calibrated in the laboratory before and after each sample collection period, using the procedure described below.

9.2.2 For accurate calibration, attach the sampling cartridge in-line during calibration. Vinyl bubble tubing (Fisher Scientific, 711 Forbes Ave., Pittsburgh, PA, 15219, Cat. No. 14-170-132, or equivalent) or other means (e.g., rubber stopper or glass joint) may be used to connect the large end of the cartridge to the calibration system. Refer to ASTM Standard Practice D3686, Annex A2 or Standard Practice D4185, Annex A1 for procedures to calibrate small volume air pumps.

10. Preparation of Sampling (PUF) Cartridges

10.1 The PUF adsorbent is white and yellows upon exposure to light. For initial cleanup and quality assurance purposes, the PUF plug is placed in a Soxhlet extractor and extracted with acetone for 14 to 24 hours at 4 to 6 cycles per hour. (If commercially pre-extracted PUF plugs are used, extraction with acetone is not required.) This procedure is followed by a 16 hour Soxhlet extraction with 5% diethyl ether in n-hexane. When cartridges are reused, 5% ether in n-hexane can be used as the cleanup solvent.

10.2 The extracted PUF is placed in a vacuum oven connected to a water aspirator and dried at room temperature for 2 to 4 hours (until no solvent odor is detected). The clean PUF is placed in labeled glass sampling cartridges using gloves and forceps. The cartridges are wrapped with hexane-rinsed aluminum foil and placed in glass jars fitted with TFE fluorocarbon-lined caps. The foil wrapping may also be marked for identification using a blunt probe.

10.3 At least one assembled cartridge from each batch should be analyzed as a laboratory blank before any samples from that batch are considered acceptable for use. A blank level of <10 ng/plug for single component compounds is considered to be acceptable. For multiple component mixtures, the blank level should be <100 ng/plug.

11. Sample Collection

11.1 After the sampling system has been assembled and calibrated as per Section 9, it can be used to collect air samples as described below.

11.2 The prepared sample cartridges should be used within 30 days of loading and should be handled only with clean latex or polyvinyl acetate gloves.

11.3 The clean sample cartridge is carefully removed from the aluminum foil wrapping (the foil is returned to jars for later use) and attached to the pump with flexible tubing. The sampling assembly is positioned with the intake downward or in a horizontal position. The sampler is located in an unobstructed area at least 30 cm from any obstacle to air flow. The PUF cartridge intake is positioned 1 to 2 m above the floor level. Air temperature(s) and barometric pressure(s) are recorded periodically on the Sampling Data Form shown in Figure 4.

11.4 After the PUF cartridge is correctly inserted and positioned, the power switch is turned on and the sampling begins. The elapsed time meter is activated and the start time is recorded. The pumps are checked during the sampling process and any abnormal conditions discovered are recorded on the data sheet.

11.5 At the end of the desired sampling period, the power is turned off and the PUF cartridges are wrapped with the original aluminum foil and placed in sealed, labeled containers for transport back to the laboratory. At least one field blank is returned to the laboratory with each group of samples. A field blank is treated exactly like a sample except

that no air is drawn through the cartridge. Samples are stored at -10°C or below until analyzed.

12. Sample Preparation, Cleanup, and Analysis

Note: Sample preparation should be performed under a properly ventilated hood.

12.1 Sample Preparation

12.1.1 All samples should be extracted within 1 week after collection.

12.1.2 All glassware is washed with a suitable detergent; rinsed with deionized water, acetone, and hexane; rinsed again with deionized water; and fired in an oven (500°C).

12.1.3 Sample extraction efficiency is determined by spiking the samples with a known solution. Octachloronaphthalene (OCN) is an appropriate standard to use for pesticide analysis using GC-ECD techniques. The spiking solution is prepared by dissolving 10 mg of OCN in 10 mL of 10% acetone in n-hexane, followed by serial dilution with n-hexane to achieve a final concentration of 1 µg/mL.

12.1.4 The extracting solution (5% ether/hexane) is prepared by mixing 1900 mL of freshly opened hexane and 100 mL of freshly opened ethyl ether (preserved with ethanol) to a flask.

12.1.5 All clean glassware, forceps, and other equipment to be used are placed on rinsed (5% ether/hexane) aluminum foil until use. The forceps are also rinsed with 5% ether/hexane. The condensing towers are rinsed with 5% ether/hexane and 300 mL are added to a 500 mL round bottom boiling flask (with no more than three boiling chips).

12.1.6 Using clean gloves, the PUF cartridges are removed from the sealed container and the PUF is placed into a 300 mL Soxhlet extractor using prerinsed forceps.

12.1.7 Before extraction begins, 100 µL of the OCN solution are added dropwise to the top of the PUF plug. Addition of the standard demonstrates extraction efficiency of the Soxhlet procedure.

Note: Incorporating a known concentration of the solution onto the sample provides a quality assurance check to determine recovery efficiency of the extraction and analytical processes.

12.1.8 The Soxhlet extractor is then connected to the 500 mL boiling flask and condenser. The glass joints of the assembly are wet with 5% ether/hexane to ensure a tight seal between the fittings. If necessary, the PUF plug can be adjusted using forceps to wedge it midway along the length of the siphon. The above procedure should be followed for all samples, with the inclusion of a blank control sample.

12.1.9 The water flow to the condenser towers of the Soxhlet extraction assembly is checked and the heating unit is turned on. As the samples boil, the Soxhlet extractors are inspected to ensure that they are filling and siphoning properly (4 to 6 cycles/hour). Samples should cycle for a minimum of 16 hours.

12.1.10 At the end of the extracting process, the heating units are turned off and the samples are cooled to room temperature.

12.1.11 The extracts are concentrated to a 5 mL solution using a Kuderna-Danish (K-D) apparatus. The K-D is set up and assembled with concentrator tubes. This assembly

is rinsed and one boiling chip is added to each concentrator tube. The lower end of the filter tube is packed with glass wool and filled with anhydrous sodium sulfate to a depth of 40 mm. The filter tube is placed in the neck of the K-D. The Soxhlet extractors and boiling flasks are carefully removed from the condenser towers and the remaining solvent is drained into each boiling flask. Sample extract is carefully poured through the filter tube into the K-D. Each boiling flask is rinsed three times by swirling hexane along the sides. Once the sample has drained, the filter tube is rinsed down with hexane. Each Synder column is attached to the K-D and rinsed to wet the joint for a tight seal. The complete K-D apparatus is placed on a steam bath and the sample is evaporated to approximately 5 mL. Do not let sample go to dryness. The sample is removed from the steam bath and allowed to cool. Each Synder column is rinsed with a minimum of hexane and sample is allowed to cool. Sample volume is adjusted to 10 mL in a concentrator tube, which is then closed with a glass stopper and sealed with TFE fluorocarbon tape. Alternately, the sample may be quantitatively transferred (with concentrator tube rinsing) to prescored vials and brought up to final volume. Concentrated extracts are stored at -10°C until analyzed. Analysis should occur no later than two weeks after sample extraction.

12.2 Sample Cleanup

12.2.1 If only organochlorine pesticides are sought, an alumina cleanup procedure is appropriate. Before cleanup, the sample extract is carefully reduced to 1 mL using a gentle stream of clean nitrogen.

12.2.2 A glass chromatographic column (2 mm i.d. x 15 cm long) is packed with alumina, activity grade IV, and rinsed with approximately 20 mL of n-hexane. The concentrated sample extract is placed on the column and eluted with 10 mL of n-hexane at a rate of 0.5 mL/minute. The eluate volume is adjusted to exactly 10 mL and analyzed as per Section 12.3.

12.2.3 If other pesticides are sought, alternate cleanup procedures may be required (e.g., Florisil). EPA Method 608 identifies appropriate cleanup procedures.

12.3 Sample Analysis

12.3.1 Organochlorine pesticides and many nonchlorinated pesticides are responsive to electron capture detection (Table 1). Most of these compounds can be determined at concentrations of 1 to 50 ng/mL by GC-ECD.

12.3.2 An appropriate GC column is selected for analysis of the extract. (For example, 4 mm i.d. x 183 cm glass, packed with 1.5% SP-2250/1.95% SP-2401 on 100/120 mesh Supelcoport, 200°C isothermal, with 5% methane/95% argon carrier gas at 65 to 85 mL/min). A chromatogram showing a mixture containing single component pesticides determined by GC-ECD using a packed column is shown in Figure 5. Corresponding chromatographic characteristics are shown in Table 2.

12.3.3 A standard solution is prepared from reference materials of known purity. Standards of organochlorine pesticides may be obtained from the National Bureau of Standards and from the U.S. EPA.

12.3.4 Stock standard solutions (1.00 µg/µL) are prepared by dissolving approximately 10 milligrams of pure material in isooctane and diluting to volume in a 10 mL volumetric flask. Larger volumes can be used at the convenience of the analyst. If compound purity is certified at 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or an independent source.

12.3.5 The prepared stock standard solutions are transferred to Teflon-sealed screw-capped bottles and stored at -10°C for no longer than six months. The standard solutions should be inspected frequently for signs of degradation or evaporation (especially before preparing calibration standards from them).

Note: Quality control check standards used to determine accuracy of the calibration standards are available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268.

12.3.6 The standard solutions of the various compounds of interest are used to determine relative retention times (RRTs) to an internal standard such as p,p'-DDE, aldrin, or octachloronaphthalens (OCN).

12.3.7 Before analysis, the GC column is made sensitive to the pesticide samples by injecting a standard pesticide solution ten (10) times more concentrated than the stock standard solution. Detector linearity is then determined by injecting standard solutions of three different concentrations that bracket the required range of analyses.

12.3.8 The GC system is calibrated daily with a minimum of three injections of calibrated standards. Consult EPA Method 608, Section 7 for a detailed procedure to calibrate the gas chromatograph.

12.3.9 If refrigerated, the sample extract is removed from the cooling unit and allowed to warm to room temperature. The sample extract is injected into the GC for analysis in an aliquot of approximately 2-6 µL using the solvent flush technique (Ref. ASTM D3687, Section 8.1.4.3-8.1.4.5). The actual volume injected is recorded to the nearest 0.05 µL. After GC injection, the sample's response from the strip chart is analyzed by measuring peak heights or determining peak areas. Ideally, the peak heights should be 20 to 80% of full scale deflection. Using injections of 2 to 6 µL of each calibration standard, the peak height or area responses are tabulated against the mass injected (injections of 2, 4, and 6 µL are recommended). If the response (peak height or area) exceeds the linear range of detection, the extract is diluted and reanalyzed.

12.3.10 Pesticide mixtures are quantified by comparison of the total heights or areas of GC peaks with the corresponding peaks in the best-matching standard. If both PCBs and organochlorine pesticides are present in the same sample, column chromatographic separation on silicic acid is used before GC analysis, according to ASTM Standards, Vol. 14.01. If polar compounds that interfere with GC-ECD analysis are present, column chromatographic cleanup on alumina (activity grade IV) is used as per Section 12.2.2.

12.3.11 For confirmation, a second GC column is used such as 4% SE-30/6% OV-210 on 100/200 mesh Gas Chrom Q or 3% OV-1 on 80/100 mesh Chromosorb WHP. For improved resolution, a capillary column is used such as 0.25 mm (i.d.) x 30 m DB-5 with 0.25 µm film thickness.

12.3.12 A chromatogram of a mixture containing single component pesticides determined by GC-ECD using a capillary column is shown in Figure 6. A table of the corresponding chromatographic characteristics follows in Table 3.

12.3.13 Class separation and improved specificity can be achieved by column chromatographic separation on Florisil as per EPA Method 608. For improved specificity, a Hall electrolytic conductivity detector operated in the reductive mode may be substituted for the electron capture detector. Limits of detection will be reduced by at least an order of magnitude.

13. GC Calibration

Appropriate calibration procedures are identified in EPA Method 608, Section 7 (11).

13.1 Establish gas chromatographic operating parameters. The gas chromatographic system may be calibrated using the external standard technique (Section 13.2) or the internal standard technique (Section 13.3).

13.2 External Standard Calibration Procedure

13.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with isooctane. One of the external standards should be at a concentration near, but above, the method detection limit and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

13.2.2 Using injections of 2 to 5 μL of each calibration standard, tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

13.2.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that compound.

13.3 Internal Standard Calibration Procedure

13.2.1 To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

13.3.2 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a

volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

13.3.3 Using injections of 2 to 5 µL of each calibration standard, tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using

$$RF = (A_s C_{is})/(A_{is} C_s)$$

where:
A_s = response for the parameter to be measured
A_{is} = response for the internal standard
C_{is} = concentration of the internal standard, µg/L
C_s = concentration of the parameter to be measured, µg/L

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

13.3.4 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

14. Calculations

14.1 The concentration of the analyte in the extract solution is taken from a standard curve where peak height or area is plotted linearly against concentration in nanograms per milliliter (ng/mL). If the detector response is known to be linear, a single point is used as a calculation constant.

14.2 From the standard curve, determine the ng of analyte standard equivalent to the peak height or area for a particular compound.

14.3 Determine if the field blank is contaminated. Blank levels should not exceed 10 ng/sample for organochlorine pesticides or 100 ng/sample for other pesticides. If the blank has been contaminated, the sampling series must be held suspect.

14.4 Quantity of the compound in the sample (A) is calculated using the following equation:

$$A = 1000 \bullet [(A_s \times V_e)/V_i]$$

where:
A = total amount of analyte in the sample, ng

A_s = calculated amount of material injected onto the chromatograph based on calibration curve for injected standards, ng
V_e = final volume of extract, mL
V_i = volume of extract injected, µL
1000 = factor for converting microliters to milliliters

14.5 The extraction efficiency (EE) is determined from the recovery of octachloronaphthalene (OCN) spike as follows:

$$EE(\%) = (S/S_a) \times 100$$

where:
S = amount of spike recovered, ng
S_a = amount of spike added to plug, ng

14.6 The total amount of nanograms found in the sample is corrected for extraction efficiency and laboratory blank as follows:

$$A_c = (A - A_o)/EE(\%)$$

where:
A_c = corrected amount of analyte in sample, ng
A_o = amount of analyte in blank, ng

14.7 The total volume of air sampled under ambient conditions is determined using the following equation:

$$V_a = [\sum_{i=1}^{n} (T_i \times F_i)]/1000$$

where:
V_a = total volume of air sampled, m^3
T_i = length of sampling segment between flow checks, min
F_i = average flow during sampling segment, L/min
1000 = factor for converting liters to cubic meters

14.8 The air volume is corrected to 25°C and 760 mm Hg (STP) as follows:

$$V_s = V_a \bullet [(P_b - P_w)/760 \text{ mm Hg}] \bullet [298/(237 + TA)]$$

where:
V_s = volume of air at standard conditions, m^3
V_a = total volume of air sampled, m^3
P_b = average ambient barometric pressure, mm Hg
P_w = vapor pressure of water at calibration temperature, mm Hg
T_A = average ambient temperature, °C

14.9 If the proper criteria for a sample have been met, concentration of the compound in a cubic meter of air is calculated as follows:

$$ng/m^3 = A_c/V_s \bullet 100/SE(\%)$$

where:
SE = sampling efficiency as determined by the procedure outlined in Section 15

If it is desired to convert the air concentration value to parts per trillion (wt/wt) in dry air at STP, the following conversion is used:

$$ppt = 0.844\ (ng/m^3)$$

The air concentration is converted to parts per trillion (v/v) in air at STP as follows:

$$pptv = 24.45\ (ng/m^3)/MW$$

where:
MW = molecular weight of the compound of interest

15. Sampling and Retention Efficiencies

15.1 Before using this procedure, the user should determine the sampling efficiency for the compound of interest. The sampling efficiencies shown in Tables 4 and 5 were determined for approximately 1 m^3 of air at about 25°C, sampled at 3.8 L/min. Sampling efficiencies for the pesticides shown in Table 6 are for 24 hours at 3.8 L/min and 25°C. Sampling efficiencies for carbonates, ureas, triazines, and pyrethrine are provided in Table 7. For compounds not listed, longer sampling times, different flow rates, or other air temperatures, the following procedure may be used to determine sampling efficiencies.

15.2 SE is determined by a modified impinger assembly attached to the sampler pump (see Figure 7). Clean PUF is placed in the pre-filter location and the inlet is attached to a nitrogen line. PUF plugs (22 mm x 7.6 cm) are placed in the primary and secondary traps and are attached to the pump.
Note: Nitrogen should be used instead of air to prevent oxidation of the compounds under test. The oxidation would not necessarily reflect what may be encountered during actual sampling and may give misleading sampling efficiencies.

15.3 A standard solution of the compound of interest is prepared in a volatile solvent (e.g., hexane, pentane, or benzene). A small, accurately measured volume (e.g., 1 mL) of the standard solution is placed into the modified midget impinger. The sampler pump is set at the rate to be used in sampling application and then activated. Nitrogen is drawn through the assembly for a period of time equal to or exceeding that intended for sampling application. After the desired sampling test period, the PUF plugs are removed and analyzed separately as per Section 12.3.

15.4 The impinger is rinsed with hexane or another suitable solvent and quantitatively transferred to a volumetric flask or concentrator tube for analysis.

15.5 The sampling efficiency (SE) is determined using the following equation:

$$\%\ SE = W_1/(W_o - W_r) \bullet 100$$

where:
W_1 = amount of compound extracted from the primary trap, ng
W_o = original amount of compound added to the impinger, ng
W_r = residue left in the impinger at the end of the test, ng

15.6 If material is found in the secondary trap, it is an indication that breakthrough has occurred. The addition of the amount found in the secondary trap, W_2, to W_1, will provide an indication of the overall sampling efficiency of a tandem-trap sampling system. The sum of W_1, W_2 (if any), and W_r must equal (approximately ± 10%) W_o or the test is invalid.

15.7 If the compound of interest is not sufficiently volatile to vaporize at room temperature, the impinger may be heated in a water bath or other suitable heater to a maximum of 50°C to aid volatilization. If the compound of interest cannot be vaporized at 50°C without thermal degradation, dynamic retention efficiency (RE_d) may be used to estimate sampling efficiency. Dynamic retention efficiency is determined in the manner described in 15.8. Table 6 lists those organochlorine pesticides for which dynamic retention efficiencies have been determined.

15.8 A pair of PUF plugs is spiked by slow, dropwise addition of the standard solution to one end of each plug. No more than 0.5 to 1 mL of solution should be used. Amounts added to each plug should be as nearly the same as possible. The plugs are allowed to dry for 2 hours in a clean, protected place (e.g., desiccator). One spiked plug is placed in the primary trap so that the spiked end is at the intake and one clean unspiked plug is placed in the secondary trap. The other spiked plug is wrapped in hexane-rinsed aluminum foil and stored in a clean place for the duration of the test (this is the static control plug, Section 15.9). Prefiltered nitrogen or ambient air is drawn through the assembly as per Section 15.3. Each PUF plug (spiked and static control) is analyzed separately as per Section 12.3.
Note: Impinger may be discarded.

15.9 Retention Efficiency (RE) is calculated as follows:

$$\% \text{ RE} = (W_1/W_0) \bullet 100$$

where:
W_1 = amount of compound recovered from primary plug, ng
W_o = amount of compound added to primary plug, ng

If a residue, W_2, is found on the secondary plug, breakthrough has occurred. The sum of $W_1 + W_2$ must equal W_o within 25% or the test is invalid. For most compounds tested by this procedure, % RE values are generally less than % SE values determined per Section 15.1. The purpose of the static RE determination is to establish any loss or gain of analyte unrelated to the flow of nitrogen or air through the PUF plug (see Table 8).

16. Method Variation

This section provides analytical procedures for a variety of pesticides other than organochlorine. Samples for the pesticides mentioned below are collected as described in Section 7.1.

16.1 Organophosphorus pesticides are responsive to flame photometric and nitrogen-phosphorus (alkali flame ionization) detection. Most of these compounds can be analyzed at concentrations of 50 to 500 ng/mL using either of these detectors. Procedures given in 12.3.2 through 12.3.9 and 12.3.11 through 12.3.3 apply, except for the selection of internal standards. Use parathion as an internal standard.

16.2 Carbamate and triazine pesticides are most commonly analyzed by HPLC because of poor thermal stability or high polarity. Detection limits will be in the 1 to 5 µg/mL range. Many carbamates and triazine pesticides may also be analyzed intact by GC on a 2 mm (i.d.) x 183 cm glass column of 3% OV-101 on Ultra-Bond and determined by HECD. Detection limits will be about 1 µg/mL.

16.3 Carbaryl®, atrazine®, propoxur®, bendiocarb® and captan® have been successfully analyzed by capillary column chromatography as discussed in Section 12.3.11.

16.4 Many urea pesticides, pyrethrins, phenols, and other polar pesticides may be analyzed by HPLC with fixed or variable wavelength UV detection. Either reversed-phase or normal phase chromatography may be used. Detection limits are 0.2 to 10 µg/mL of extract. An acceptable procedure follows: Select HPLC column (for example, Zorbax-SIL, 4.6 mm i.d. x 25 cm, or u-Bondapak C18, 3.9 mm x 30 cm, or equivalent). Select solvent system (for example, mixtures of methanol or acetonitrile with water or mixtures of heptane or hexane with isopropanol). Follow analytical procedures given in 12.3.2 through 12.3.9. If interferences are present, adjust the HPLC solvent system composition or use column chromatographic clean-up with silica gel, alumina or Florisil. An electrochemical detector may be used to improve sensitivity for some ureas, carbamates and phenolics. Much more care is required in using this detector, particularly in removing dissolved oxygen from the mobile phase and sample extracts. Chlorophenols have been successfully analyzed intact by GC on a 4 mm (i.d.) x 60 cm glass column packed with double support-bonded diethylene glycol succinate (DEGS).

16.5 Mass spectrometric analyses may be used for more unambiguous confirmation of pesticides. Essentially all pesticides may be determined by GC-MS or HPLC-MS.

16.5.1 Many of the pesticides shown in Table 1 have been successfully analyzed by GC-MS by the following procedure:

16.5.1.1 GC column carrier gas and flow rate as described in 12.3.2.

16.5.1.2 Temperature program, 40°C (2 min) to 295°C (10°C per min).

16.5.1.3 Splitless injection, 2 µL maximum volume (injection time 30 to 40 sec); injector temperature, 205°C.

16.5.1.4 Interface temperature, 240°C.

16.5.1.5 Mass spectrometer, quadrupole, electron ionization, multiple ion detection mode.

16.5.1.6 Internal standards, D_{10}-phenanthrene and D_{12}chrysene.

16.6 See ASTM Standard Practice D3687 for solvent-flush injection technique, determination of relative retention times, and other procedures pertinent to GC and HPLC analyses.

16.7 If concentrations are too low to detect by the analytical procedure of choice, the extract may be concentrated to 1 mL or 0.5 mL by carefully controlled evaporation under an inert atmosphere. The following procedure is appropriate:

16.7.1 Place K-D concentrator tube in a water bath and analytical evaporator (nitrogen blow-down) apparatus. The water bath temperature should be 25°C to 50°C.

16.7.2 Adjust nitrogen flow through hypodermic needle to provide a gentle stream.

16.7.3 Carefully lower hypodermic needle into the concentrator tube to a distance of about 1 cm above the liquid level.

16.7.4 Continue to adjust needle placement as liquid level decreases.

16.7.5 Reduce volume to slightly below desired level.

16.7.6 Adjust to final volume by carefully rinsing needle tip and concentrator tube well with solvent (usually n-hexane).

17. Performance Criteria and Quality Assurance

This section summarizes required quality assurance (QA) measures and provides guidance concerning performance criteria that should be achieved within each laboratory.

17.1 Standard Operating Procedures (SOPs)

17.1.1 Users should generate SOPs describing the following activities accomplished in their laboratory:

- assembly, calibration, and operation of the sampling system, with make and model of equipment used
- preparation, purification, storage, and handling of sampling cartridges
- assembly, calibration, and operation of the GC-ECD system, with make and model of equipment used
- all aspects of data recording and processing, including lists of computer hardware and software used

17.1.2 SOPs should provide specific stepwise instructions and should be readily available to, and understood by, the laboratory personnel conducting the work.

17.2 Process, Field, and Solvent Blanks

17.2.1 One PUF cartridge from each batch of approximately twenty should be analyzed, without shipment to the field, for the compounds of interest to serve as a process blank.

17.2.2 During each sampling episode, at least one PUF cartridge should be shipped to the field and returned, without drawing air through the sampler, to serve as a field blank.

17.2.3 Before each sampling episode, one PUF plug from each batch of approximately twenty should be spiked with a known amount of the standard solution. The spiked plug will remain in a sealed container and will not be used during the sampling period. The spiked plug is extracted and analyzed with the other samples. This field spike acts as a quality assurance check to determine matrix spike recoveries and to indicate sample degradation.

17.2.4 During the analysis of each batch of samples, at least one solvent process blank (all steps conducted but no PUF cartridge included) should be carried through the procedure and analyzed.

17.2.5 Blank levels should not exceed 10 ng/sample for single components or 100 ng/sample for multiple component mixtures (e.g., for organochlorine pesticides).

17.3 Sampling Efficiency and Spike Recovery

17.3.1 Before using the method for sample analysis, each laboratory must determine its sampling efficiency for the component of interest as per Section 15.

17.3.2 The PUF in the sampler is replaced with a hexane-extracted PUF. The PUF is spiked with a microgram level of compounds of interest by dropwise addition of hexane solutions of the compounds. The solvent is allowed to evaporate.

17.3.3 The sampling system is activated and set at the desired sampling flow rate. The sample flow is monitored for 24 hours.

17.3.4 The PUF cartridge is then removed and analyzed as per Section 12.3.

17.3.5 A second sample, unspiked, is collected over the same time period to account for any background levels of components in the ambient air matrix.

17.3.6 In general, analytical recoveries and collection efficiencies of 75% are considered to be acceptable method performance.

17.3.7 Replicate (at least triplicate) determinations of collection efficiency should be made. Relative standard deviations for these replicate determinations of ±15% or less are considered acceptable performance.

17.3.8 Blind spiked samples should be included with sample sets periodically as a check on analytical performance.

17.4 Method Sensitivity

Several different parameters involved in both the sampling and analysis steps of this method collectively determine the sensitivity with which each compound is detected. As the volume of air sampled is increased, the sensitivity of detection increases proportionately within limits set by the retention efficiency for each specific component trapped on the polyurethane foam plug and the background interference associated with the analysis of each specific component at a given site sampled. The sensitivity of detection of samples recovered by extraction depends on the inherent response of the particular GC detector used in the determinative step and the extent to which the sample is concentrated for analysis. It is the responsibility of the analyst(s) performing the sampling and analysis steps to adjust parameters so that the required detection limits can be obtained.

17.5 Method Precision and Bias

17.5.1 Precision and bias in this type of analytical procedure are dependent upon the precision and bias of the analytical procedure for each compound of concern, and the precision and bias of the sampling process.

17.5.2 The reproducibility of this method has been determined to range from 5 to 30% (measured as the relative standard deviation) when replicate sampling cartridges are used (N>5). Sample recoveries for individual compounds generally fall within the range of 90 to 110%, but recoveries ranging from 65 to 125% are considered acceptable. PUF alone may give lower recoveries for more volatile compounds (e.g., those with saturation vapor pressures $>10^{-3}$ mm Hg). In those cases, another sorbent or a combination of PUF and Tenax GC should be employed.

17.6 Method Safety

This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the user's responsibility to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

18. References

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Table 1. Pesticides Determined by Gas Chromatography/Electron Capture Detector (GC-ECD)

Aldrin
BHC (α-and β-Hexachlorocyclohexanes)
Captan
Chlordane, technical
Chlorothalonil
Chlorpyrifos
2,4,-D esters
p,p,-DDT
p,p,-DDE
Dieldrin
Dichlorvos (DDVP)
Dicofol
2,4,5-Trichlorophenol
Folpet
Heptachlor
Heptachlor epoxide
Hexachlorobenzene
Lindane (γ-BHC)
Methoxychlor
Mexacarbate
Mirex
trans-Nonachlor
Oxychlordane
Pentachlorobenzene
Pentachlorophenol
Ronnel

Table 2. Chromatographic Characteristics of the Single Component Pesticide Mixture (5 µl) Determined by GC-ECD Using a Packed Column

Retention Time	Compound Name	Concentration in pg on Column	Area/ Height
2.77	gamma-BHC (Lindane)	500	8.2
3.37	Heptachlor	500	10.4
4.03	Aldrin	500	12.0
8.90	Dieldrin	500	24.7
14.63	p,p'-DDT	500	39.0
24.87	Dibutylchlorendate*	2500	61.4
26.82	Methoxychlor	2500	57.5

* Internal standard used for earlier pesticide detection.

Table 3. Chromatographic Characteristics of the Single Component Pesticide Mixture (2 µl) Determined by GC-ECD Using a Capillary Column

Retention Time	Compound Name	Concentration in pg on Column	Area/ Height
14.28	gamma-BHC (Lindane)	200	5.2
17.41	Heptachlor	200	5.3
18.96	Aldrin	200	5.4
23.63	Dieldrin	200	5.8
27.24	p,p'-DDT	200	5.6
29.92	Methoxychlor	1000	5.5
31.49	Dibutylchlorendate*	1000	5.4

* Internal standard used for earlier pesticide detection.

Table 4. Sampling Efficiencies for Some Organochlorine Pesticides

Compound	Quantity Introduced, µg	Air Volume, m^3	Sampling Efficiency, % mean	RSD	n
α-Hexachlorocyclohexane (α-BHC)	0.005	0.9	115	8	6
β-Hexachlorocyclohexane (Lindane)	0.05-1.0	0.9	91.5	8	5
Hexachlorobenzene**	0.5, 1.0	0.9	94.5	8	5
Chlordane, technical	0.2	0.9	84.0	11	8
p,p'-DDT	0.6, 1.2	0.9	97.5	21	12
p,p'-DDE	0.2, 0.4	0.9	102	11	12
Mirex	0.6, 1.2	0.9	85.9	22	7
Pentachlorobenzene**	1.0	0.9	94	12	5
Pentachlorophenol**	1.0	0.9	107	16	5
2,4,5-Trichlorophenol**	1.0	0.9	108	3	5
2,4-D Esters:					
isopropyl	0.5	3.6	92.0	5	12
butyl	0.5	3.6	82.0	10	11
isobutyl	0.5	3.6	79.0	20	12
isooctyl	0.5	3.6	>80*	--	--

* Not vaporized. Value based on %RE = 81.0 (RSD = 10%, n = 6).
** Semivolatile organochlorine pesticides.

Table 5. Sampling Efficiencies for Organophosphorus Pesticides

Compound	Quantity Introduced,[b] µg	Sampling Efficiency, % mean	RSD	n
Dichlorvos (DDVP)	0.2	72.0	13	2
Ronnel	0.2	106		
Chlorpyrifos	0.2	108	9	12
Diazinon[a]	1.0	84.0	18	
Methyl parathion[a]	0.6	80.0	19	18
Ethyl parathion[a]	0.3	75.9	15	18
Malathion[a]	0.3	100[c]	--	--

[a] Analyzed by gas chromatography with nitrogen phosphorus detector or flame photometric detector.
[b] Air volume = 0.9 m^3.
[c] Decomposed in generator; value based on %RE = 101 (RDS = 7, n = 4).

Table 6. Extraction and 24-hour Sampling Efficiencies for Various Pesticides and Related Compounds

Compound	Extraction Efficiency, %* mean	RSD	Sampling Efficiency**, %, at: 10 ng/m³ mean	RSD	100 ng/m³ mean	RSD	1000 ng/m³ mean	RSD
Chlorpyrifos	83.3	11.5	83.7	18.0	92.7	15.1	83.7	18.0
Pentachloro-phenol	84.0	22.6	66.7	42.2	52.3	36.2	66.7	42.2
Chlordane	95.0	7.1	96.0	1.4	74.0	8.5	96.0	1.4
Lindane	96.0	6.9	91.7	11.6	93.0	2.6	91.7	11.6
DDVP	88.3	20.2	51.0	53.7	106.0	1.4	51.0	53.7
2,4-D methyl ester	--	--	75.3	6.8	58.0	23.6	75.3	6.8
Heptachlor	99.0	1.7	97.3	13.6	103.0	17.3	97.3	13.6
Aldrin	97.7	4.0	90.7	5.5	94.0	2.6	90.7	5.5
Dieldrin	95.0	7.0	82.7	7.6	85.0	11.5	82.7	7.6
Ronnel	80.3	19.5	74.7	12.1	60.7	15.5	74.7	12.2
Diazinon	72.0	21.8	63.7	18.9	41.3	26.6	63.7	19.9
trans-Nonachlor	97.7	4.0	96.7	4.2	101.7	15.3	96.7	4.2
Oxychlordane	100.0	0.0	95.3	9.5	94.3	1.2	95.3	9.5
α-BHC	98.0	3.5	86.7	13.7	97.0	18.2	86.7	13.7
Chlorothalonil	90.3	8.4	76.7	6.1	70.3	6.5	76.7	6.1
Heptachlor epoxide	100.0	0.0	95.3	5.5	97.7	14.2	95.3	5.5

* Mean values for one spike at 550 ng/plug and two spikes at 5500 ng/plug.
** Mean values for three determinations.

Table 7. Sampling Efficiencies for Carbamates, Ureas, Triazines, and Pyrethrins

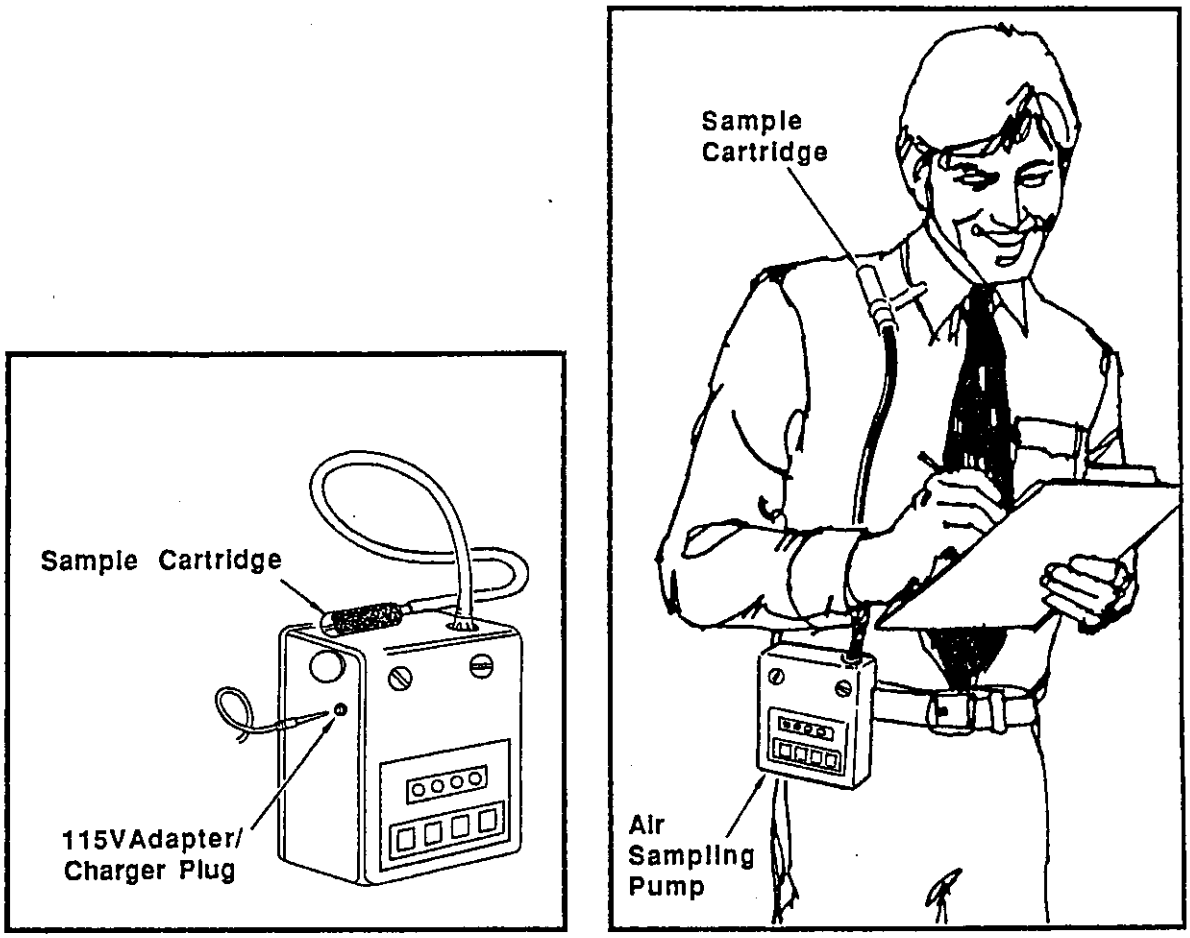
Compound	Spike Level,[a] µg/plug	Static Recovery,% mean	RSD	n	Retention Efficiency,% mean	RSD	n	Sampling Efficiency,% mean	RSD	n
Carbamates:										
Propoxur	5	61.4	10	6	77.6	37	6	96.7	11	6
Carbofuran	15	55.3	12	6	64.2	46	6	87.2	14	6
Bendicarb	50	57.3	11	6	69.8	43	6	62.1	14	6
Mexacarbate	10	62.8	19	6	62.7	41	6	89.8	14	6
Carbaryl	100	56.6	14	6	63.6	53	6	b	13	6
Ureas:										
Monuron	19	87.0		6	91.2	6	5	c		
Diuron	20	84.1	8	6	90.0	2	5	c		
Linuron	20	86.7	8	6	92.5	4	5	c		
Terbuthiuron	18	85.0	8	6	88.8	8	5	c		
Fluometuron	20	91.4	10	6	101	3	5	c		
Chlortoluron	20	86.2	11	6	92.0	7	5	c		
Triazines:										
Simazine	10	103	6	5	101	9	6	c		
Atrazine	10	104	7	5	98.9	7	6	c		
Propazine	10	105	11	5	99.9	14	6	c		
Pyrethrins:										
PyrethrinI	(9.7)[d]	90.5	10	6	95.6	22	5	c		
PyrethrinII	(6.1)[d]	88.6	11	6	69.9	29	5	c		
Allethrin	25	69.2	9	5	58.3	12	6	c		
d-trans-Allethrin	25	76.8	9	6	74.4	9	5	c		
Dicrotophos	25	72.0	22	6	71.7	8	5	c		
Resmethrin	25	76.5	14	6	66.7	14	6	c		
Fenvalerate	25	87.9	3	6	57.2	20	3	c		

[a]Air volume = 0.9 m^3.
[b]Decomposed in generator.
[c]Not vaporized.
[d]Estimated on the basis of 20 µg Pyrethrin with a composition of 48.4% and 30.3% by weight of Pyrethrins I and II, respectively.

Table 8. Extraction and 24-Hour Sampling Efficiencies for Various Pesticides and Related Compounds

Compound	Extraction Efficiency*, % mean	RSD	Retention Efficiency**, %, at: 10 ng/m^3 mean	RSD	100 ng/m^3 mean	RSD	1000 ng/m^3 mean	RSD
Dicofol	57.0	8.5	38.0	25.9	65.0	8.7	69.0	--
Captan	73.0	12.7	56.0	--	45.5	64.3	84.3	16.3
Methoxlychlor	65.5	4.9	--	--	--	--	78.5	2.1
Folpet	86.7	11.7	--	--	78.0	--	93.0	--

* Mean values for one spike at 550 ng/plug and two spikes at 5500 ng/plug.
** Mean values for generally three determinations.



(a) Fixed Site Monitoring (b) Personal Monitoring

Figure 1. Sampling for Pesticides

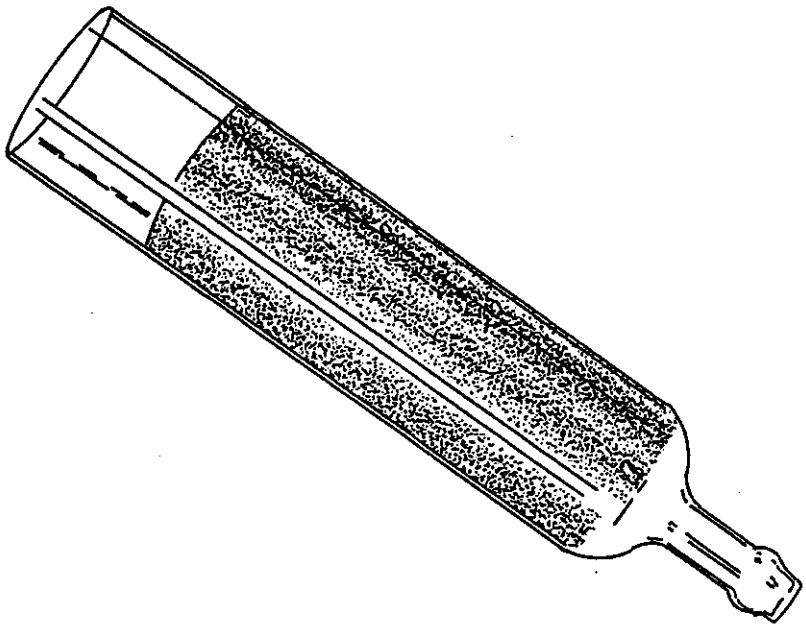


Figure 2. Polyurethane Foam (PUF) Sampling Cartridge

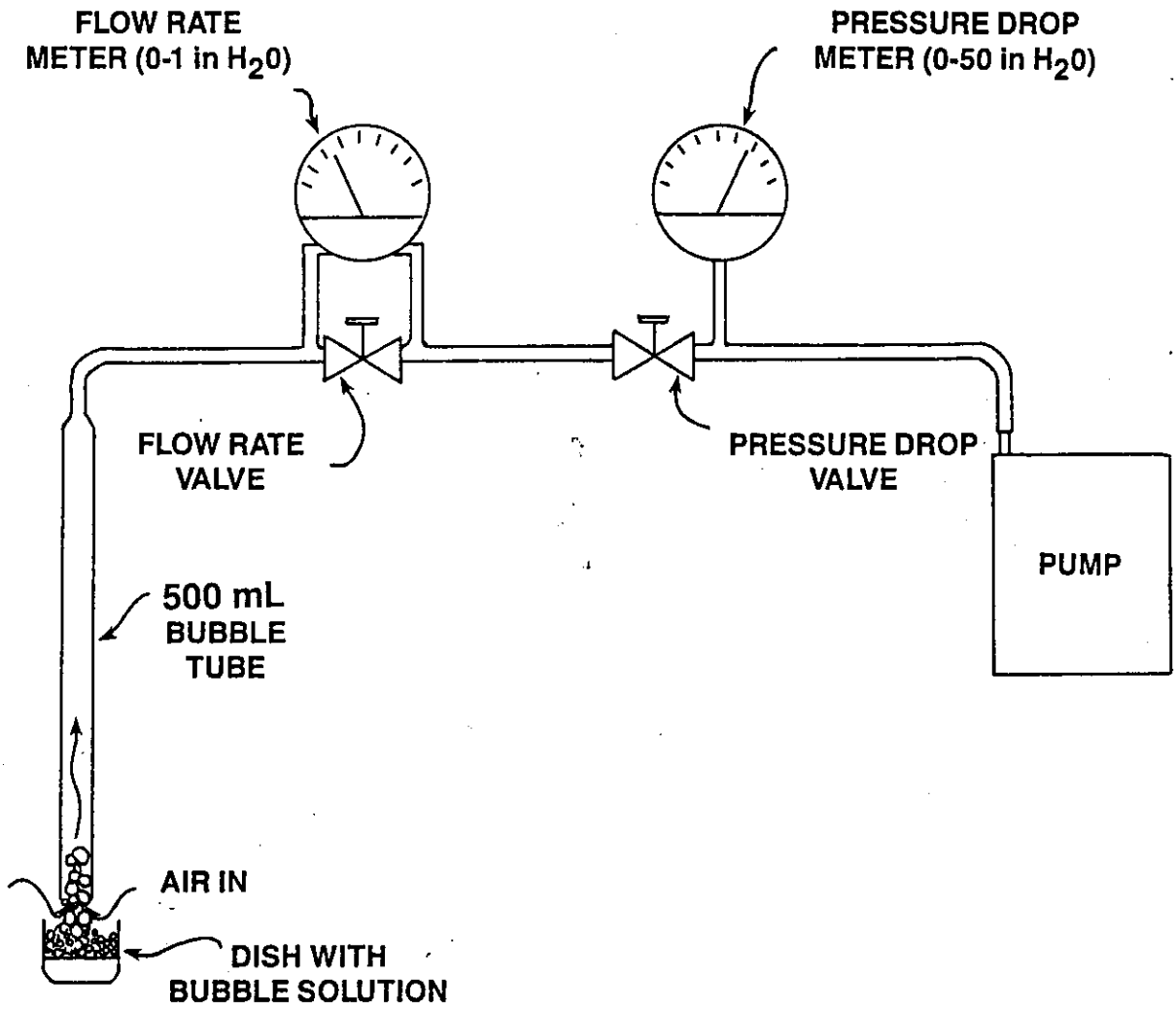


Figure 3. Calibration Assembly for Air Sampler Pump

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Site Date Performed by

Sampler S/N	Sampling Location I.D	Height Above Ground	PUF Cart. No.	Sampling Period Start	Sampling Period Stop	Sampling Time min.	Pump Timer hr. min.	Low flow Indication Yes	Low flow Indication No	Comments

Checked by_____

Date_____

Figure 4. Low Volume Pesticide Sampling Data Form

OPERATING CONDITIONS

Column Type:	1.5% SP 2250/1.95% SP 2401, ¼" glass.
Temperature:	200°C isothermal.
Detector:	Electron Capture.
Carrier Gas:	5% Methane/95% Argon.
Flow Rate:	65 to 85 mL/min.

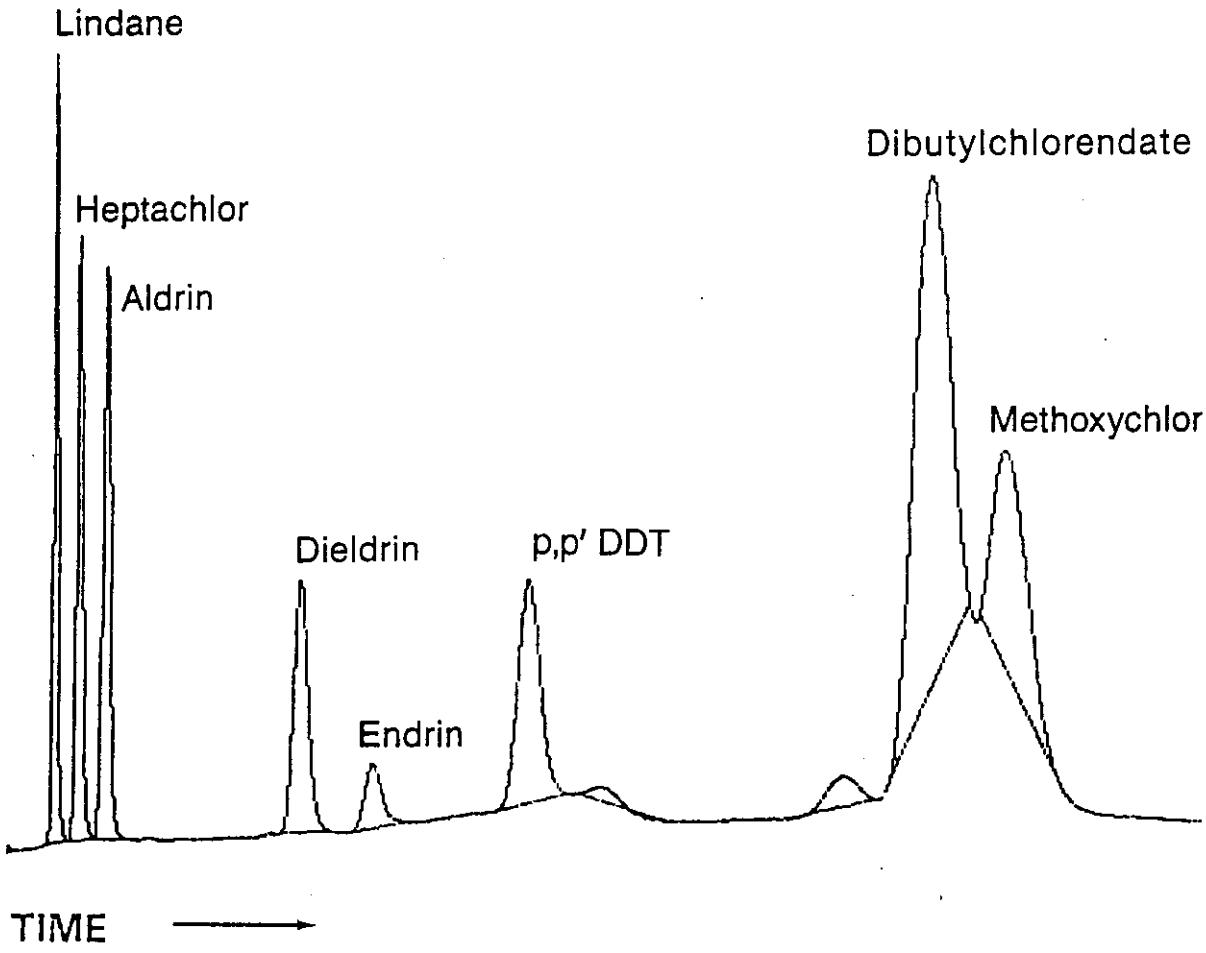

Figure 5. Chromatograph Showing a Mixture of Single Component Pesticides Determined by GC/ECD Using a Packed Column

OPERATING CONDITIONS

Column Type:	DB-5 0.32 capillary, 0.25 um film thickness
Column Temperature Program:	90°C (4 min)/16°C per min to 154°C/4°C per min to 270°C.
Detector:	Electron Capture
Carrier Gas:	Helium at 1 mL/min.
Make Up Gas:	5% Methane/95% Argon at 60 mL/min.

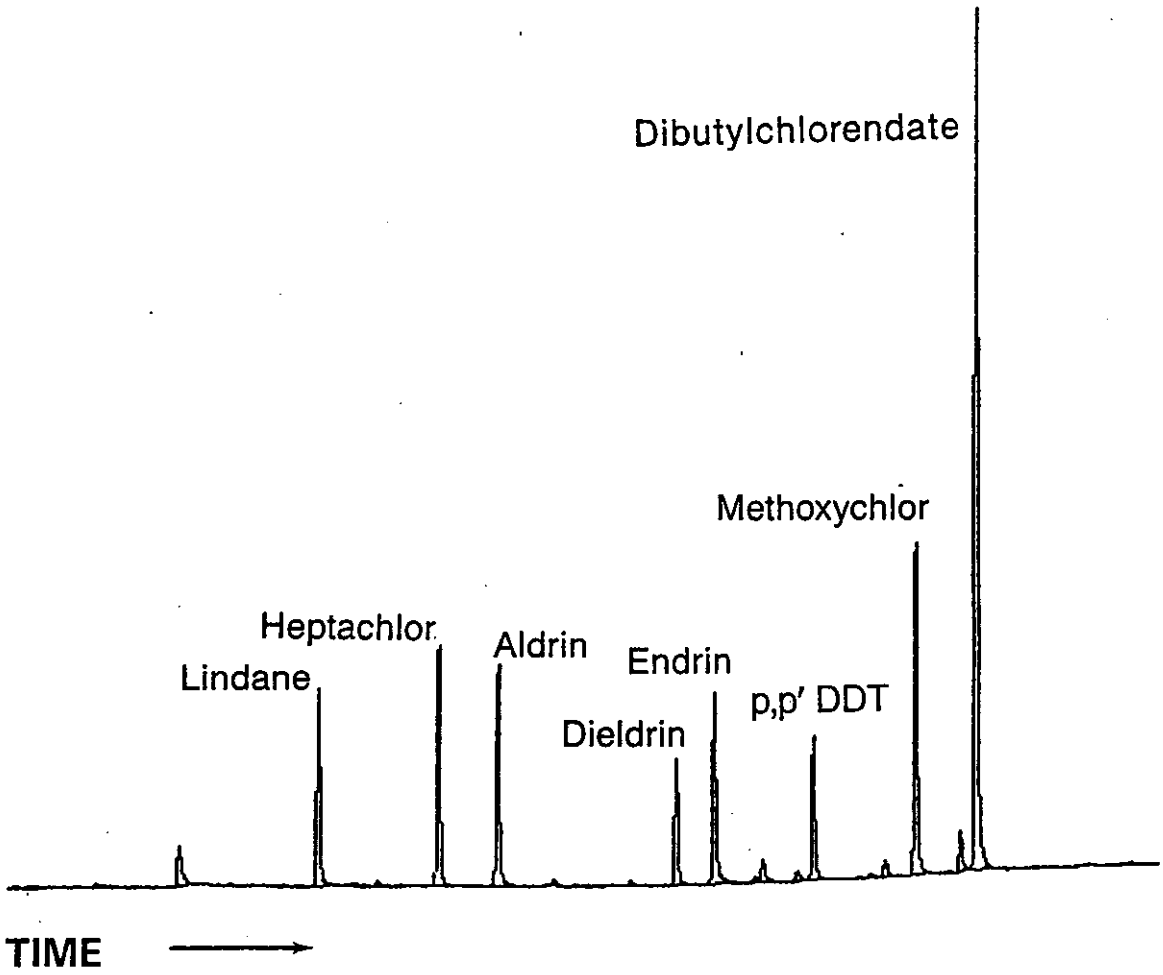

Figure 6. Chromatograph Showing a Mixture of Single Component Pesticides Determined by GC/ECD Using a Capillary Column

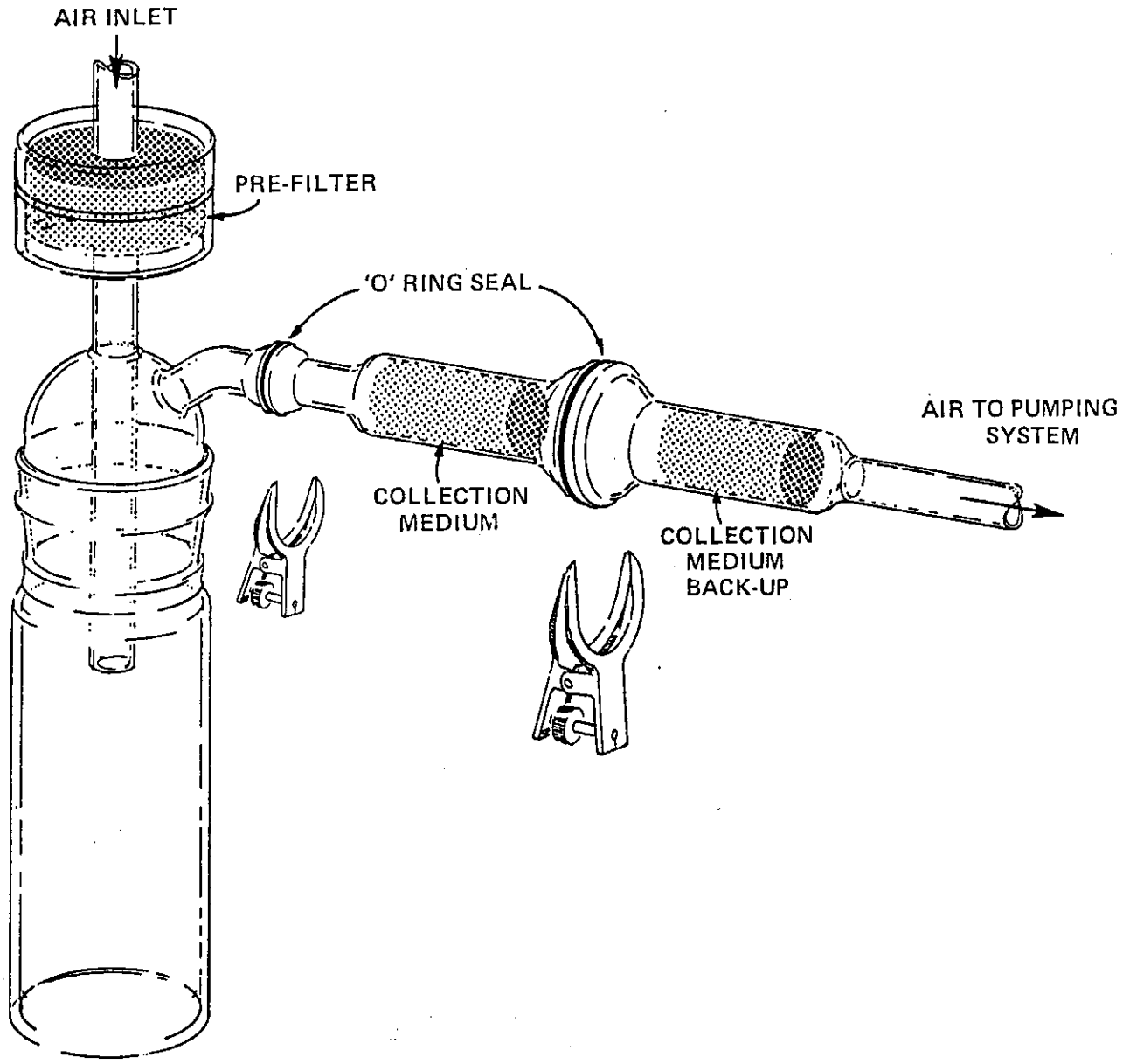

Figure 7. Apparatus for Determining Sampling Efficiencies

Chapter IP-9

DETERMINATION OF REACTIVE ACIDIC AND BASIC GASES AND PARTICULATE MATTER IN INDOOR AIR (ANNULAR DENUDER TECHNIQUE)

1. Scope

This document describes a sampling and analytical protocol for the annular denuder system (ADS). This system was developed to measure reactive acidic and basic gases and particulate matter which are contained in indoor ambient air. The chemical species which can be measured by the ADS are gaseous SO_2, HNO_2, HNO_3 and NH_3 and particulate $SO_4^=$, NO_3^-, NH_4^+ and H^+. Other similar chemical species can be successfully collected by the system with just a few simple modifications (i.e., changing the denuder coating solutions, the denuder sequence and the liner or filter types and sequence). Once collected, the pollutant concentrations are quantified by ion chromatography (IC) analysis and/or Technicon colorimeter autoanalysis. The IC protocols for sample preparation, analysis and quantification are detailed within the ADS method. The Technicon autoanalyzer protocols are utilized to quantify ammonia (NH_3), nitrate (NO_3^-), and sulfate ($SO_4^=$) in ambient air samples.

2. Applicability

2.1 Recently, these and other acid gases and aerosols, and particulate matter have been of growing concern to indoor air quality groups. Much emphasis has been directed to understanding the many chemical forms in which these pollutants can exist and the conditions which cause chemical changes to occur. Industrial and commercial facilities, as well as hazardous waste storage and treatment facilities, contribute significantly to indoor air contamination through various source-specific emissions. Although several of the previously mentioned pollutants can be instrumentally measured to quantify their concentration in the ambient air, many of the established methods are not adequate (or sensitive enough) to measure these pollutants at the levels typically found in non-urban locations. As a result, monitoring and research efforts have been designed to assess what sources are responsible for targeted pollutant emissions, what health and ecological impacts are incurred, and what the maximum allowable ambient concentrations should be.

2.2 The ADS has been utilized in such research efforts. The system's configuration has made it a very appealing asset to monitoring crews. Its ability to collect the chemical species of interest with little or no interference from sampling artifacts has separated it from other air monitoring techniques. Each sampling network can assemble the treated denuders and filters in such a manner that specific pollutants, which can cause ambient concentrations to be falsely assessed, are withdrawn from the air stream before interfering chemical reactions can occur. Subsequently, it is very important to investigate all possible chemical reactions between the species of interest before setting up the ADS.

2.3 As with all monitoring methods, the ADS has its limitations. Operation below 20% relative humidity may result in less than quantitative collection of SO_2. Also, the annular denuders are fragile and require great care when handled. Studies are being conducted to

determine how well Teflon® coated aluminum denuders collect acid aerosols. Other studies include identifying interferen ich can cause under- or over-estimations of pollutant concentrations to be made and unting for interferant reactions in the calculations.

Method IP-9

DETERMINATION OF REACTIVE ACIDIC AND BASIC GASES AND PARTICULATE MATTER IN INDOOR AIR (ANNULAR DENUDER TECHNIQUE)

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Appendix - Spectra-Physics Integrator Program for IC Analysis

Method IP-9

DETERMINATION OF REACTIVE ACIDIC AND BASIC GASES AND PARTICULATE MATTER IN INDOOR AIR (ANNULAR DENUDER TECHNIQUE)

1. Scope

1.1 This document describes the protocol for the quantitative measurement of reactive acidic and basic gases and particulate matter which are contained in indoor atmospheres.

1.2 The chemical species which can be determined by this method are gaseous SO_2, HNO_2, HNO_3, and NH_3 and particulate $SO_4^{=}$, NO_3^{-}, NH_4^{+}, and H^{+}, as well as the mass of fine particulate matter ($d_{50} < 2.5\ \mu m$). Detection and quantitation limits are given in Table 1.

1.3 The methodology detailed in this document is a composite of methodologies developed by U.S. Environmental Protection Agency (USEPA), Harvard University and the CNR Laboratories. It is currently employed in a number of air pollution studies in Italy, U.S.A., Canada, Mexico, Germany, Austria, and Spain, and in such institutions as public health services, epidemiology and environmental research centers.

1.4 The equipment described herein is utilized to measure acidic and basic gases and particulate matter contained in both indoor and outdoor atmospheres. The outdoor method was originally developed for monitoring regional-scale acidic and basic gases and particulate matter in support of U.S. EPA field programs involving the Integrated Air Cancer Research Program and the Acid Deposition Network. Similarly, the methodology has been used to characterize the urban haze in Denver, Houston and Los Angeles.

2. Applicable Documents

2.1 ASTM Standards

D1356 Definitions of Terms Related to Atmospheric Sampling and Analysis

2.2 Other Documents

Ambient Air Studies (1-9)
U.S. EPA Technical Assistance Document (10)

3. Summary of Method

3.1 Indoor air is drawn through an elutriator-accelerator jet assembly, an impactor frit and coupler assembly, and past glass denuder walls which have been etched and coated with chemicals that absorb the gaseous species of interest. The remaining air stream is then filtered through Teflon® and Nylasorb® membrane filters. Teflon® and nylon membrane filters are used to capture ammonium and nitrate aerosol and sulfate particulate matter. Nitric acid and sulfur dioxide will also be collected by the nylon filter but these measurements are treated as interference. Figure 1 illustrates the annular denuder system (ADS) assembled ready for testing. Figure 2 shows the field sampling box with the ADS and pump-timer (11).

3.2 After sampling, the annular denuders are extracted with 5 mL of deionized water. The extracted solutions are subsequently analyzed for ions corresponding to the collected gaseous species (see Figure 1). The filters are placed into filter bottles where five or ten mL of the IC eluent are pipetted into each filter bottle with the filters face downward and completely covered by the eluent. The filter bottle is capped and put in an ultrasonic bath for 30 minutes. The bottles are stored in a clean refrigerator at 5°C until analysis.

3.3 The analysis of anion and cation concentrations collected by the denuders and filter pack is typically performed by ion chromatographic and Technicon® colorimeter autoanalytic procedures. The H^+ concentration of extracts from the Teflon® filter downstream of the denuders is performed by use of pH measurements using commercially available pH meters calibrated with standards (11).

4. Significance

4.1 Reactive acidic (SO_2, HNO_2 and HNO_3) and basic (NH_3) gases and particles are found in the atmosphere as a result of emission from a variety of fossil fuel combustion sources including industrial and commercial facilities, hazardous waste storage and treatment facilities, etc. Measurements of these chemical species are currently being used in a broad range of environmental studies such as in 1) epidemiological programs to assess the impact of acid aerosols on respiratory impairment, 2) receptor modeling to determine the origin of particles that impact EPA's PM-10 air particulate standard, 3) assessment of the impact of particulate nitrate and sulfate on visibility, and 4) the quantification of the impact of acidic and basic air pollutants on issues related to acid rain.

4.2 The unique features of the annular denuder which separates it from other established monitoring methods are elimination of sampling artifacts due to interaction between the collected gases and particles, and the preservation of the samples for subsequent analysis which is accomplished by removing NH_3 in the gas stream by the citric acid coated denuder and reducing the probability of the particulate sulfate ($SO_4^=$) captured by the filter pack being neutralized to ammonium sulfate [$(NH_4)_2SO_4$]. If NH_3 is not extracted from the gas stream prior to filtration, correction of particulate sulfate and gaseous sulfur dioxide would be required for accurate measurements to be obtained.

5. Definitions

Definitions used in this document and any user prepared Standard Operating Procedures (SOPs) should be consistent with ASTM D1356. All abbreviations and symbols are defined within this document at the point of use.

5.1 Particulate mass - a generic classification in which no distinction is made on the basis of origin, physical state, and range of particle size. (The term "particulate" is an adjective, but it is commonly used incorrectly as a noun.)

5.2 Primary particles (or primary aerosols) - dispersion aerosols formed from particles that are emitted directly into the air and that do not change form in the atmosphere. Examples include windblown dust and ocean salt spray.

5.3 Secondary particles (or secondary aerosols) - dispersion aerosols that form in the atmosphere as a result of chemical reactions, often involving gases. A typical example is sulfate ions produced by photochemical oxidation of SO_2.

5.4 Particle - any object having definite physical boundaries in all directions, without any limit with respect to size. In practice, the particle size range of interest is used to define "particle." In atmospheric sciences, "particle" usually means a solid or liquid subdivision of matter that has dimensions greater than molecular radii (~10 nm); there is also not a firm upper limit, but in practice it rarely exceeds 1 mm.

5.5 Aerosol - a disperse system with a gas-phase medium and a solid or liquid disperse phase. Often, however, individual workers modify the definition of "aerosol" by arbitrarily requiring limits on individual particle motion or surface-to-volume ratio. Aerosols are formed by 1) the suspension of particles due to grinding or atomization, or 2) condensation of supersaturated vapors.

5.6 Coarse and fine particles - these two fractions are usually defined in terms of the separation diameter of a sampler. Coarse particles are those with diameters greater than 2.5 μm but less than 10 μm and that are collected by the sampler; the fine particles are those with diameters less than 2.5 μm and that are collected by the sampler.
Note: Separation diameters other than 2.5 μm have been used.

5.7 Annular - of, rotating to, or forming a ring. In the annular denuder sampler, the annular refers to the cylinder to which coating is applied to the interior parallel planes to remove gaseous pollutants by diffusion chemistry.

5.8 Denuder - the denuder refers to the process gaseous pollutants from the gas stream.

6. Interferences

6.1 Operation below 20% relative humidity (RH) may result in less than quantitative collection of SO_2. Atmospheric water vapor in concentrations above 30% RH has been shown not to be an interferant for SO_2 collection.

6.2 Studies are being conducted to identify interferents and calculations are being developed to correct the measurements obtained by the annular denuder system for identifiable interferents. For example, the presence of ozone (O_3) is known to oxidize nitrous acid (HNO_2) to nitric acid (HNO_3); therefore, measurements of HNO_2 are often underestimates. Calculations have been developed to adjust for this oxidation process and provide more accurate estimations of HNO_2 concentrations in the atmosphere.

6.3 Other studies include the possible chemical reactions (organic and inorganic) which may occur with selected coating solutions which interfere with the accurate measurement of the chemical species of interest.

6.4 The efficiency of impactor collection decreases when the impactor surface is loaded. The average operational time before such loading occurs has not been determined.

7. Apparatus

Note: The following descriptions relate to Figure 2. Most of these parts are available commercially by University Research Glassware. However, it is important to note that these items can be made by any qualified vendor; therefore, it is not necessary that these specific items are obtained and utilized.

7.1 Sampling

7.1.1 Elutriator and acceleration jet assembly - Under normal sampling conditions, the elutriator or entry tube is made of either Teflon® coated glass or aluminum. When using glass, the accelerator jet assembly is fixed onto the elutriator and the internal surfaces of the entire assembly are coated with Teflon®. When aluminum is used, the accelerator jet assembly is removable. The jet is made of Teflon® or polyethylene and the jet support is made of aluminum. Again, all internal surfaces are coated with Teflon®. Both assemblies are available with 2, 3 and 4 mm inside diameter jets (nozzles) [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.2 Teflon® impactor support pin and impactor frit support tools - Made of either Teflon® or polyethylene and are used to aid in assembling, removing, coating and cleaning the impactor frit [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.3 Impactor frit and coupler assembly - The impactor frit is 10 mm x 3 mm and is available with a porosity range of 10-20 μm. The frits should be made of porous ceramic material or fritted stainless steel. Before use the impactor frit surface is coated with a Dow Corning 660 oil and toluene solution for use, and sits in a Teflon® seat support fixed within the coupler. The coupler is made of thermoplastic and has Teflon® clad sealing "0"-rings which are located on both sides of the seat support inside the coupler. The couplers are composed of two free moving female threads which house the support tools when assembling and removing the impactor frit, and couple the denuders when sampling. There are arrows printed on the metal band which holds the female threads together. These arrows should be pointing in the direction of air flow (see Figure 1) when the ADS is assembled.

Note: In situations when there are substantial high concentrations of coarse particles (>2.5 μm), it is recommended that a Teflon®-coated aluminum cyclone be used in place of the acceleration jet and impactor assembly, as illustrated in Figure 3. The cyclone is made of Teflon®-coated stainless steel. Figure 4 illustrates the location of the cyclone with respect to the denuder, heated enclosure and meter box assembly ready for sampling [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.4 Annular denuder - The denuder consists of two concentric glass tubes. The tubes create a 1 mm orifice which allows the air sample to pass through. The inner tube is inset 25 mm from one end of the outer tube; this end is called the flow straightener end. The other end of the inner tube is flush with the end of the outer tube. Both ends of the inner tube are sealed. In this configuration, the glass surfaces facing the orifice are etched to provide greater surface area for the coating. There are three types of denuders available. One is the older version which accommodates the impactor support pin assembly, and can

only be the first denuder in sequence. It is available in glass with the impactor support holder made of glass and the impactor support pin assembly made of Teflon®. The denuder is 265 mm long with size #30 threads for coupling. It is available with flow straighteners at both ends; however, most denuders in use today only have one flow straightener end. The second most recent denuder version, which can be used as any denuder in sequence, is available in glass with only one flow straightener end. It is 242 mm long and has size #30 threads. Finally, the third denuder design involves two inner concentric glass tubes (1 mm separation) positioned around a solid center glass rod as illustrated in Figure 5. Once again, the glass surfaces are etched to provide greater surface area for the coating. The inner glass tubes and coater rod are inset 25 mm from one end of the outer Teflon®-coated stainless steel tube to serve as the flow straightener end. All denuder types should be equipped with thermoplastic or polyethylene caps when purchased [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.5 Caps for annular denuder - Caps are made of either polyethylene or thermoplastic and are used in the coating and drying processes, for storage and for shipment. The thermo-plastic caps include a removable Teflon® seal plate when purchased. Repeated reuse of these types of caps have caused some contamination due to the improper cleaning of the cap and Teflon® seal plate, i.e., fluid tends to be trapped under the seal plate. The polyethylene caps are not equipped with seal plates. Observation has concluded that polyethylene caps tend to dry faster and seal better than the thermoplastic caps. Less sample contamination has been reported, also [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.6 Annular denuder couplers - The couplers should be made of thermoplastic and equipped with Teflon® "O"-rings which sandwich a silicone rubber ring on three sides. This provides elasticity for better sealing under extremely cold temperature conditions in which Teflon® does not give. There are two types of couplers available. In the older version, the couplers have removable seal rings. Problems with denuder breakage and leakage due to improper threading of the couplers with the denuders led to the development of a second type of coupler. The new couplers are equipped with permanent seal rings which provide more even threading and a better seal when coupled. Some couplers have built-in flow-straighteners. The couplers are used to couple the annular denuders together and for coupling the last denuder with the filter pack [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.7 Drying manifold assembly - The manifold is made of pyrex and is available to accommodate as many as 4 drying denuders. The denuders are attached to the manifold with back-to-back Bakalite bored caps. The bored caps are connected with a Teflon® connector ring. Air is pushed through an air dryer/ cleaner bottle made of 2 1/2 inch heavy wall pyrex which contains silica gel, calcium sulfate and activated charcoal (not available with assembly). The tubing which connects the dryer/cleaner bottle to the drying manifold should be secured at each cap with either Teflon® washers or Teflon® washers coupled with Teflon® hose barbs [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.8 Filter pack assembly - The filters are supported by stainless steel porous screens and are housed in a polyethylene filter ring housing. The Teflon® filter ring housing directly follows the Teflon® filter housing inlet component. The "nylon" filter ring housing follows the Teflon® filter ring housing and sits on a Teflon® "O"-ring which seals the filter ring housing components to the filter housing outlet component. (There can be up to 4-filters in series depending on the species of interest.) The filter housing outlet component is aluminum and accommodates a polyethylene screw sleeve which seals the filter pack assembly. The sleeve is available in different lengths to accommodate up to 4 filter ring housing units. A stainless steel "Quick-Release" plug screws into the aluminum outlet component for connecting the pump-timer to the filter pack assembly. It is equipped with an orange "dust cover" (male plug) upon purchase [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.9 Vacuum tubing - Low density polyethylene tubing, 3/8 inch diameter for distances of less than 50 ft., 1/2 inch diameter for distances greater than 50 ft. Since this tubing is used downstream from the sampler, similar sized tubing or pipe of any material may be substituted. The tubing must have sufficient strength to avoid collapsing under vacuum [Fisher-Scientific, 711 Forbes Ave., Pittsburgh, PA, 15219, (412-787-6322)].

7.1.10 Tube fitting - Compression fittings (Swagelok®, Gyrolok® or equivalent) to connect vacuum tubing (above) to an NPT female connector or filter holder and connect vacuum tubing to fitting on differential flow controller. The fittings may be constructed of any material since they are downstream of the sampler [Fisher-Scientific, 711 Forbes Ave., Pittsburgh, PA, 15219, (412-787-6322)].

7.1.11 Annular denuder system (ADS) sampling box - The housing box is made of a "high-impact" plastic and is insulated with polyurethane. It is 4 feet long by 6 inches wide and 6 inches deep. There are two heater units, a fan blower and an air outlet located in the lid of the housing. Also, located on the lid are the automatic and manual control switches and a 12-V power supply outlet for the heater and fan. The bottom of the box houses the ADS. The elutriator end of the ADS protrudes through one end of the box, while the denuders are supported in the box by chrome plated spring clips. If the Teflon®-coated aluminum cyclone is used to remove coarse particles, it is also housed in the heated sampling box, with the elutriator end protruding through the sampling box, as illustrated in Figure 4. There is a vacuum plug known as a "quick-release" coupler that is linked to the filter pack of the ADS. This connects the ADS to 1 1/4 in. Teflon® rubber "clad" shrink tubing which exhausts the air stream to the ambient air. The box is sledge hammer proof [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.12 Annular denuder field-to-lab case - The field-to-lab case is made of rigid plastic and insulated with polyurethane. It is made to be hand carried, not shipped, and is used to transport 4 total annular denuder systems each consisting of either 3 annular denuder sections or 2 annular denuder sections and 1 denuder-impactor assembly. The systems are packed already assembled and capped, and either ready for sampling or ready for sample analysis. The case has a carrying handle, a lock and 3 latches and is equipped with 2 keys

[University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.13 Annular denuder shipping case - The shipping case is made of formica, backed with plywood and insulated with polyurethane. The corners are reinforced with metal. It is made to withstand shipping by truck, UPS and Federal Express. Each case is stackable and lockable and has a carrying handle. Seven total annular denuder systems can be packed in the case, provided each system contains 4 denuders each. The systems can consist of either 3 denuders (242 mm long) and 1 denuder-impactor assembly (265 mm long) or 4 denuders (242 mm long). Each component of the system is packed in its own storage compartment. The personal sampler assemblies can also be placed and shipped in this case [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.14 Differential flow controller (pump) - This unit pumps air through the sampler at a fixed rate of between 5 and 20 standard L/min (typically 10 L/min) with a precision of ±5% over the range of 25 to 250 mm Hg vacuum [University Research Glassware, 118 E. Main St., Carrboro, NC, 27510, (919-942-2753)].

7.1.15 Dry gas meter (DGM) - The DGM should pull 10 L of gas per revolution [Nutech, Corp., 2806 Cheek Rd., Durham, NC, 27704, (919-682-0402)].

7.2 Analysis

7.2.1 Ion chromatograph - A chromatograph equipped with the appropriate anion and cation exchange resin filled separator and suppressor columns and conductivity detector for measuring acidic (SO_2, HNO_2 and HNO_3) and basic (NH_3) ions in solution (i.e. denuder and filter extracts) [Dionex Corp., 1228 Titan Way, Sunnyvale, CA, 94086, (408-737-0700)].

7.2.2 Technicon colorimeter autoanalyzer - Colorimetic analyzer able to detect specific ions of interest in aqueous extracts [Technicon Industrial Systems Corp., 511 Benedict Ave., Tarrytown, NY, 10591-5097, (800-431-1970)].

7.2.3 pH meter - A pH or pH/ion meter with an "integral" automatic temperature compensation and calibrated with (EPA, N.S.T.) standard buffers (pH 4 and 7). Including 2 and 4 mL analysis cups (Orion and other vendors).

7.2.4 Polyethylene bottles with polyethylene screw caps - 50 mL and 100 mL, used for storage of coating solutions, best source.

7.2.5 Erlenmeyer flasks - 250 mL and 2 L borosilicate glass or polyethylene flasks with calibration, best source.

7.2.6 Graduated cylinders - 10 mL and 100 mL borosilicate glass or polyethylene cylinders, best source.

7.2.7 Pipets - Class A 5 mL and 10 mL borosilicate glass pipettes or automatic pipettes. Calibrated "to deliver," best source.

7.2.8 Pipet bulb - Made of natural rubber. Recommended to meet OSHA requirements, best source.

7.2.9 Micropipettes - Recommended 50 µL, calibrated "to contain," borosilicate glass micropipette, best source.

7.2.10 Forceps - Recommended dressing forceps made of stainless steel or chrome-plated steel and without serrations. Used for handling filters (Millipore).

7.2.11 Stopwatch - Used for measuring flow rate of gas stream through DGM, best source.

7.2.12 Ultrasonic cleaner - Used for filter extractions and parts cleaning. Most are temperature controlled. It is recommended to control the temperature during extraction at 65°C [Cole-Palmer Instrument Co., 7425 N. Oak Park Ave., Chicago, IL, 60648, (800-323-4340)].

7.2.13 Clean air hood - Closed air hood with ammonia free air circulation. Used for Teflon® filter extraction for pH analysis, best source.

8. Reagents and Materials

8.1 Teflon® filters - Zefluor® (PTFE) membrane filters 47 mm diameter with a 2 μm pore size. Only one side is Teflon® coated; this side should face the air stream [Gelman Sciences, 600 S. Wagner Rd., Ann Arbor, MI, 48106, (800-521-1520)].

8.2 Nylasorb® filters - Membrane filters 47 mm diameter with a 1 μm pore size. These filters are specially prepared and batch analyzed for low $SO_4^=$, NO_2^-, and NO_3^- background levels. If other brands of nylon membrane filters are used, they should be batch analyzed to ensure low and replicable levels of $SO_4^=$, NO_2^-, and NO_3^- [Gelman Sciences, 600 S. Wagner Rd., Ann Arbor, MI, 48106, (800-521-1520)].

8.3 Denuder extract storage vials - 30 mL (1 oz) screw-cap polyethylene sampling vials (Nalgene or equivalent). Allow eight (8) per sample for each sampling period, best source.

8.4 Filter extract storage vials - 100 mL polyethylene vials (Nalgene or equivalent). Allow two (2) vials for each sampling period, best source.

8.5 IC analysis vials and caps - The vials are available in 5 mL and 0.5 mL and are made of polypropylene. The filter caps are made of plastic and contain a Teflon® filter through which the sample is extracted for analysis. Both the vials and filter caps should be disposable, best source.

8.6 Labels - Adhesive, for sample vials, best source.

8.7 Parafilm - Used for covering flasks and pH cups during pH analysis, best source.

8.8 Kimwipes® and Kay-dry towels - Used for cleaning sampling apparatus and analysis equipment, best source.

8.9 Stoppers - Cork or polyethylene, best source.

8.10 Sodium carbonate (Na_2CO_3) - ACS reagent grade, best source.

8.11 Sodium chloride (NaCl) - ACS reagent grade, best source.

8.12 Methanol (methyl alcohol - CH_3OH) - ACS reagent grade, best source.

8.13 Toluene - ACS reagent grade, best source.

8.14 Glycerol (glycerin - $CH_2OHCHOHCH_2OH$) - ACS reagent grade, best source.

8.15 Citric acid (monohydrate - $HOC(CH_2CO)OH)_2COOH : H_2O$) - ACS reagent grade, best source.

8.16 Hydrogen peroxide (H_2O_2) - ACS reagent grade, best source.

8.17 Ethanol (C_2H_5OH) - ACS reagent grade, best source.

8.18 Sulfuric acid (H_2SO_4) - ACS reagent grade, best source.

8.19 Potassium chloride (KCl) - ACS reagent grade, best source.

8.20 Perchloric acid ($HClO_4$) - ACS reagent grade (60-62°C.), best source.

8.21 Distilled deionized water (DDW) - ASTM Type I water.

8.22 pH buffers - Standard buffers 4.00 and 7.00 for internal calibration of pH meter, best source.

8.23 Silica gel - ACS reagent grade (indicating type), best source.

8.24 Sodium bromide (NaBr) - ACS reagent grade, best source.

8.25 Activated charcoal - ACS reagent grade, best source.

8.26 Balance - Electronic analytical with internal calibration weights and enclosed weighing chamber. Precision of 0.1 mg [Fisher-Scientific, 711 Forbes Ave., Pittsburgh, PA, 15219, (412-787-6322)].

8.27 Gloves - Polyethylene disposable. Used for impactor frit assembly and filter pack assembly, best source.

8.28 Dow Corning high temperature vacuum oil - Dow Corning 660 oil used for impactor frit coating solution, best source.

8.29 Zero air - A supply of compressed clean air, free from particles, oil, NO, NO_2, SO_2, HNO_3, and HONO. The supply may be either from a commercial cylinder or generated on site, best source.

8.30 IC eluent solution - For extracting filters. This should be the same eluent as used for the ion chromatographic analysis of the filters. If the filter analysis is not to be performed by ion chromatography, then a slightly basic solution (e.g., 0.003 N NaOH or sodium carbonate/bicarbonate) should be used to extract the Nylasorb® filter, while the Teflon® filter should be extracted with DDW.

9. Preparation of Coating and Extraction Reagents

9.1 Impactor frit coating solution preparation - Weigh 1 g of silicone oil (Dow Corning high temperature 660 oil) and place in a 100 mL polyethylene storage bottle. Add 100 mL of toluene. Mix thoroughly, close container, and store at room temperature. (WARNING - FLAMMABLE LIQUID).

9.2 Impactor frit extraction solution preparation - Add 100 mL of IC eluent to a clean polyethylene storage container. Pipette 5 mL of methanol into container. Mix thoroughly. Store, covered at room temperature.

9.3 Annular Denuder Coating Solutions Preparation

Note: Different coatings may be used depending on the chemical species of interest.

9.3.1 NaCl coating solution - Clean a 100 mL polyethylene storage vial and let dry at room temperature. Weigh 0.1 g of reagent grade NaCl and add to vial. Add 90 mL of deionized water and 10 mL of methanol. Mix thoroughly; store, covered at room temperature.

9.3.2 Na_2CO_3 coating solution - Clean a 100 mL polyethylene storage vial and let dry at room temperature. Measure 50 mL of methanol (WARNING - TOXIC, FLAMMABLE LIQUID) with a graduated cylinder and pour into vial. Measure 50 mL of DDW with a graduated cylinder and add to vial. Weigh 1 g of glycerol and add to DDW. Weigh 1 g of a_2CO_3 and add to vial. Mix thoroughly, solution may fizz; wait for fizzing to stop before sealing vial. Store at room temperature.

9.3.3 Citric acid coating solution - Clean a 100 mL polyethylene storage vial and let dry at room temperature. Measure 50 mL of methanol (WARNING - TOXIC, FLAMMABLE LIQUID) with a graduated cylinder and pour into vial. Weigh 0.5 g of citric acid and add to vial. Mix thoroughly; store, covered at room temperature.

10. Elutriator and Acceleration Jet (Inlet) Assembly

Note: Figure 6A shows the all glass configuration.

10.1 The internal walls of the elutriator and jet assembly are coated with Teflon® to prevent losses of reactive species (SO_2, HNO_3, NH_3) during sampling. The elutriator prevents water and large particles from entering the inlet and thus extends the life of the impaction surface located immediately downstream of this assembly.

10.2 Figure 6B shows an aluminum version of this inlet. All inner surfaces of the aluminum unit are Teflon® coated. The main difference between the all glass and the aluminum inlet is the jet component of the aluminum inlet is replaceable as shown in Figure 3B. The jet component is made of either Teflon® or polyethylene and is available in various diameters as needed to accommodate selected sample flow rates. The jet may be replaced using the tool shown in Figure 6B. The jet diameter for a sample flow rate of 10 L/min is 3.33 mm. At this flow rate the inlet has a D_{50} cutpoint of 2.5 μm. If a different flow rate is to be used, the jet diameter must be changed to retain a D_{50} cutpoint to 2.5 μm. Figure 7A shows the relationship between jet diameter and flow rate to retain a D_{50} at 2.5 μm. Table 2 contains the jet diameters and Reynolds number to maintain a D_{50} of 2.5 μm cutpoint at different flow rates between 1 and 20 L/min.

Note: If the sampling area has substantial concentrations of coarse particles (>2.5 μm), the user may select to replace the acceleration jet and impactor assembly with the Teflon®-coated aluminum cyclone. The D_{50} cutpoint at a flow rate of 10 L/min is 2.5 μm, as illustrated in Figure 7B.

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11. Impactor Frit Preparation and Installation

11.1 Impactor Frit Installation

11.1.1 Impactor-coupler - The impactor-coupler assembly shown in Figure 8 is comprised of two parts: the replaceable impactor frit and the coupler-impactor housing seat. The impactor surface is a porous ceramic or porous stainless steel frit, 10 mm x 3 mm. This frit is inserted into the coupler-impactor housing using the tools shown in Figure 9. It is imperative that the in-tool is completely screwed in behind the impactor seat before the frit is pressed into place. The impactor frit is pressed gently but firmly into the seat of the impactor housing with your clean gloved finger. The impactor should fit into the housing so that it does not protrude above the seat. The impactor frit has a slight bevel. The narrow surface should be inserted into the impactor seat.

11.1.2 Impactor-denuder - The impactor-denuder assembly shown in Figure 9 is comprised of three parts: the replaceable impactor frit, the impactor seat support pin and the annular denuder impactor-pin support. The impactor frit is the same as described in Section 11.1.1 and is inserted, as previously described, into the impactor seat support pin. The impactor support pin can either be hand-held while inserting the frit or it can be placed upright into the aluminum frit holder #3 (see Figure 10). Press the support pin into the denuder pin support. The pin is grooved and has a viton "O"-ring to keep the pin snug in the denuder support during cold weather use (Teflon® tends to shrink at low temperatures). The support pin is removed by using the removal tool shown in Figure 9.

11.2 Impactor Frit Preparation

With the impactor frit in the impactor seat of either the coupler (see Figure 8) or the Teflon® impactor seat support pin which fits into the first denuder (see Figure 9), pipette 50 μL of the toluene-660 oil coating solution onto the impactor frit surface and allow to dry at room temperature. Cap both sides of the coupler impactor or denuder-impactor until use.

12. Filter Pack Preparation and Assembly

Note: Any number of filters can be used depending on the target species of interest. The configuration referred to in this section does not collect NH_{4+}.

12.1 With clean gloves, disassemble the filter pack (see Figure 11) by unscrewing the large outer Teflon® collar (sleeve) from the aluminum filter housing outlet component.
Note: It is necessary to remove the polyethylene cap first. Lay the pieces out on clean Kimwipes®. Insert black viton "O"-rings (see Figure 11).

12.2 Lay a clean Teflon® filter ring housing, with its large opening face-up, on a clean Kimwipe®. Place a clean stainless steel screen in the filter ring housing.

12.3 Using clean filter forceps, place a Nylasorb® nylon filter on the screen. Insert a second filter ring housing on top of the nylon filter with its large opening face-up. This forms a "sandwich" with the nylon filter held between the two filter ring housings.

12.4 Place another clean screen on the second filter ring housing. Using clean filter forceps, place a Teflon® filter on the screen.
Note: If a Teflasorb® Teflon® filter is used, be sure to place the Teflon® coated side, not the webbed side, toward the air stream. If the webbed side is facing the air stream, $SO_4^=$ extraction from the filters may be inefficient.

12.5 Place the Teflon® filter housing inlet component (see Figure 11) on top of the Teflon® filter. This forms another "sandwich" with the Teflon® filter held between the second filter ring housing and the housing inlet component. The housing inlet component connects the filter pack assembly to the last annular denuder through a thermoplastic coupler. Be careful not to twist the filterpack components, or damage will occur to the filters.

12.6 Lay the aluminum filter housing outlet component, with its large opening face-up, on a clean Kimwipe®. Insert a black viton "O"-ring in the aluminum filter base.

12.7 Insert the filter ring sandwiches (prepared in Sections 12.1-12.5) with the filter housing inlet component extending upward, on the viton "O"-ring in the aluminum filter base. Place the large outer Teflon® sleeve over the filter sandwich and screw onto the aluminum filter base. DO NOT OVERTIGHTEN! AND DO NOT TWIST FILTER PACK COMPONENTS!

12.8 Install the "Quick-Release" plug into the filter outlet component. DO NOT OVERTIGHTEN!

12.9 Install the polyethylene cap onto the filter inlet component and the orange dust cover onto the Quick-Release plug until ready to attach denuders.

13. Annular Denuder System Preparation

All new annular denuder parts obtained from suppliers should be cleaned by placing them in a dilute soap solution in an ultrasonic cleaner for about 10 minutes. The parts should then be thoroughly rinsed in DDW and allowed to dry at room temperature.

13.1 Annular Denuder Coating Procedure

Note: If the first denuder holds the impactor, a blank Teflon® impactor support pin should be installed in the pin support holder before the coating procedure.

13.1.1 Cap the end of the denuder which has the inner tube flush to the outer tube and set denuder upright on the capped end. For the denuders with flow-straighteners at both ends, either end can be capped. Measure 10 mL of the appropriate coating solution into a graduated cylinder. Pipette the 10 mL into the flow-straightener end of the upright capped annular denuder.

13.1.2 Cap the open end of the denuder and holding horizontally, rotate the denuder to distribute the coating solution evenly (see Figure 12).

13.1.3 Remove cap from flow-straightener end of denuder and decant excess coating solution into a clean denuder extract storage bottle labeled "denuder blank." Bottle label should include denuder number, coating solution and date.

13.1.4 Repeat this procedure with each denuder; label the denuders and bottles appropriately.

13.2 Annular Denuder Drying Procedure

Note: As denuders dry, they change from translucent to a frosted appearance. Denuders are dry when they become uniformly frosted.

13.2.1 Drying train and manifold clean air flow should be adjusted to 2 to 3 L/min. Close toggle valve controlling clean air flow through manifold before attaching denuders.

13.2.2 Attach flow-straightener end to drying manifold port at the back-to-back bored caps (see Figure 13).

13.2.3 Open toggle valve and allow clean air to flow through the tube for several minutes.

13.2.4 Close toggle valve, and reverse ends of tubes attached to manifold.

13.2.5 When an even frosted appearance is achieved, remove tubes from manifold, cap both ends with clean caps and store until ready for use. Turn off air to drying manifold.

13.3 Annular Denuder System (ADS) Assembly

Note: Described herein is an annular denuder system consisting of 4 denuders in series. Any number of denuders can be used as per the operators discretion. It is recommended to assemble the denuders in such a way that the flow-straightener end always follows the flush end of the previous denuder, except, in the event that denuders with flow-straighteners at both ends are used. This type of assembly allows laminar flow conditions to be restored.

13.3.1 Lay the ADS pieces on a clean surface (i.e., Kimwipes®).

13.3.2 Remove the end caps from the first denuder. Denuder 1 is coated with NaCl and may or may not hold the impactor frit pin support. If the first denuder is equipped with the impactor frit pin-support, remove the blank impactor support pin. Gently insert the impactor support pin and coated frit assembly into the denuder-pin support. If the first denuder does not hold the impactor pin-support, attach the impactor frit seat equipped coupler assembly to the flow-straightener end of the first denuder.

Note: DO NOT TIGHTEN! Do not tighten during the following procedure until Section 13.4.12 is reached.

13.3.3 Attach a thermoplastic coupler to the opposite denuder end. Place a Teflon® clad "O"-ring inside the coupler, if needed.

13.3.4 Remove the end caps of the second denuder (Na_2CO_3 coated). Attach the end with the flow-straightener section to the first denuder-coupler assembly.

13.3.5 Attach a thermoplastic coupler to the opposite denuder end. Place a Teflon® clad "O"-ring inside the coupler, if needed.

13.3.6 Remove the end caps of the third denuder (Na_2CO_3 coated). Attach the end with the flow-straightener section to the second denuder-coupler assembly.

13.3.7 Attach a thermoplastic coupler to the opposite denuder end. Place a Teflon® clad "O"-ring inside the coupler, if needed.

13.3.8 Remove the end caps from the fourth denuder (citric acid coated). Attach the end with the flow-straightener section to the third denuder-coupler assembly.

13.3.9 Attach a thermoplastic coupler to the opposite denuder end. Place a Teflon® clad "O"-ring inside the coupler, if needed.

13.3.10 Attach the filter pack inlet to the fourth denuder coupler assembly.

13.3.11 When using the first denuder equipped with the impactor frit-pin support, a thermoplastic coupler with a Teflon® clad "O"-ring is used to attach the inlet assembly. Attach but do not tighten!

13.3.12 Attach the elutriator-acceleration jet assembly to the first denuder-coupler assembly. Tighten very gently - DO NOT OVERTIGHTEN or breakage will result. (This applies when using either first denuder described).

13.3.13 Tighten the remaining couplers very gently - do not overtighten or breakage will result (see Figure 1).

13.3.14 Cap elutriator with orange dust cover until use.

Note: When collecting and measuring gaseous $HN0_2$, HNO_3, SO_2, and NH_3, and particulate NO_3^-, NH_4^+, and $SO_4^=$, it is essential to assemble the annular denuders as previously described. It is impossible to distinguish the difference between deposited HNO_2 and HNO_3 if the NaCl coated denuder does not precede the Na_2CO_3 coated denuder. It is impossible to quantify the amount of HNO_2 collected if there are not two Na_2CO_3 coated denuders in series. Also, NH_3 must be taken out of the gas stream prior to the air stream entering the filter pack. Otherwise, reaction of the unneutralized sulfate will result. If ammonia (NH_3) and/or H^+ measurements are not to be analyzed for, then the use of a citric acid coated denuder is not important. However, with the removal of NH_3, some nitrate collected on the Teflon® filter will tend to evaporate and be found on the nylon filter.

13.4 Laboratory Leak-Check of ADS

Note: CAUTION - Do not subject the system to sudden pressure changes or filters may tear.

13.4.1 Remove the orange dust cap from the impactor opening. Attach the "Quick-Release" to a pump module. Turn on the pump. Be certain that flow through the ADS occurs by checking the rotameter.

13.4.2 Briefly cap the elutriator with the orange dust cap. The flow as indicated on the rotameter should drop to zero if no leaks exist.

13.4.3 Disconnect the pump from the ADS at the "Quick-Release" plug. Cap the "Quick-Release" plug with an orange dust cover. Turn off the pump. REMEMBER - Never overtighten joints or breakage will result. If the joints can not be sealed with gentle tightening, then the Teflon® "O"-rings are worn or defective and must be replaced.

13.4.4 Place the assembled sampler in its field-to-lab carrying case for transport to the field (see Figure 14).
Note: It is recommended that the ADS joints be loosened slightly when extreme temperature changes are incurred during transportation. This will prevent unnecessary breakage or distortion of the ADS components. Remember to allow the system to adjust to the indoor air temperature before tightening the joints and checking for leaks.

14. Sampling

14.1 Start-up

14.1.1 Remove the ADS from its field-to-lab carrying case and load into the field sampling box. The ADS field sampling box is insulated with polyurethane which is configured to hold the ADS without allowing movement. Chromeplated spring clips hold the denuders in place. Automatic and manual control switches allow the sampling box to control the temperature of the ADS. The automatic switch should be used when the ADS is not in use and when the ADS is sampling for extended periods of time without constant supervision to prevent low temperature or sudden pressure change exposure of the ADS (these types of exposure can cause leaks to occur, condensation, or the filters to tear). When sampling, the ADS should be kept 1°C above the indoor temperature to prevent condensation. The sampling box has two connections with the pump timer: the plastic suction hose connected with "Quick-Release" couplers and the 12-V power cord with a "Quick-Disconnect" coupler. The power cord remains connected, and the suction hose is disconnected from the box each time the unit is opened. Inside the box, the hose is connected to the top of the filter pack with a "Quick-Release" coupler. During sampling the sample box is kept securely closed (see Figure 2).

14.1.2 Allow the pump to warm up for 20-30 minutes prior to testing so the pump will provide steady flow during testing.

14.1.3 To check the Heat/Cool cycles, flip one switch from "AUTO" to "MANUAL" and the other between "COOL" and "HEAT." Check to insure that the fan and heater (i.e., light bulb) work, respectively.

14.1.4 With the elutriator still capped, turn on the pump with the switch on the timer. The rotameter should indicate zero flow. If there is a flow, the assembly pieces need to be recoupled. Run leak check for 5-10 seconds, then turn off pump and remove elutriator cap. Record leak rate on Field Test Data Sheet (see Figure 15).

14.1.5 Attach DGM output to elutriator inlet. Turn on pump. Record start time on Field Test Data Sheet (see Figure 15). Using a stopwatch, record the time for 20.0 L to pass through the DGM. Record the DGM temperature and the absolute pressure of the DGM.

14.1.6 Calculate the flow rate as follows:

$$Q_{STD} = (V/T)(P_b/P_{STD})(T_{STD}/T_m)(F_c)$$

where:
Q_{STD} = flow rate corrected to standard conditions, O°C and 760 mm Hg, L/min
V = volume of gas pulled through denuder system, L

T = time required to pull 20 L of gas through denuder system, minutes
P_b = barometric pressure, mm Hg
P_{STD} = standard barometric pressure, 760 mm Hg
T_{STD} = standard temperature, 273°C
T_m = temperature of dry gas meter, 273°C + T_m
F_c = dry gas meter correction factor, dimensionless

14.1.7 If the calculated flow rate is not between 5 and 16 L/min, typical 10 L/min, then readjust the flow rate and repeat Sections 14.1.4 and 14.1.5 until the rate is in the above range. Preliminary studies should be conducted to obtain an estimate of the concentrations of the species of interest.

14.1.8 Record the flow rate on Field Test Data Sheet.

14.1.9 Remove DGM connection tubing from elutriator inlet. Pump should remain running so that sampling continues. Higher flow rates may be used for shorter sampling periods. Concentration of the species of interest in indoor air and the configuration of the sampling equipment, determine the appropriate flow rates. Sampling at 10 L/min, requires a sampling time of 24 hours for the collection of pollutant concentrations between 0.02 and 0.83 $\mu g/m^3$.

14.2 Sample Shutdown

14.2.1 Attach DGM connection tubing elutriator inlet with pump still running. Measure flow rate as in Sections 14.1.5 and 14.1.6. Record flow time, temperature, and pressure on Field Test Data Sheet (See Figure 15).

14.2.2 Turn off pump. Record time and elapsed time meter reading on log sheet. Remove DGM connection tubing from elutriator inlet. Remove ADS from the sampling box, cap the ends, and place the ADS in field-to-lab carrying case for transport to lab. Be careful not to stress the ADS during the transfer or breakage will result. CAUTION - When the ADS is brought from a cold field sampling location to a warm laboratory, it is necessary to loosen the denuder couplings to prevent thermal expansion from breaking the denuders.

14.3 Corrective Action for Leak Test Failure

Note: These steps should be followed when failure occurs during testing at the laboratory before transport to the field and in the field before testing.

14.3.1 Sampler leaks - Note the problem on the Field Test Data Sheet. Check assembly of ADS components. Replace gaskets. Check for proper seating of denuder surfaces. Replace any defective parts.

14.3.2 Cracked or chipped denuders or elutriator assemblies - Note problem on Field Test Data Sheet. Discard defective pieces. Do not try to extract cracked pieces. WARNING - use caution when disassembling cracked glassware. Pieces may shatter and cause severe cuts. Wear protective clothing.

14.3.3 Contaminated blank solutions - Note problem on Field Test Data Sheet. Follow parts cleaning procedures closely. Examine the sampler preparation area for possible sources of contamination and remove source, if found. Check DDW being used in the

solution preparations and extractions: Fill a clean 25 mL polyethylene extraction bottle with the DDW used in solution preparation and extraction, send to lab for analysis. If contaminated, correct deionization system.

14.3.4 Flow rate disagreement - Note problem on Field Test Data Sheet. Check vacuum gauge on flow module. If a high vacuum exists then the sampler has become blocked. This may be due to dust or smoke particles clogging the filters or to obstructions in the system or tubing. Check flow module. Repair as needed.

14.3.5 Inadequate flow rate - Note problem on Field Test Data Sheet. Check rotameter on flow controller. If adequate flow is shown here, then a leak exists between the controller and the DGM. If no flow is shown on rotameter, then check vacuum gauge on controller. If no vacuum exists, then pump needs repair. If a high vacuum is shown, then an obstruction exists in the system. Check to see that the paper filter dividers were not accidentally installed with the filters in the filter pack. Check tubing for kinks.

Note: Typically the pressure drop across the filters should be approximately 1 inch Hg at 10 L/min flow rate at sea level. This pressure drop can vary from 1-10 L/min depending on elevation.

15. ADS Disassembly

15.1 Remove the ADS from the field-to-lab carrying case using both hands. To prevent stress, hold the ADS by its ends. CAUTION - Do not stress the ADS while removing it from the case.

15.2 Decouple the elutriator - jet assembly from the first denuder-impactor-coupler assembly.

15.3 When using the denuder-impactor, the frit-pin must be removed from the support in the denuder before removing the frit from the pin (see Figure 9). The frit is then extracted from the pin using pin tool #3 and the frit extraction tool (see Figure 10). When using the impactor-coupler assembly, the frit is removed from the coupler seat using pin tool #3 and the "out" frit removal tool (see Figure 16). Put frit in covered dish and set aside for chemical extraction.

15.4 Remove the denuders from the couplers and cover each end of the denuders with clean end caps until extraction.

15.5 Label a clean 100 mL polyethylene bottle with the sampler ID number and filter type (i.e., Teflon® or Nylasorb®, as appropriate) for each of the filters.

15.6 Disassemble the filter pack in a clean, ammonia-free air hood. Clean all hood surfaces and utensils with methanol. Wearing clean gloves and using clean filter forceps, remove the filters and place each in its storage (protective) bottle, with the exposed filter surface facing downward, until extraction.

Note: Be careful to place the filters in the properly labeled bottles.

16. Extraction Procedures

Special precaution: Samples should be analyzed as soon after collection as possible. It is imperative that the solutions and extraction procedures are prepared and performed on the day of pH analysis. Extraction must take place in a clean, ammonia-free, air hood. The extracts must be processed in the order in which they will be analyzed, so that each sample will have a similar time interval between extraction and analysis. Denuder extracts and filters should be stored in the refrigerator until just prior to analysis. Samples stored longer than 30 days tend to degrade due to bacteria growth and/or losses to the walls of the extraction vessel.

16.1 Impactor Frit Coating Extraction

16.1.1 Place the impactor (which was removed before denuder extraction) into a small extraction bottle.

16.1.2 Label the bottle appropriately. Pipet 10 mL of impactor extraction solution into the bottle. The solution must cover the surface of the impactor frit.

16.1.3 Close the extraction bottle and place in an ultrasonic bath for 30 minutes.

16.2 Denuder Extraction

Note: If the denuder was the first denuder, which is equipped with the impactor frit-pin support, insert a clean Teflon® impactor frit-pin, without frit in place. Then extract as described below. This procedure is to be followed for each denuder.

16.2.1 Cap one end of the denuder. Add 5 mL of DDW with a pipet. Cap other end.

16.2.2 Rotate the denuder to wet all surfaces thoroughly with the water. Remove the cap and pour the liquid into a clean 25 mL polyethylene extraction bottle.

16.2.3 Repeat this procedure with a second 5 mL of DDW extract (total extract volume is 10 mL which is placed into a single bottle).

16.2.4 Replace the extraction bottle cap and label the bottle with the sampler ID number, denuder number and type (as appropriate).

16.3 Filter Extraction

16.3.1 Teflon® Filter Extraction (for pH analysis followed by ion chromatography (IC) analysis)

Note: Teflon® is not wet by water; therefore, the filter will float on top of aqueous solutions. It is imperative that the solutions and extraction procedures are prepared and performed on the day of pH analysis. Extraction of the filters must take place in a clean, ammonia-free, air hood. The filters must be processed in the order in which they will be analyzed, so that each sample will have a similar time interval between extraction and analysis.

16.3.1.1 Allow the hood to be flushed with ammonia-free air for at least 5 minutes before filter extraction. All of the hood surfaces and extraction utensils must be cleaned with a Kimwipe® moistened with ethanol.

16.3.1.2 Pipet 3 mL of 0.0001 N perchloric acid ($HClO_4$) solution into the appropriately labeled extraction vial (4 mL).
Note: It is necessary to use $HClO_4$ because it inhibits CO_2 from dissolving into the solution and keeps the organic compounds in solution from dissociating. Both these activities, if allowed to take place, can cause the ionic strength of solution to change.

16.3.1.3 Place the Teflon® filter in the extraction vial. Cap tightly. Store at 5°C in the dark until ready for analysis.

16.3.1.4 When ready for analysis, the filter must be prepared (within the air hood) in the following manner: Using forceps and gloved hands, lift the filter from the extraction vial. Let the excess solution drain off into the vial. Holding the filter over the extraction vial, and using an automatic pipet, apply 100 ± 5 mL of ethanol to the filter. Add the ethanol slowly to ensure that all portions of the membrane are wet with ethanol. Immerse the filter in the aqueous solution once again. Tap the forceps against the inside of the vial to remove liquid. Tightly replace cap. Put in ultrasonic bath for 15 minutes total, rotating the rack 90° every 5 minutes.
Note: Perchloric acid is used in place of potassium chloride, initially, to prevent interference in the measurements of cations and anions by ion chromatography. Potassium chloride must be added to the portions of the sample extract which are used for pH analysis (the purpose of the salt, final concentration 0.04 M, is to increase the ionic strength and thus to reduce the time for equilibrium of the pH electrode used for measurement). Note also that it is necessary to use the same bottle (freshly opened) of ethanol for the extraction of the Teflon® filters that is used for the preparation of sulfuric acid standards.

16.3.1.5 When ready for pH analysis, the extracts are prepared in the order of pH measurement. Inside the air hood, remove the caps from 4 mL extraction vials. Wipe off any drops which may leak onto the outside of the cup.

16.3.1.6 Using gloved hands and a 1 mL automatic pipet, transfer 1 mL of the extract to each of two correspondingly labeled 2 mL cups.
Note: The first 2 mL cup for each extract has the same I.D.# as the 4 mL cup and the second 2 mL cup has the same I.D.# with a hyphen (-). This is the same system used with the working standards.

16.3.1.7 After transferring the extracts to the 2 mL cups, recap the 4 mL extract cup. Then store the 4 mL cups at 5°C in a refrigerator pending sulfate analysis by IC.

16.3.2 Nylon Filter Extraction

16.3.2.1 Pipet 10 mL of IC eluent into the appropriately labeled filter vial or bottle with caps.
Note: Be sure that the filter lies flat on the bottom of the bottle and that all of the filter is covered by the extraction solution.

16.3.2.2 Replace the bottle's cap and put in an ultrasonic bath for 30 minutes.

16.3.2.3 Store the bottles in a clean (i.e., pollutant free) refrigerator at 5°C in the dark until analysis.

17. Ion Chromatography Analysis

Note: The analytical procedure scribed here is not the only appropriate procedure available for quantifying the analyt of interest. It is not necessary that an automated system be utilized. This particular analytical procedure was chosen because it is presently being utilized by EPA. Modifications to this procedure may be required depending on the intended use of the data, however, any modifications made must be justified in order to obtain comparable data quality.

17.1 Standards Preparation

Special Precaution: Storage of these solutions should be no longer than one week. All of the working standard solutions are used to calibrate the IC and are made from reagent grade stock. The crystals are dried overnight in covered petri dishes at 110°C in a vacuum oven prior to preparing the standard solutions. Any yellowish discoloration of the dried crystals indicates decomposition and crystals should be discarded.

17.1.1 Sodium Sulfate Stock Solution

17.1.1.1 In a clean, calibrated, 1 L flask, add 500 mL of DDW.

17.1.1.2 On weighing paper, weigh out enough reagent (Na_2SO_4) to make the solution 2000 ppm concentration. The target weight is 0.7394 g. Record the gross weight. Note: It is best to weigh out slightly more than the target weight due to the adherence of the residual crystals to the weighing paper (the residual left on the paper is generally between 0.1 mg and 1 g).

17.1.1.3 Add the reagent crystals to the 500 mL of DDW. Reweigh weighing paper and subtract weight from the gross weight. The difference is the actual net weight.

17.1.1.4 Using a proportion, calculate the actual volume needed to make the solution 2000 ppm (see below).

$$\text{target wt/actual net wt} = 500 \text{ mL (target)/actual volume}$$

or

$$\text{actual volume} = (500 \text{ mL} * \text{actual net wt})/\text{target wt}$$

17.1.1.5 Using the appropriate calibrated pipet, add the amount of DDW needed to achieve the calculated actual volume. Mix well and cover with parafilm.

17.1.2 Sodium Nitrate Stock Solution

17.1.2.1 In a clean, calibrated, 1 L flask, add 500 mL of DDW.

17.1.2.2 On weighing paper, weigh out enough reagent ($NaNO_3$) to make the solution 2000 ppm concentration. The target weight is 0.6854 g. Record the gross weight.
Note: It is best to weigh out slightly more than the target weight due to the adherence of residual crystals to the weighing paper.

17.1.2.3 Follow Sections 17.1.1.3 through 17.1.1.5.

17.1.3 Sodium Nitrite Stock Solution

17.1.3.1 In a clean, calibrated, 1 L flask, add 500 mL of DDW.

17.1.3.2 On weighing paper, weigh out enough reagent ($NaNO_2$) to make the solution 1000 ppm concentration. The target weight is 0.7499 g. Record the gross weight.

Note: It is best to weigh out slightly more than the target weight due to the adherence of residual crystals to the weighing paper.

17.1.3.3 Follow Sections 17.1.1.3 through 17.1.1.5.

17.1.4 Standard working solutions - The working solutions are made up as follows: Add 10 mL each of the three stock solutions (Na_2SO_4, $NaNO_3$, and $NaNO_2$) to a 200 mL volumetric flask and dilute to the mark with DDW. Subsequent dilutions are carried out using a 10 mL volumetric pipet and appropriate flasks. Standards of 20, 10, 5 and 1 ppm Na_2SO_4 and $NaNO_3$ (and one-half these concentrations of $NaNO_2$) are prepared. These are used to calibrate the IC.

17.2 Reagent Preparation

Note: Storage of these reagents should be no longer than one week.

17.2.1 Anion eluent - The anion eluent is a solution of 1.8 μm Na_2CO_3 and 1.7 μm $NaHCO_3$. A concentrated solution can be prepared and diluted as needed.

Note: See Anion Storage Solution

17.2.1.1 Concentrated Na_2CO_3 solution (0.36 M) - Weigh out 38.156 g of Na_2CO_3 (MW = 105.99). Dissolve into 1 L of DDW. Store in refrigerator until ready to dilute.

17.2.1.2 Concentrated $NaHCO_3$ solution (0.34 M) - Weigh out 28.564 g of $NaHCO_3$ (MW = 84.01). Dissolve into 1 L of DDW. Store in refrigerator until ready to dilute.

17.2.1.3 Dilution of stock solutions - Bring both solutions to room temperature. Accurately pipet 10 mL of each solution into a 2000 mL volumetric flask which has been partially filled with DDW. Bring to the mark with DDW (1:200 dilution).

17.2.2 Anion regenerant - The regenerant is a 0.025 N H_2SO_4 solution. VERY CAREFULLY dispense 2.8 mL of concentrated Ultrex sulfuric acid (36 N) into a graduated cylinder. Partially fill the regenerant reservoir with DDW (3 L). Slowly add the acid to the regenerant reservoir. Bring to the mark with DDW (4 L).

Note: Protective clothing and eye protection should be utilized.

17.2.3 Cation eluent - There are two cation eluents that are used for the analysis of monovalent and divalent cations. The strong cation eluent is: 48 μm HCl, 4 μm DAP.HCl, 4 μm Histidine.HCl (DAP = Diaminoproprionic acid). The weak eluent consists of 12 μm HCl, 0.25 μm DAP.HCl, 0.25 μm Histidine.HCl.

17.2.3.1 Strong cation eluent - Weigh 0.560 g DAP and 0.840 g histidine into a one liter volumetric flask. Add 48 mL of 1 M HCl (Ultrex) to the flask. Bring the eluent to the final volume by bringing to the mark with DDW. Mix thoroughly to dissolve.

17.2.3.2 Weak cation eluent - Place 63 mL of the strong cation eluent in a 1 L flask. Add 9 mL of 1 M HC1 to the flask. Bring the eluent to the final volume by bringing to the mark with DDW. Mix thoroughly to dissolve.

17.2.4 Cation regenerant - The cation regenerant consists of 100 μM Tetrabutyl-ammoniumhydroxide (TBAOH). Place the TBAOH container into a warm water bath to dissolve any crystals that may have formed. Measure 266.7 mL of the TBAOH (stock reagent is supplied as 1.5 M, 40% in water) into a graduated cylinder. Add the TBAOH to 4 L of DDW.

17.2.5 Anion storage solution - Since the anion columns contain carbonates from the eluent, protection must be taken against microorganisms that will live on this food source and clog up the columns. If the columns are not being used for long periods of time (>2 weeks), a storage solution of 0.1 M NaOH should be pumped into them.

17.3 Sample Preparation

17.3.1 Mark the auto sampler vials with the appropriate identification numbers. Place the vials in an (IC) autosampler tray.

17.3.2 Using clean, calibrated 0.5 mL pipets transfer the denuder and the remainder of the filter extracts from the extraction vials to a clean disposable 0.5 mL (IC) autosampler (polyethylene) vial. Fill the autosampler vial up to the line on the side.

Note: If refrigerated, the contents of the 4 mL extraction vial must be vortex-mixed prior to transfer to the autosampler vials.

17.3.3 Place black filter caps on top of the vials. Use the tool provided to push the caps into the vials until they are flush with the top. (see the IC manual for more detailed instructions).

17.3.4 Wipe away any excess fluid from the top of the vial to avoid contamination from other samples.

17.3.5 After all of the trays are filled, place them into the left side of the autosampler. The white dot on the tray indicates the first sample. Press the button labeled RUN/HOLD to the RUN position. The trays should move until the first sample is under the sampling head. The front panel should indicate a READY message. Press local/remove switch to remove.

17.4 Basic System Operations - Start-up and Shut-down

17.4.1 Start-up Procedure for Ion Chromatograph

17.4.1.1 Figure 17 illustrates the major components of the Dionex 2020i Ion Chromatography system. Turn helium and nitrogen tanks on by opening the valve on top of each tank (pressure in either tank should not be less than 500 psi. Replace if necessary). Open valves at the outlet end of both regulators. Pressure on the nitrogen regulator is adjusted to 100 psi. Pressure on the helium regulator is adjusted to 14 psi.

17.4.1.2 Check the level of eluents and regenerating solutions. Turn the chromatography (CMA) values for the anion channel switch ON. Verify that the pressure

reading on the face of the degassing unit is 7 psi. Adjust by turning dial next to pressure gauge. Turn the degas switch to HIGH.

17.4.1.3 Turn the eluent reservoir switches, corresponding to the eluents to be degassed, to the ON position. Let the eluents degas on HIGH for 3-5 minutes, then turn degas switch to LOW.

17.4.1.4 Select the appropriate program on the gradient pump module using the PROGRAM switch. (Programs are recalled from memory by first pressing the PROGRAM switch, then the single digit reference number corresponding to the appropriate program).

17.4.1.5 Priming the eluent lines.

Note: All of the eluent lines used during analysis must be primed to remove any air bubbles that may be present. The selected program identifies which lines are used.

- Open the gradient pump drawer. Turn the pump to the START position for 10 seconds, or until a CLICK is heard, then turn the pump OFF. This step opens the valve to the eluent line displayed on the front panel.
- Attach a 10 mL syringe to the priming block on the face of the gradient pump module. With the priming block valve closed, pull the syringe plunger out to the end of the syringe.
- Open the priming block valve. The syringe will quickly fill with eluent. Close the valve on the priming block when the syringe is almost full. Remove syringe from block and discard collected eluant.
- This priming procedure can be repeated if necessary. All of the eluent lines that are to be used during a day of analysis should be primed at this time.

17.4.1.6 Open the door of the Advanced Chromatography Module. On the back of the door, at the bottom, is the conductivity detector. There are four labeled lines (anion, cation, waste, and cell) located next to the cell. The plumbing must be configured according to the type of analysis to be performed. If anions are being analyzed, the ANION line must be attached to the CELL line, and the CATION line must be attached to the WASTE line. If cations are being analyzed, the CATION line must be attached to the CELL line, and the ANION line must be attached to the WASTE line. The line coming from the pump must be attached to the correct port on the advanced chromatography module. SYSTEM 1 on the left is for anions, SYSTEM 2 on the right is for cations.

Note: If switching from one system to the other, the pump and the lines coming from the pump must be purged of the original eluent. This is done by disconnecting the pump line from the chromatograph module, turning the pump on and running the new eluent into a waste beaker for 2-3 minutes.

17.4.1.7 Select the columns to be used (labeled pH or NO_2) by pressing the blue button located below the labels. To verify that the correct columns are being used, the switch should be pressed at least once, and then set to the appropriate position. This is done in case the indicator light is reflecting a "default" setting, regardless of the actual position of the switch.

17.4.1.8 Turn the power switch on the autosampler ON (switch is located on the back of the unit, on the right). The default settings will be displayed on the front panel. Attach

the SAMPLE OUT line from the autosampler to the advanced chromatography module. The connection should be made to the port marked SAMPLE of the appropriate system. Turn the pump to START.

17.4.1.9 Turn the conductivity cell ON. Switch is located on the gradient pump module. Turn the REGEN switch for the appropriate system ON. Verify that regenerant is flowing by inspecting the regenerant waste line which empties into the sink. Open the advanced chromatography module door and inspect for leaks at columns, fittings, etc. Shut pump off if leaks are found.

17.4.1.10 Turn stripchart recorder ON. Baseline should stabilize in less than 20 minutes. If baseline is not stable, see troubleshooting Section 17.5 for assistance.

17.4.2 Data acquisition start-up - The following is a description of the current data acquisition program used by the U.S. EPA. The program is available (U.S. EPA, Atmospheric Chemistry and Physics Division, Office of Research and Development, Research Triangle Park, NC) and is for IBM or IBM compatible computers. Other appropriately designed programs may be used to compile the data collected for any given sampling network. It is not necessary to use a computer programmed integrator for the computation of data, however, for large sampling networks, it is recommended.

17.4.2.1 Turn on the IBM XT computer. From the C:>prompt, type: cd/cchart, then type: cchart. This loads the Chromatochart software. Turn switch on relay box to ENABLE, indicator light could go on.

17.4.2.2 Press F2 to enter the methods development module. Select option number 1 - "select channel # and load method file." "Select channel # <0>" type 0 or press ENTER to select the default choice shown in the brackets (in this case 0). "Load method file named" type the name of the appropriate method, then press ENTER. A directory of all of the current methods in memory can be obtained by pressing the F2 function key.

17.4.2.3 Press F3 to enter the Data Acquisition module. At this point you will be asked to save the method file. If there has not been any changes to the methods file, it does not need to be saved. Select option #4 - "Collect Data." Press ENTER to deactivate the method queue. "Load Run Queue named," type the name of the run queue if one has been created. Type ENTER to deactivate the run queue.

17.4.2.4 "Total # runs for method <1>," type how many times the method is to be repeated (total number of samples). "Autoanalyze Data" type Y. "Autosave data to disc" type Y. "Data file name (xxxx) change?", type data file name. "Press ENTER to begin methods." Press ENTER only after the samples have been loaded into the autosampler and the baseline has stabilized.

17.4.2.5 Figure 18 illustrates the chromatograms for each of the samples as output by the programmed Spectra-Physics integrator. The program used to generate these outputs can be found in the Appendix of this method. Note that actual output is by individual run as illustrated by Figure 19. Most information provided here is optional to the operator.

17.4.3 Calibration of IC - The instrument should be brought to normal conditions with a warm-up time of at least thirty minutes.

17.4.3.1 With the "Reading" light on, check to ensure the flow rate is 1.5 mLs/minute, the fluid pressure is 600 psi ± 100 psi and the conductivity is constant as measured by offset difference.

17.4.3.2 Fill the IC vials with the prepared standard solutions and (10, 5 and 1 ppm Na_2SO_4 and $NaNO_3$) and pure eluent. This will allow a four-point calibration curve to be made.

Note: For low-level applications, more standards and blanks may be necessary in order to obtain accurate reference curves.

17.4.3.3 Load the four vials into the sample vial holder, and place the holder in the automated sampler tray.

17.4.3.4 The tray is controlled by a Spectra-Physics SP4200 or SP4270 Computer Integrator. Use the integrators operation manual to begin calibrating. (A typical program in Basic for integrators which illustrates integrator capability is shown in the Appendix of this procedure). By using the RUN command the analysis and data treatment phases of the calibration are set in motion. Four calibration standards are run, the chromatograms and peak areas displayed for each run, and the run results for each anion are fitted to a quadratic curve by a least squares regression calculation. The three curves are plotted and the correlation coefficients are calculated. The values of the coefficients are normally greater than 0.999, where 1.000 indicates a perfect fit. Values of less than 0.99 indicate the calibration procedure should be repeated.

Note: Recalibration should be carried out whenever standard concentrations show consistently high or low results relative to the calibration curve is compared to the calibration curve from the old standards. Comparability of points should be within ± 0.1 ppm or ± 10%. For standard concentrations of greater than 1 ppm, comparability will normally be within 5% or better. Old standards are assumed correct since they are referenced to the entire historical series of previous standard solutions all of which are comparable.

17.4.4 System Shut-down

17.4.4.1 Shut off the pump. Turn the REGEN switch and the conductivity cell to the OFF position.

17.4.4.2 Switch the eluent degas switch to HIGH.

17.4.4.3 Turn the stripchart recorder OFF, cap the pen. Press the F10 function key on the computer. Select option 3, to exit to DOS. Shut off the printer and the computer.

17.4.4.4 Shut the eluent degas system and reservoir switches and the autosampler to the OFF position. Close the valves on both gas cylinders. Then close the regulator valves.

17.5 Basic Troubleshooting

Before proceeding with the troubleshooting guide, make sure that the reagents used were prepared correctly, and are not "old."

17.5.1 Unstable Baselines

17.5.1.1 Wavy baseline - The most common reason for a wavy baseline is an air bubble in the gradient pump. This is diagnosed by observing the pump head indicator lights on the gradient pump module front panel. If the baseline is pulsing in phases with pump pistons, it usually indicates a bubble. Other possibilities include a dirty or stuck check valve, piston seal or "O"-ring, as well as an air bubble in the conductivity cell.

17.5.1.2 Drifting baseline - Steadily increasing or decreasing baselines usually indicate that the suppressor column is not performing as it should. Parameters to change include the regenerant and eluent concentrations and flow rates. Check temperature routinely as changes in temperature can cause drifting. Balancing these should stabilize the baseline, if the suppressor is functioning correctly. The Dionex manual describes clean-up procedures if the suppressor is believed to be contaminated.

17.5.1.3 High baselines - As with drifting baselines, the parameters to change are eluent and regenerant concentrations and flow rates. A high baseline usually indicates that there is not enough baseline suppression, this can be controlled by increasing the regenerant flow rate.

17.5.1.4 Low baselines - Low baselines usually indicate that there is too much suppression. Oversupression can be controlled by decreasing the flow of the regenerant.

17.5.2 Backpressure - Variations in system backpressure are common and should not raise concern UNLESS the pressure change is greater than 200 psi.

17.5.2.1 High backpressure - The system is protected from pressure related damage through the high and low pressure alarm settings on the front panel of the gradient pump module. If the high pressure setting is correctly selected (200 psi above normal operating range), the pump will automatically shut-off if this value is exceeded. The reason for high backpressure is that there is some kind of blockage in the system. Possibilities include: loading against a closed valve; a plugged line; contaminated columns; etc. Diagnosis of the problem is done by removing one component of the system and observing how the pressure changes.

17.5.2.2 Low pressure - Low pressure readings usually indicate a leak somewhere in the system. Carefully check all fittings for leaks, tighten if necessary.

17.5.3 Flow

17.5.3.1 Regenerant lines - If there is no flow at the waste outlet end of the regenerant line, check the following:

- Make sure that the correct regenerant switch is turned on
- Verify that the reservoir is not empty
- Make sure the nitrogen tank is turned on
- Check that the regulator is correctly set

17.5.3.2 Eluent lines - If there is no flow at the outlet end of the eluent lines check the following:

- Check that the pump is on
- Check that the eluent lines are connected to the correct port

17.5.4 Software - refer to the ChromatoChart manual for detailed information on software problems.

18. Ammonia Analysis By Technicon Autoanalysis

Presented in Sections 18.1 and 18.2 are the recipes for the standards and reagents required for the analysis of the ammonium ion (NH_4^+ - or ammonia (NH_3)) by Technicon autoanalysis. The prelude of these Sections briefly describes the TRAACS 800 autoanalyzer and the sample flow through the TRAACS 800 for NH_4^+ analysis. The Technicon TRAACS 800 autoanalyzer is illustrated in Figure 20. This instrument is capable of quantifying, from a single sample, three different species, simultaneously. An aliquot of the sample is taken from an automated sampler by syringe. A splitter divides the aliquot into the appropriate volumes required for the particular analyses. Each of the volumes is then transferred to the appropriate analytical cartridge. Sample flow diagrams which illustrate $SO_4^=$, NO_3^- and NH_4^+ analysis can be shown separately and independently of one another. Hence, for a one-channel system, one can readily adapt the sample preparation and analysis protocols for each individual analysis. The data computation (by computer) and quality assurance protocols, however, can not be readily adapted to single-channel instruments. These protocols need to be specific to the individual analytical instrument. In brief, for NH_4^+ analysis, Figure 21 illustrates how the sample is carried through the Technicon autoanalyzer. The samples, along with all standards, are taken from the auto-advance sampler tray by the use of a proportioning pump and automated syringe. Air and EDTA are first added to the samples and are mixed in the first set of coils. After mixing, phenolate is added and mixed in the next set of coils. Nitroprusside is then added and mixed, followed by the addition and mixing of hypochlorite. At this stage, the sample should be a bright blue color. After the last mixing stage, the sample is sent through a heated bath, followed by another mixing stage. Finally the sample is sent through a colorimeter where the results are recorded on a digital printer and stored in a computer file for further manipulation.

18.1 Standards and Stock Solutions Preparation

Note: Before discarding the old solution, it should be checked against the fresh solution by comparing calibration curves on the working solutions prepared from them. Slopes and intercepts are calculated for each set of standards. The old slope and intercept are used to calculate concentration values from readings for the new standards. This determines if the old solution has deteriorated or if an error has been made in preparing the new solution.

18.1.1 Ammonium solution standard (1000 µg/mL) - Dry ammonium chloride in an oven for one hour at 50 to 60°C and desiccate over silica gel for one hour. Weigh 2.9470 g ammonium chloride and dissolve in 800 mL DDW. Dilute to one liter with DDW and mix thoroughly. This solution is stable for one year.

18.1.2 Intermediate ammonium standards - To make a 100 µg/mL ammonium standard, pipet 10 mL of ammonium stock standard into a 100 mL volumetric flask. Dilute to volume with DDW and mix thoroughly. Keep refrigerated. This solution remains stable for one month. To make a 10 µg/mL ammonium standard, pipet 1.0 mL of ammonium stock standard into a 100 mL volumetric flask. Dilute to volume with DDW and mix thoroughly. This solution remains stable for one week.

18.1.3 Working ammonium standards in DDW - Pipet aliquots of the 100 µg/mL ammonium intermediate standards with appropriate volumes of nitrate and sulfate intermediate standards into 100 mL volumetric flasks according to the table below. Dilute to volume with DDW. Prepare fresh daily.

Standard	Stock or Intermediate Standard (µg/mL)	Aliquot (mL)	Concentration (µg/mL)
A	1000	40.0	40.0
B	100	4.0	4.0
C	100	3.0	3.0
D	100	2.0	2.0
E	100	1.0	1.0
F	100	0.5	0.5
G	10	2.0	0.2
H	10	1.0	0.1

18.1.4 Sodium citrate stock solution - Dissolve 294.1 g of sodium citrate in 800 mL DDW. Dilute to 1 liter and mix thoroughly. Store at room temperature.

18.1.5 20% citric acid/5% glycerol stock solution - Dissolve 25 g citric acid in 80 mL DDW. Add 5 mL glycerol and dilute to 100 mL with DDW. Mix thoroughly and store at room temperature.

18.1.6 Sodium citrate/citric acid/glycerol working solution - Put 100 mL sodium citrate stock solution into a 1000 mL volumetric flask. Add 20 mL of the 10% citric acid/5% glycerol stock solution and dilute to volume with DDW. Mix thoroughly and store at room temperature.

Note: This solution will be used to make up ammonium working standards for citric acid/glycerol-impregnated filter extract analyses.

18.1.7 Working ammonium standards in sodium citrate/ citric acid/glycerol working solution - Pipet aliquots of the 100 µg/mL volumetric flasks according to the table in Section 18.1.1.3. Dilute to volume with sodium citrate/citric acid/glycerol working solution and mix thoroughly. Prepare fresh daily.

18.1.8 Potassium chloride stock solution - Dissolve 74.6 g potassium chloride in 800 mL DDW. Dilute to one liter with DDW and mix thoroughly. Store at room temperature.

18.1.9 Potassium chloride working solution - Put 100 mL of the potassium chloride stock solution into a 1000 mL volumetric flask. Dilute to volume with DDW.

18.1.10 Working ammonium standards in potassium chloride working solution - Pipet aliquots of the 100 µg/mL ammonium stock standard or intermediate standards into 100 mL volumetric flasks according to the table below. Dilute to volume with potassium chloride working solution and mix thoroughly. Prepare fresh daily.

Standard	Stock or Intermediate Standard (µg/mL)	Aliquot (mL)	Concentration (µg/mL)
A	1000	40.0	40.0
B	100	4.0	4.0
C	100	3.0	3.0
D	100	2.0	2.0
E	100	1.0	1.0
F	100	0.5	0.5
G	10	1.0	0.1
H	10	0.5	0.05

18.2 Reagent Preparation

Note: When reagents are prepared, label the container with the contents, concentration, date prepared, and the preparer's initials.

18.2.1 Alkaline phenol - To 800 mL DDW in a one liter volumetric flask, add 83.0 g loose crystallized phenol. Keeping the flask in anice bath or under tap water, slowly add 96.0 mL 50% sodium hydroxide solution. Shake the flask while adding the sodium hydroxide. Cool to room temperature, dilute to one liter with DDW and mix thoroughly. Store in an amber glass container. This solution remains stable for three months, if kept out of direct light.

18.2.2 Sodium hypochlorite solution - The amount of sodium hypochlorite solution varies from batch to batch of sodium hypochlorite (5% commercial grade). Therefore, for each new batch, a base and gain experiment must be run to adjust the amount of sodium hypochlorite required to obtain the existing base and gain values. In a 150 mL volumetric flask, dilute 86 mL of 5% sodium hypochlorite solution to 100 mL with DDW and mix thoroughly. Check base and gain values. Reduce or increase the amount of sodium hypochlorite to obtain the same base and gain values as the previous sodium hypochlorite batch. This solution remains stable for one day.

18.2.3 Sodium nitroprusside solution - Dissolve 1.1 g of sodium nitroprusside in about 600 mL of DDW, dilute to 1 liter with DDW and mix thoroughly. Store in an amber container, and keep in refrigerator. This solution remains stable for one month, if kept out of direct light.

18.2.4 Disodi&2um EDTA solution - Dissolve 1.0 mL of 50% w/w sodium hydroxide and 41.0 g of disodium EDTA mix thoroughly. Add 3.0 mL of Brij-35 and mix. Store in plastic container. This solution remains stable for six months.

19. pH Analysis

19.1 Standard and Reagent Preparation

19.1.1 Standard H_2SO_4 Solution

Note: Each of the standard H_2SO_4 stock solutions must be prepared fresh the day of pH analysis.

19.1.1.1 Label seven 25 mL polyethylene stoppered volumetric flasks. Also, label each flask with the volume of 1 N H_2SO_4 solution indicated in the following table:

Flask #	Volume of IN Stock (μL)	Standard Concentration (μN)
1	0	0
2	25	1
3	50	2
4	100	4
5	200	8
6	400	16
7	800	32

19.1.1.2 Use the 25 μL automatic pipet to add 1 N stock H_2SO_4 to flasks #1-3. Use the 100 μL pipet to add 1 N stock H_2SO_4 to flasks #4-7. Dilute all flasks to the 25 mL mark with absolute ethanol. Cap with stoppers or parafilm and mix well.

19.1.2 2 M Potassium Chloride (KCl) Solution

19.1.2.1 Weigh 149.2 ± 0.1 g of KCl. Add the KCl to a 2 L flask.

19.1.2.2 Add about 700 mL of DDW water to the flask. Swirl the solution until the KCl is completely dissolved.

19.1.2.3 Pour this mixture into a 1 L graduated cylinder. Rinse the flask with a small amount of water and transfer the rinse into the cylinder. Fill the cylinder to the 1 L mark.

19.1.2.4 Pour the solution from the cylinder into the 1 L polyethylene bottle. Cap and shake the bottle to mix well. Mark the bottle with date of preparation.

19.1.3 0.1 N Perchloric Acid ($HClO_4$) Solution

19.1.3.1 Fill a 1 L graduated cylinder about 1/2 full with DDW. Transfer 10 ± 0.1 mL of 60-62% $HC10_4$ into the 1 L cylinder with a 10 mL pipet.

19.1.3.2 Fill the cylinder to the 1 L mark. Pour the solution into the 1 L polyethylene bottle.

19.1.3.3 Cap and shake the bottle to mix well. Mark the date of preparation on the bottle.

19.1.4 0.01 N $HClO_4$ Solution

19.1.4.1 Fill a 1 L graduated cylinder about 1/2 full with DDW.

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19.1.4.2 Measure 100 mL of the 0.1 N $HClO_4$ solution with the 100 mL graduated cylinder. Add this to the 1 L cylinder.

19.1.4.3 Fill a 1 L cylinder with DDW to the 1 L mark. Pour the solution into the 1 L polyethylene bottle.

19.1.4.4 Cap and shake the bottle to mix well. Mark the date of preparation on the bottle.

19.1.5 Extraction Solution (ES)

Note: This solution has the same composition as the solution used to fill the sample vials for Teflon® filters. It must be prepared fresh on the day of pH analysis.

19.1.5.1 Measure 100 ± 10 mL of DDW into a 1 L graduated cylinder. Transfer to a 2 L erlenmeyer flask.

19.1.5.2 Using a 5 mL calibrated automatic pipet, add 10 ± 0.1 mL of 0.01 N perchloric acid ($HClO_4$), to flask of water.

19.1.5.3 Mix well and cover with parafilm until ready for use.

19.1.6 EA Solution

19.1.6.1 Measure 150 ± 2 mL of ES (prepared in 18.1.5) into a 250 mL graduated cylinder. Transfer to a 250 mL erlenmeyer flask.

19.1.6.2 Using a 5 mL graduated cylinder, add 5 ± 0.1 mL of ethanol (this must be from the same fresh bottle of ethanol that was used to prepare the standards in 18.1.1) to the flask.

19.1.6.3 Again using a 5 mL graduated cylinder, add 3 ± 0.1 mL of 2 M potassium chloride (KCl) solution to the flask.

19.1.6.4 Mix well and cover with parafilm until ready for use.

19.1.7 Working Standard Test Solutions

19.1.7.1 Place fourteen-4 mL polystyrene sample cups (as used with Technicon Auto-Analyzer II system) labeled 1, 1*, 2, 2*...7, 7* into racks. Using the calibrated dispensing pipet bottle, add 3 mL of ES solution to each 4 mL cup.

19.1.7.2 Using the displacement pipet, add 50 uL of absolute ethanol to each cup. Pour about 3 mL of standard (H_2SO_4 solution) #1 into a labeled 4 mL cup.

19.1.7.3 Immediately, pipet 50 uL of this standard into the 4 mL cups labeled 1 and 1* containing the ES solution and ethanol.

Note: This transfer must be done without delay to prevent the standard concentration from increasing significantly due to evaporation of the ethanol solvent.

19.1.7.4 Repeat the procedure for each of the other 6 standards. If there is a delay of more than 5 minutes between the preparation of these mixtures, and the next step, put caps on the 4 mL cups.

19.1.7.5 To prepare for analysis, each must be mixed, then two aliquots from each cup are transferred to 2 mL sample cups. Place cup #1 in a rack. In a second rack place two-2 mL cups labeled 1 and 1-. Use the 1 mL automatic pipet to mix the contents of 4 mL cup #1 by drawing 1 mL into the pipet tip and then dispensing it back into the 4 mL

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cup three times. Then use the same pipet to transfer 1 mL of the mixture to each of the two labeled 2 mL cups. Place [illegible]on the two 2 mL cups. After transferring the two aliquots to 2 mL cups, rinse the automatic pipet tip in a flask of DDW. Repeat the transfer procedure for each of the other working standard pairs.

19.2 Calibration of pH Meter

The pH meter requires temperature calibration whenever a new electrode is used. Use the manufacture's procedure in the instrument manual. This calibration should be repeated every three months while not in use. The pH meter is left with the power cord plugged into the AC outlet, the mode control knob is left in the standby position, the electrode lead is partially disconnected by pressing the plastic ring on its outer edge, and the combination electrode is immersed in a 4 M KCl solution (a slit rubber stopper seals the bottle with the electrode in it). Keep a record of the temperature calibrations in a lab notebook.

19.3 Pre-Analysis Calibration

19.3.1 Use pH lab analysis log form 418 to record all date. While still in standby mode, reconnect the electrode lead at the back of the pH meter.

19.3.2 Fill three 4 mL cups with pH 7 buffer. Withdraw the electrode from the 4 M KCl bottle and wipe the tip gently with a Kimwipe® to remove the bulk of the solution. Rinse the electrode with one cup of pH 7 buffer. Do not test pH of the first cup.

19.3.3 Immerse the electrode in the second cup of the pH 7 buffer. Use a small bottle or other support to hold the cup up to the electrode while waiting for the meter reading to equilibrate.

19.3.4 Test the pH by turning to the pH mode of the meter. Allow the reading to stabilize for at least 30 seconds. Record the result on the log for "1st cup."

19.3.5 Turn to standby mode, and then test the last cup of pH 7 buffer. Record the results on the log for the "2nd cup." If the pH value for the 2nd cup is not 7.00 ± 0.01, adjust the "calib." knob to obtain a reading of 7.00. Note this adjustment on the log.

19.3.6 Fill three 4 mL cups with pH 4 buffer. With the meter in the standby mode, remove the cup containing pH 7 buffer, wipe the tip of the electrode gently with a Kimwipe®, and then rinse the electrode with the first cup of pH 4 buffer.

19.3.7 Test the next two cups of pH 4 buffer as above, recording the results on the log. If the pH value for the 2nd cup is not 4.00 ± 0.01, adjust the "slope" knob to get a reading of 4.00. If the value for the second cup was not 4.00 ± 0.03, the calibrations at pH 7 and at pH 4 must both be repeated.

19.4 pH Test 0.01 N $HClO_4$ Solution

Note: The 0.01 N $HClO_4$ solution is used to prepare the ES solution which, in turn, is used to prepare the EA solution. It is imperative that the pH value for the EA solution be 4.09 ± 0.04. If this pH value is not achieved, then the 0.01 N $HClO_4$ solution must be reprepared.

19.4.1 Calibrate the pH meter with pH 4 buffer.

19.4.2 Rinse the pH electrode with DDW. Wipe the tip of the electrode with a Kimwipe®.

19.4.3 Fill three 4 mL cups with EA solution. Measure the pH of the test EA solution as with the buffer solutions this value must be 4.09 ± 0.04.

19.4.4 If the above pH value is not achieved, follow the steps 18.1.3 - 18.1.6 to reprepare the solutions. Test the pH of the new solutions. Repeat as necessary to obtain a pH of 4.09 ± 0.04.

19.4.5 Leave the electrode immersed in the "2nd cup" with the meter in the standby mode until ready to start analysis of the working standards.

19.5 Analysis of Working Standard

Note: Immediately following the EA analysis, start testing the working standards.

19.5.1 With the pH meter still in the standby mode, remove the last cup from the electrode, gently wipe the tip with a Kimwipe®, and then immerse the electrode into the working standard cup #1.

Note: Only two cups are available for each working standard (also for filter extracts). Thus, pH measurement is made for both of the two cups for each sample. Also, the electrode tip is not wiped between the 1st and 2nd cups of each sample.

19.5.2 After testing the pH of cup #1, test cup #1-. Record the results of both on the log sheet.

19.5.3 With the meter in the stand-by mode, remove the #1- sample cup, wipe the electrode with a Kimwipe® and test one 2 mL cup of EA solution, rinse with DDW.

19.5.4 Test a 2nd cup of EA solution; record the results for both cups on the logsheet. Discard the 1st cup of EA, but retain the 2nd cup to be used as the 1st cup for the next EA test.

19.5.5 Continue testing the remainder of the working standards, #1*, 1*-, ... 7, 7-, 7*, 7*-. Remember that the electrode tip is wiped both before and after each pair of test solutions, but not in between two cups of the same sample.

Note: If there is trouble in obtaining constant pH values, it may be necessary to use a magnetic stirrer to keep the contents to be measured uniform. If employed, ensure that the sample cups are insulated from any temperature increase of the stirring platform which may occur during extended use.

19.5.6 Use the mode control knob in the "temp." position to measure the temperature of the test solutions every 5-10 samples and record the results on the logsheet.

19.6 Analysis of Filter Extracts

Following measurement of the pH of the working standards, measure the pH of the filter extracts and record all results on the log. After all the filter extracts have been tested make an additional test with the EA solution. At the end make a final test of pH 4 buffer. With the mode control in the standby mode, shut down the pH meter by disconnecting the electrode lead at the back of the meter, leaving the meter power cord plugged into the AC line. Immerse the electrode tip in the bottle of 4 M KCl.

20. Atmospheric Species Concentration Calculations

The system described in the previous sections collects nitric acid (HNO_3), nitrous acid (HNO_2), sulfur dioxide (SO_2), ammonia (NH_3) particulate sulfate ($SO_4^=$), and particulate nitrate (NO_3^-). Figure 1 illustrates the collection of each of these species. Nitric acid and sulfur dioxide gases are collected on denuders one and two. Some SO_2 gas is collected on denuder three also. Nitrous acid gas is collected on denuders two and three. Ammonia gas is collected on denuder four. Particulate sulfate and nitrate are collected on the first (Teflon)® filter, while some of the particulate nitrate collected on the Teflon® filter can evaporate and be collected on the second (nylon) filter. Also collected on the Teflon® filter are fine particles which contain hydrogen ions (H^+), though probably not free H^+. Hydrogen ions are most likely present in the H_3O^+ form. The concentration of these H^+ ions indicates the atmospheres acid aerosol content. It is necessary to prepare the Teflon® filter extracts for pH analysis prior to IC analysis for the particulate sulfate contents. Special precautions must be taken to prevent contamination of the Teflon® filters by ammonia before either of the analyses.

20.1 Assumptions of the Annular Denuder System

There are a number of assumptions which are made about performance of the annular denuder system in order for validity of the calculations to be presented later in this section to hold true. As discussed in Section 6, there are significant interferences which need to be considered in order for accurate estimations of species concentrations to be made. The assumptions are as follows:

- The first denuder stage collects 100% of sampled HNO_3 as nitrate. (Since the diffusivity of HNO_3 is high, diffusion to the side walls is assumed to be very quick.)
- The second denuder stage collects 100% of sampled HNO_2 as nitrite, which can oxidize to nitrate.
- The first and second denuder stages together collect 100% of the SO_2 as sulfite, which can oxidize to sulfate.
  Note: Before analysis, it is recommended to add hydrogen peroxide (H_2O_2) to oxidize the sulfite (SO_3^-) to sulfate ($SO_4^=$) to simplify the calculations.
- The amounts of nitrite and nitrate collected on denuder 3 (d3) represent amounts of interfering gases such as NO_2 collected on denuder 2 (d2).
- The fourth denuder stage collects 100% of the sampled ammonia (NH_3) as ammonium ion (NH_4^+).
- The Teflon® filter (f1) is 100% efficient for particulate sulfate, nitrate and ammonia. Particle losses are less than 1% on each denuder. This assumption may or may not stand true depending on the concentrations of the components in the air sampled. Modifications may be needed to avoid low (or underestimates of) acidic measurements. For example, it may be necessary to add another filter stage to more accurately account for the particulate ammonia content of the air sampled. If ammonium nitrate (NH_4NO_3) was collected on the Teflon® filter, its probability of evaporation is high. Therefore, a citric acid-impregnated filter downstream would correct for the loss from the Teflon® filter. Also, interaction of ammonia and sulfuric acid neutralizes the filter

and causes the acidic measurement to be biased. (Again diffusion rules the particle loss assumption; particles have lower diffusivities than gases).

- The nylon filter (f2) collects any nitrate that evaporates from the Teflon® filter (f1).

20.2 Calculations Using Results from IC Analysis

These assumptions lead directly to equations for computing atmospheric concentrations from denuder measurements.

20.2.1 Figure 22 illustrates the equation for nitric acid quantification. In this equation, $C_g(HNO_3)$ is the concentration of nitric acid gas expressed in $\mu g/m^3$. Subscript g denotes "gas." The computation depends on NO_3^- (d1), which is the measured amount nitrate in μg collected on denuder 1. The factor 1.016 represents the ratio of molecular weights of HNO_3 and NO_3^-. In the denominator, V is the sampled air volume expressed in m^3.

20.2.2 Figure 23 illustrates how the concentration of nitrous acid is deduced. The numerical factors 1.022 and 0.758 (both in μg) are used to convert the measured nitrite and nitrate to equivalent amounts of nitrous acid. Measured nitrate has to be included because some of the collected nitrite may oxidize to nitrate during sampling or during sample storage. Because a small portion of NO_2 may be collected on denuders 2 (d2) and 3 (d3), the nitrite and nitrate amounts measured on denuder 3 (d3) represent corrections for NO_2 and other interfering gases.

20.2.3 Figure 24 illustrates how sulfur dioxide concentrations are deduced. Because sulfur dioxide is collected on both stages d1 and d2, the results for both stages are added. To simplify the calculation, oxidize the collected sulfite to sulfate by adding H_2O_2 to the sample vial. Hence, the quantification of $SO_4^=$ gas directly estimates sulfur dioxide. A more complicated equation would result if the collected sulfite had not been fully oxided to sulfate. Sulfate measurements are expressed in mg. Sulfur dioxide concentrations are expressed in mg/m^3.

20.2.4 Figure 25 illustrates the equation for ammonia quantification. The numerical factor 0.944 is used to convert the measured ammonium ion to its equivalent amount of ammonia. Therefore the product of the factor and the NH_4^+ collected by d4 directly estimates the ammonia concentration ($C_g(NH_3)$).

20.2.5 Figure 26 illustrates how particulate sulfate concentration ($C_p(SO_4^=)$) is computed. The subscript p denotes "particle." This formula expresses the assumption that essentially all of the particulate sulfate is collected on the Teflon® filter (f1), and no evaporation occurs.

20.2.6 Figure 27 shows how particulate ammonium concentration is computed. This formula expresses the assumption that essentially all of the particulate ammonia is collected on the Teflon® filter (f1), and no evaporation occurs.

20.2.7 Figure 28 illustrates how the particulate nitrate concentration is computed. This equation is similar to the one for sulfate except that nitrate measured on the nylon filter (f2) must be included because nitrate collected on the Teflon® filter (f1) can evaporate. Note: It is important to note that four of the measurements are not used. For example, sulfate measured on the nylon filter represents a sulfate blank for nylon that is irrelevant to sulfate collected on Teflon®. Also, nitrite collected on the nylon filter represents the

possibility that some NO_2 is collected on the nylon filter, but that is not relevant to the way that nitrate is determined in the denuder system. The remaining unused data represent low concentrations and are also not relevant to deducing the concentrations of the atmospheric species considered here.

20.3 Estimates of Errors In Concentrations Deduced From Denuder Data

Note: The assumptions and formulas used to calculate the uncertainty of the measurements are illustrated in Section 20.3.3.

20.3.1 Figure 29 shows the formula used for the uncertainty in particulate sulfate. It includes errors in measuring sulfate and in deducing the air-volume sampled. It also includes a 3% error to account for the possibility of 1% particle loss in each of the three denuder stages. Error equations for the other species are shown in Section 20.3.3.

20.3.2 Assumptions on which error equations are based:

- X is the measurement error for species X.
- Measurement errors are random and uncorrelated among species.
- Possible particle-losses of 1% in each denuder introduces an overall uncertainty of + 3% for particulate sulfate and nitrate concentrations.
- Gases such as H_2S and CH_3HS can be collected on the denuder stages, and bias the results. Amounts collected on denuder stage 3 can be used to estimate the uncertainties that result from such bias. Thus, $SO_4^{=}(d3)$ is an estimate of the uncertainty in the amount of SO_2 collected on denuders 1 (d1) or 2 (d2).

20.3.3 Error equations:

For $SO_4^{=}$:

$$[\delta C_p(SO_4^{=})/C_p(SO_4^{=})]^2 = [\delta SO_4^{=}+(f1)/SO_4^{=}+(f1)]^2 + [0.03]^2 + [\delta V/V]^2$$

For NO_3^-:

$$[\delta C_p(NO_3^-)/C_p(NO_3^-)]^2 = [NO_3^-(f1) + NO_3^-(f2)] + [0.03]^2 + [\delta V/V]^2$$

For HNO_3 and HNO_2:

$$[\delta C_g(HNO_3)/C_g(HNO_3)]^2 = [\delta NO_2^-(d1)/NO_3^-(d1)]^2 + [\delta V/V]^2$$

$$[\delta C_g(HNO_2)/C_g(HNO_2)]^2 = [\delta A/(VC_g(HNO_2)]^2 + [\delta V/V]^2$$

where:

$$A^2 = (1.022)^2 [\delta NO_2^-(d2)^2 + NO_2^-(d3)^2] + (0.758)^2 [\delta NO_3^-(d2)^2 + NO_3^-(d3)^2]$$

20.4 Calculations Using Results from pH Analysis

Earlier determinations of pH have been based on the pH buffer concentrations, the activity of the solution, and the antilog of the measured pH value. More recent studies have steered away from the issue of activity by comparing the results of the standards, thus, alleviating errors introduced by basing the activities of ions retained on filters on those

retained in solution. The methodology developed from these more recent studies is described herein. The end results are reported in terms of mass of equivalent of ions. Appropriate values of accuracy and precision with respect to H^+ concentration for this method are 10% and 5%, respectively, for sample pH values in the 4.00 to 7.00 range.

20.4.1 Summary of method - There are two parts to this methodology, determination of the "nominal EQ," and determination of the "actual (EQ_N)." The nominal EQ is defined as the equivalent $\mu g\ H_2SO_4/m^3$ for a nominal 5.76 m^3 sample volume (24 hours at 4 LPM). The actual EQ_A is defined as the equivalent $\mu g\ H_2SO_4/m^3$ based on the actual sample air volume.

20.4.1.1 Determine the nominal EQ_N as follows:

20.4.1.1.1 To account for the difference between standards prepared with filters and standards prepared without filters, adjust the measured concentration values for the working standards (without filters) for each analysis day.

20.4.1.1.2 Calculate the standard curve, using a linear regression of the equivalent of $\mu g\ H_2SO_4/m^3$ (for 5.76 m^3 volume of sample) for each working standard vs the adjusted concentration values for the working standards.

20.4.1.1.3 Use the standard curve to determine EQ_N for each sample filter.

20.4.1.1.4 Calculate the actual air flow rate to determine the actual air sample volume. Divide the actual air sample volume into EQ_N to determine EQ_A.

20.4.1.2 Determine the actual EQ_A as follows:

20.4.1.2.1 The actual sample air volume, V, for each sample is calculated using data from the field log sheet. This data includes the initial and final elapsed time, the initial rotameter reading, and the rotameter I.D. No.

20.4.1.2.2 The calibration curve for the given rotameter reading is used to calculate the flow for the sample (LPM).

20.4.1.2.3 The nominal EQ_N is divided by the calculated flow to give the actual EQ_A.

20.4.2 Adjustment for filter vs. non-filter standards - This adjustment is necessary because experiments showed that the measured acid concentration from filters doped with H_2SO_4 stock standards yielded concentrations, as measured by the difference from EA solution, which were about 3% lower than the values found for working standards (prepared without filters from the same stock standards). The results gave the following relation (by linear regression):

$$C_f = -0.11 + 0.971\ (C_{nf}) \qquad (1)$$

where:

C_f = difference in units of 10^{-5} N, calculated using the pH of each filter standard and the pH of EA tested after that standard

C_{nf} = the same difference for non-filter standards (or the apparent net (strong acid concentration of H_2SO_4)

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For each working standard (non-filter), on a given analysis day, calculate the "apparent net concentration of H_2SO_4" as follows:

$$C_{nf} = 10^{-pHWS} - 10^{-pHEA} \quad (2)$$

where:
pHWS = measured pH for a working standard (or apparent strong acid concentration for H_2SO_4 - doped filter standards)
pHEA = measured pH for the EA solution (or apparent strong acid concentration for non-filter, non-H_2SO_4 doped standards)

After calculating the C_{nf} values for each working standard, use equation (1) above to calculate the adjusted values of Cf for each.

20.4.3 Determination of standard curve - For each working standard, the corresponding EQ_N value (the equivalent of μg H_2SO_4/m^3 [assuming a sample volume of 5.76 m^3]) is determined as follows:

$$EQ_N = m/5.76\ (10^6\ \mu g)/g \quad (3)$$

Note: 5.76 is the volume for a sample collected for 24 hours at 4 LPM, in m^3.
Note: It is the analyst's preference as to whether concentration or mass is calculated here and used to create the standard curve. If mass is used, a nominal sample air volume is not necessary. The value of m is determined as follows:

$$m = [1.000]\ [S/25]\ [5 \times 10^{-5}]\ [49] \quad (4)$$

where:
1.000 = concentration of the commercial standard H_2SO_4, in units of equivalents/L
S = volume of commercial standard H_2SO_4 used to prepare a given stock standard solution, mL
25 = volume of each stock standard solution, mL
5×10^{-5} (50 uL) = is the volume of each stock standard solution used to prepare its respective working standard, L
49 = equivalent weight of H_2SO_4, units of grams/equivalent
Note: When the value of S is 1 mL or greater for a final volume of 25 mL, the standard curve illustrates non-linearity. This is due to incomplete dissociation of bisulfate. An example table of the values of the nominal EQ_N for each working standard is shown in Table 3. For each analysis day, the standard curve should be determined by calculating the linear regression of EQ_N vs. C_f, with the result in the following equation:

$$EQ_N = \text{intercept} + [C_f]\ [\text{slope}] \quad (5)$$

20.4.4 Determination of nominal EQ_N for filter samples - The apparent net strong acid concentration of each sample filter extract, C_s, is calculated as with the working standards:

$$C_s = 10^{-pHS} - 10\text{-}pHEA \quad (6)$$

where:
pHS = measured pH of the sample filter extract (or apparent strong acid concentration for sample filters extracts)
pHEA = measured pH for the EA solution (or apparent strong acid concentration for non-filter, non-H_2SO_4 standards)

Note: The C_s values for the filter extracts are directly comparable to the C_f values for the working standards, since the C_f values have been adjusted for the difference in apparent acid concentration for tests made with filters and tests made without filters. Therefore, to determine the nominal EQ_N values for filter samples, use equation (5) transformed as follows:

$$EQ_N = \text{Intercept} + [C_s]\,[\text{Slope}] \quad (7)$$

20.4.5 Determination of actual EQ_A - The actual sample air value, V, for each sample is calculated using the data from the field log sheet. These data includes the initial and final elapsed times, the initial rotameter reading, and the rotameter I.D. No. Use the calibration curve for the given rotameter to calculate the flow for the sample, in LPM. Calculate the value of V as follows:

$$V = [F][T] \quad (8)$$

where:
F = flow from the calibration curve, LPM
T = net elapsed time, min

Since the nominal EQ_N values were determined assuming a flow of exactly 4 LPM and a net elapsed time of exactly 24 hours, the assumed volume was 5.76 m^3, therefore, calculate the value of the "actual EQ_A" by:

$$EQA = [EQ_N]/V \quad (9)$$

where:
EQ_A = units of $\mu g/m^3$

Nominal EQ_N as determined by Equations 3 and 4:

$$EQ_N = m/5.76\ (10^6\ \mu g/g)$$

where:
m = [1.000] [S/25] [5 x 10^{-5}] [49]
1.000 = concentration of commercial standard H_2SO_4, units of equivalents/L
S = volume of commercial standard H_2SO_4 used to prepare a given stock standard solution, mL
25 = volume of each stock standard solution, mL
5 x 10^{-5} (50 uL) = volume of each stock standard solution used to prepare its respective working standard, L
49 = equivalent weight of H_2SO_4, units of grams/equivalent

Working Standard #	S (mL)	M (g)	EQ_{N_3} ($\mu g/m^3$)
1	0.000	0	0.00
2	0.025	2.45	0.43
3	0.050	4.9	0.85
4	0.100	9.8	1.70
5	0.200	19.6	3.40
6	0.400	39.2	6.81
7	0.800	78.4	13.61
8	1.600	156.8	27.22

21. Variations of Annular Denuder System Usage

As mentioned in Section 3 and Section 4, the ADS as described previously, is used to measure reactive acidic (SO_2, HNO_2 and HNO_3) and basic (NH_3) gases and particles found in indoor air. The unique features of the ADS which separates it from established air monitoring methods are the ability of sampling artifacts to be eliminated from the collected gases and particles, and the preservation of the samples for subsequent analysis which is accomplished by removing NH_3 in the gas stream with a citric acid coated denuder, thus reducing the probability of the particulate acid sulfates ($SO_4^=$) captured on the Teflon® filter from being neutralized. The ADS configuration described in Section 13 clearly illustrates these unique features. The elutriator is designed to allow only particles with <2.5 μm diameter into the system. The impactor is designed to reduce the possibility of coarse particle infiltration even further. And finally, the sequence of the denuders reduces interference of possible chemical reactions which could cause under-or over-estimations of concentrations to be made. Although this configuration is recommended for measuring these gases and particulates, it may be in the interest of the user to measure only one or two of the chemical species. The following discussion will present possible variations of the ADS to accommodate such usages.

21.1 Today, the ADS is being used in intercomparison studies to assess NH_3 concentration differences indoors and outdoors. The assembly used here consists of an elutriator-impactor assembly, an annular denuder and a filter pack assembly. The elutriator-impactor assembly and the annular denuder are both smaller than those described earlier. The filter pack is available in the smaller size, but an adaptor is also available to assemble the smaller annular denuder to the larger filter pack assembly. This system is referred to as the personal sampler (see Figure 30). It is designed for sampling while attached to the shirt of a worker. The personal sampler can be used to measure other chemical species in indoor air by simply changing the reactive surface (coating) of the annular denuder and or by changing the types of filters used.

21.2 Another variation of ADS application is simultaneous use in parallel with a fine particle sampler. The fine particle sampler assembly is very similar to the annular denuder assembly. The main difference is that a flow-straightener tube replaces the annular denuder. The flow-straightener is a shorter version, 1-1/4 to 4 inches long, of the annular

denuder and serves to create even air flow across the filters for the collection of particulate matter. Figure 31 illustrates an exploded view of the fine particle sampler. Again the elutriator-impactor assembly and flow-straightener are available in smaller sizes with accommodating filter pack assemblies. In addition, the ADS carrying and shipping cases as well as the sampling box can be adjusted to accommodate the ADS and fine particle sampler. Figure 32 illustrates the assemblies as they would appear in the sampling box ready for sampling.

22. Method Safety

This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the user's responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

23. Performance Criteria and Quality Assurance (QA)

Required quality assurance measures and guidance concerning performance criteria that should be achieved within each laboratory are summarized and provided in the following section.

23.1 Standard Operating Procedures (SOPs)

23.1.1 SOPs should be generated by the users to describe and document the following activities in their laboratory: 1) assembly, calibration, leak check, and operation of the specific sampling system and equipment used; 2) preparation, storage, shipment, and handling of the sampler system; 3) purchase, certification, and transport of standard reference materials; and 4) all aspects of data recording and processing, including lists of computer hardware and software used.

23.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the personnel conducting the monitoring work.

23.2 Quality Assurance Program

The user should develop, implement, and maintain a quality assurance program to ensure that the sampling system is operating properly and collecting accurate data. Established calibration, operation, and maintenance procedures should be conducted on a regularly scheduled basis and should be part of the quality assurance program. Calibration procedures provided in Sections 17 and 19, operation procedures in Sections 14 and 17, and maintenance proeedures in Section 17 of this method and the manufacturer's instruction manual should be followed and included in the QA program. Additional QA measures (e.g., trouble shooting) as well as further guidance in maintaining the sampling system are provided by the manufacturer. For detailed guidance in setting up a quality assurance program, the user is referred to the code of Federal Regulations (12) and the EPA Handbook on Quality Assurance (13).

24. References

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12. 40 CFR Part 58, Appendix A, B.

13. *Quality Assurance Handbook for Air Pollution Measurement Systems*, Volume II - Ambient Air Specific Methods, EPA 600/4-77-0272, May, 1972.

Table 1. Estimated Detection and Quantification Limits for the Annular Denuder System[1]

Detection Limits ($\mu g/m^3$)	Sampling Period		
	1 hour	1 day	1 week
a) gaseous species			
SO_2	3.1	0.13	0.02
HNO_3	2.0	0.08	0.01
HONO	0.5	0.02	0.01
NH_3	5.6	0.25	0.04
b) particulate			
$SO_4^=$	1.6	0.07	0.01
NO^{-3}	1.8	0.08	0.01

Quantification Limits ($\mu g/m^3$)	Sampling Period		
	1 hour	1 day	1 week
a) gaseous species			
SO_2	10.4	0.43	0.06
HNO_3	6.8	0.28	0.04
HONO	1.6	0.07	0.01
NH_3	20.0	0.83	0.12
b) particulate			
$SO_4^=$	5.3	0.22	0.03
NO_3^-	6.1	0.25	0.04

[1]Samples analyzed by ion chromatography. Detection limits are taken as three standards deviations above field blanks. Quantification limits are taken as ten standard deviations above field blanks. Both the detection and quantification limits were estimated assuming that the variance is independent of concentration.

Table 2. Accelerator Jet Diameters and Corresponding Reynolds Number (Re) for Selected Flow Rates to Obtain 2.5 μM Aerodynamic D_{50} Separation

Flow Rate L/min	Jet Diameter	Re
1.0	1.55	900
2.0	1.97	1400
5.0	2.65	2700
10.0	3.33	4200
12.0	3.55	4700
15.0	3.85	5500
16.7	4.00	6000
20.0	4.25	6600

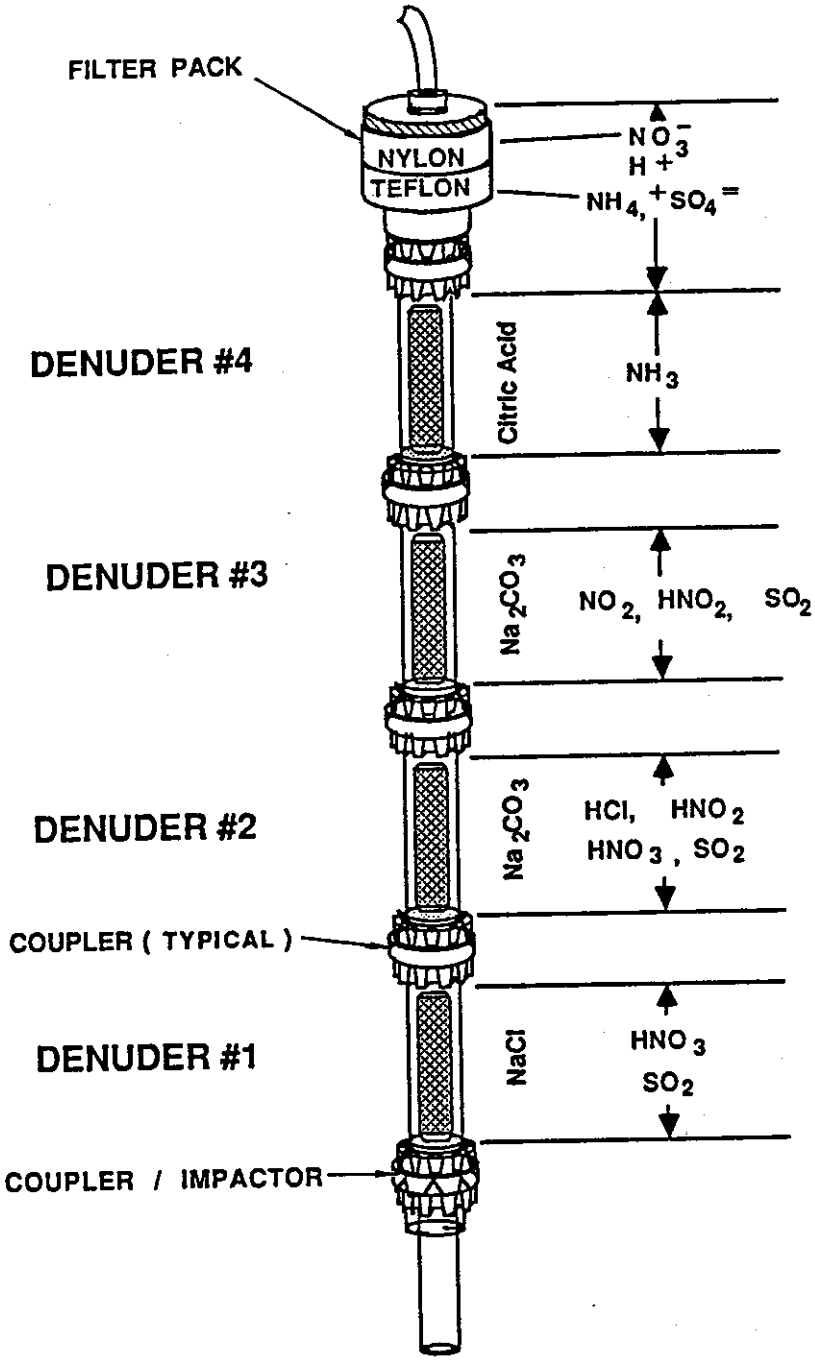

Figure 1. Schematic View of Annular Denuder Showing Species Collected

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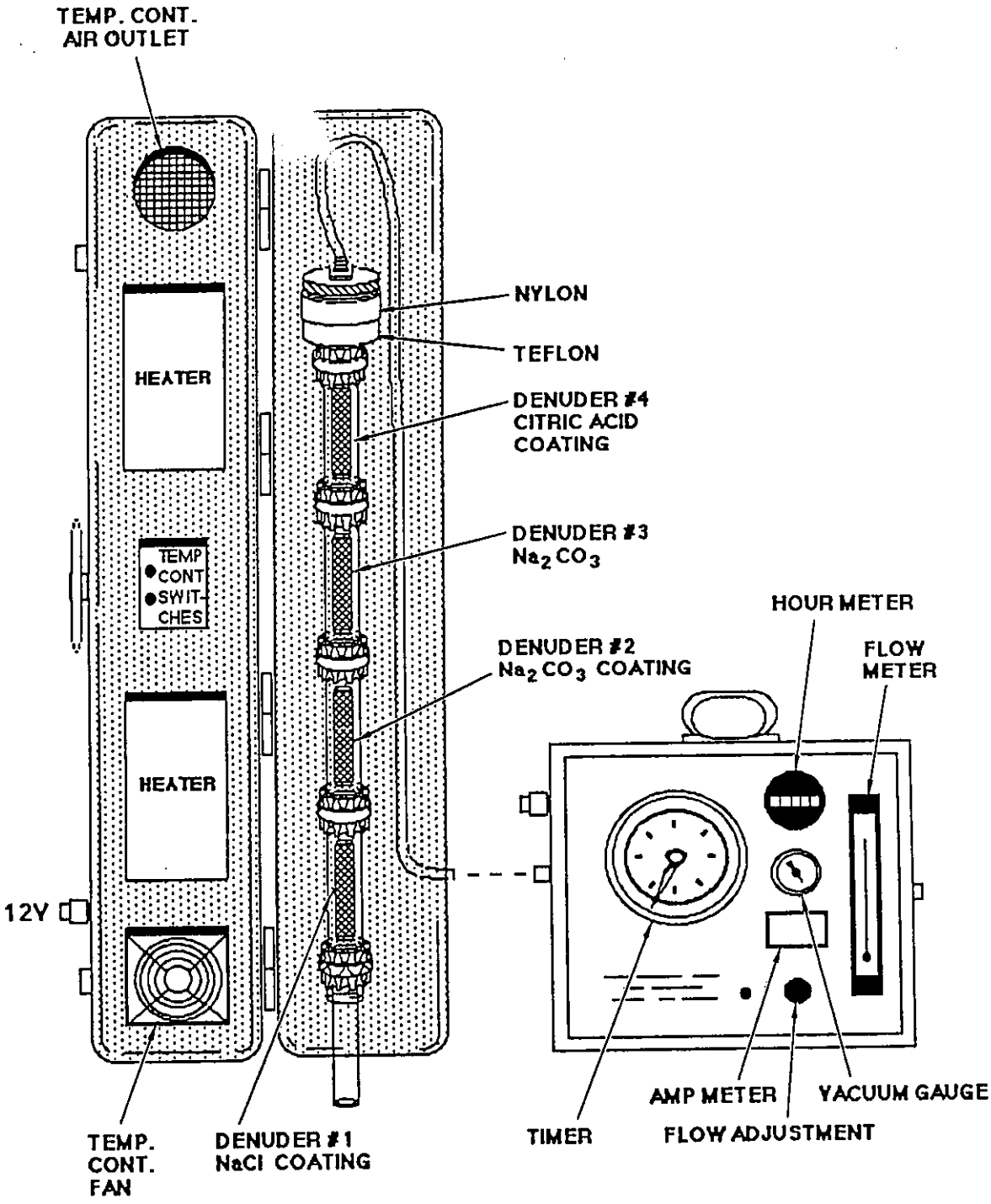


Figure 2. Annular Denuder System

726

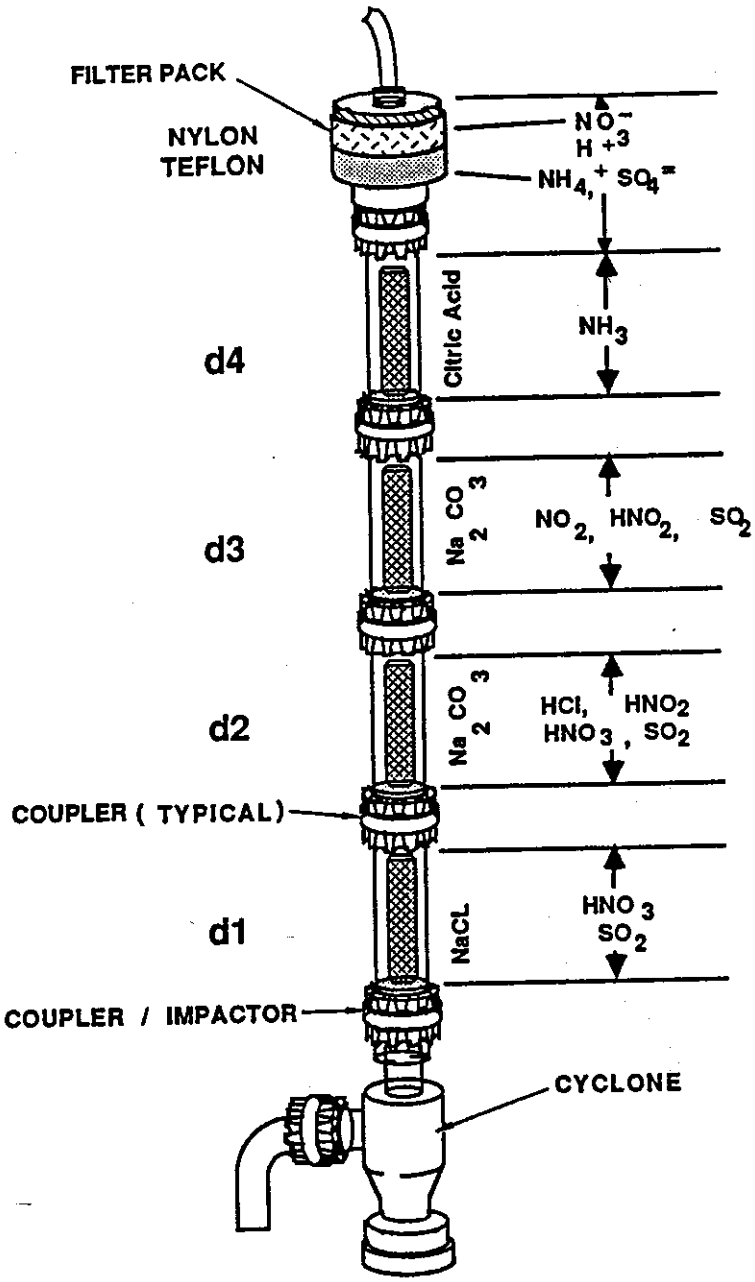


Figure 3. Schematic View of Annular Denuder with Cyclone Adaptor for Removal of Coarse Particles

727

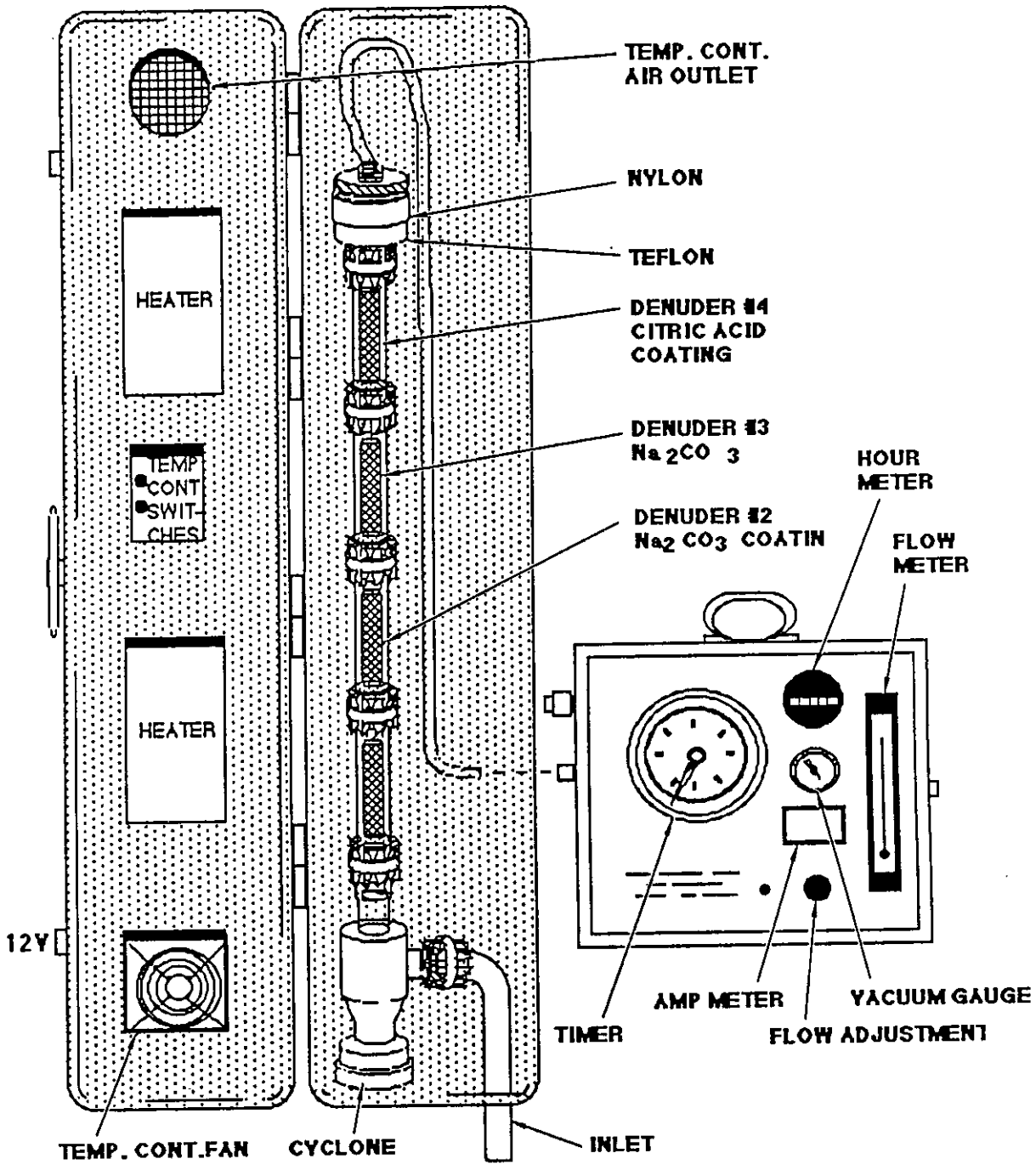


Figure 4. Annular Denuder System with Cyclone in Heated Sampling Case

728

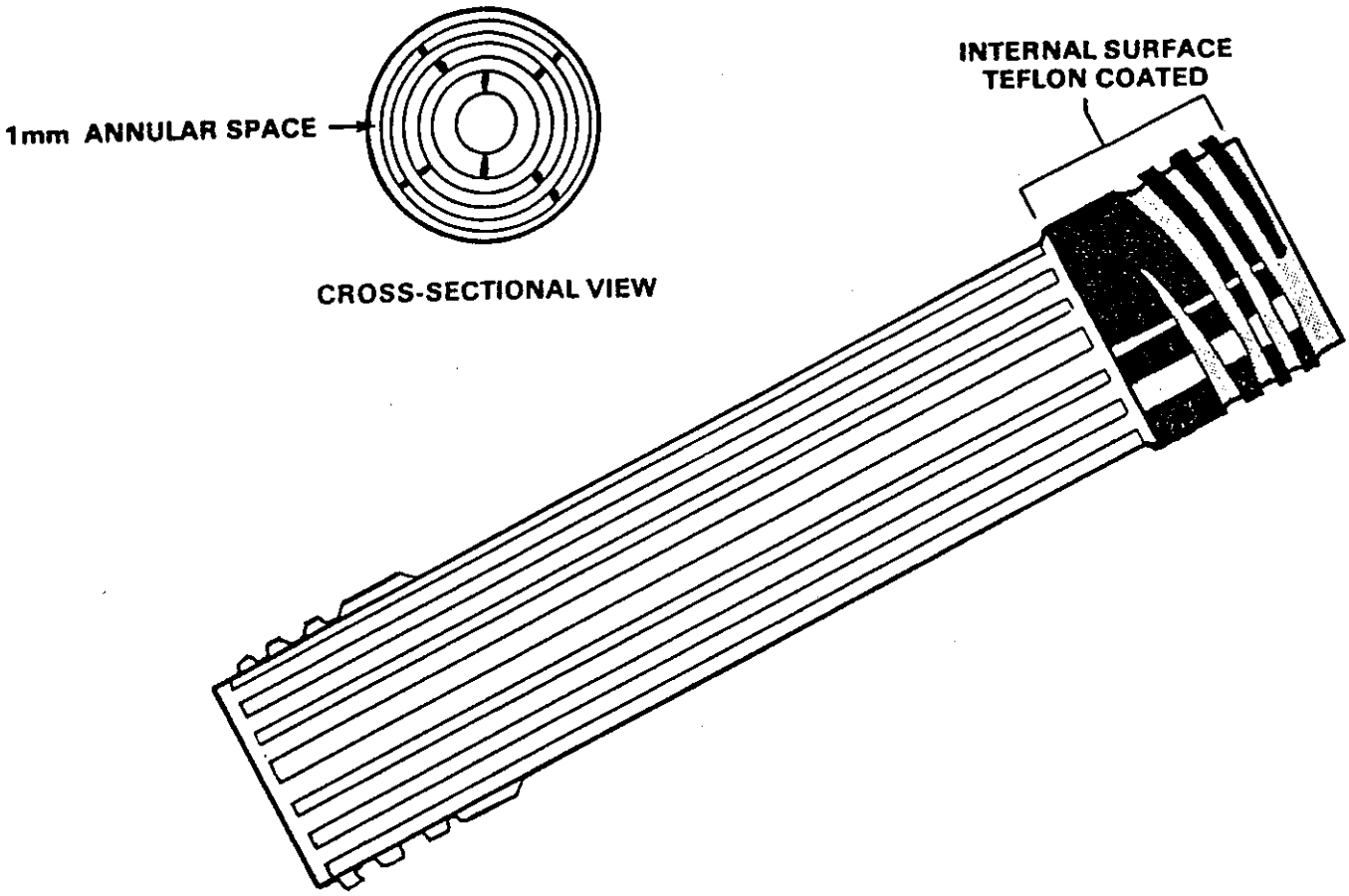


Figure 5. Internal Schematic of Annular Denuder

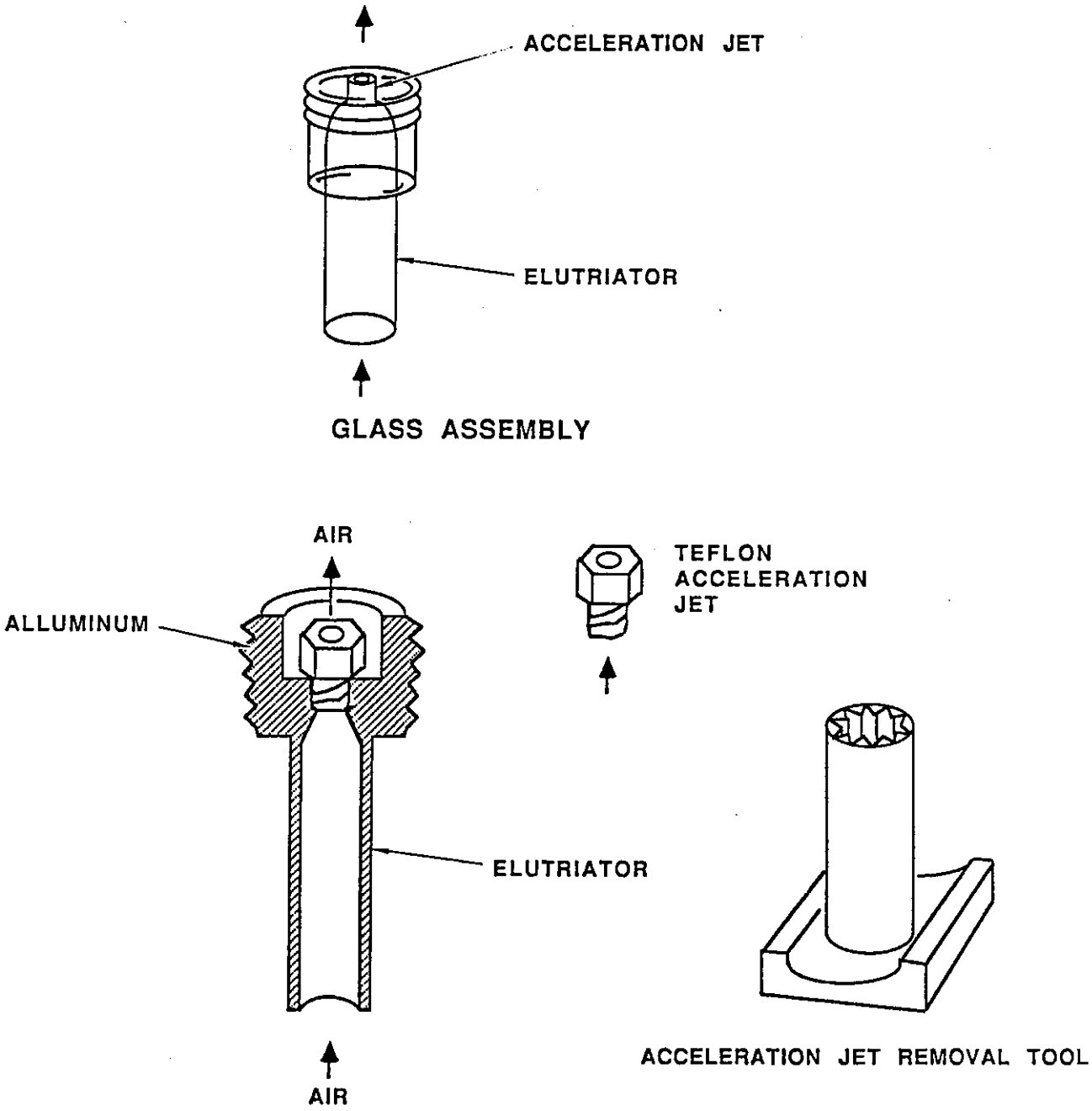


Figure 6. Available Elutriator and Acceleration Jet Assemblies

730

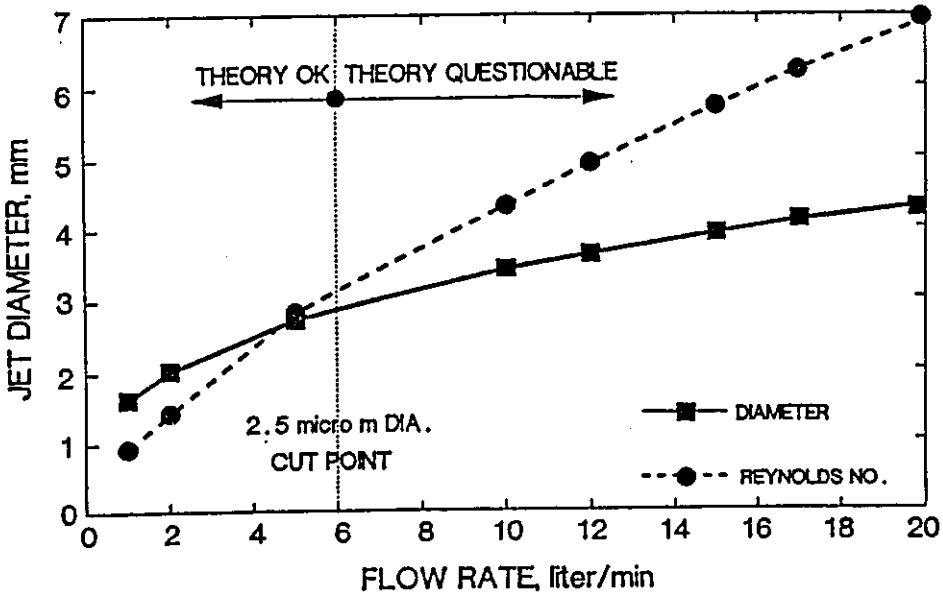


Figure 7A

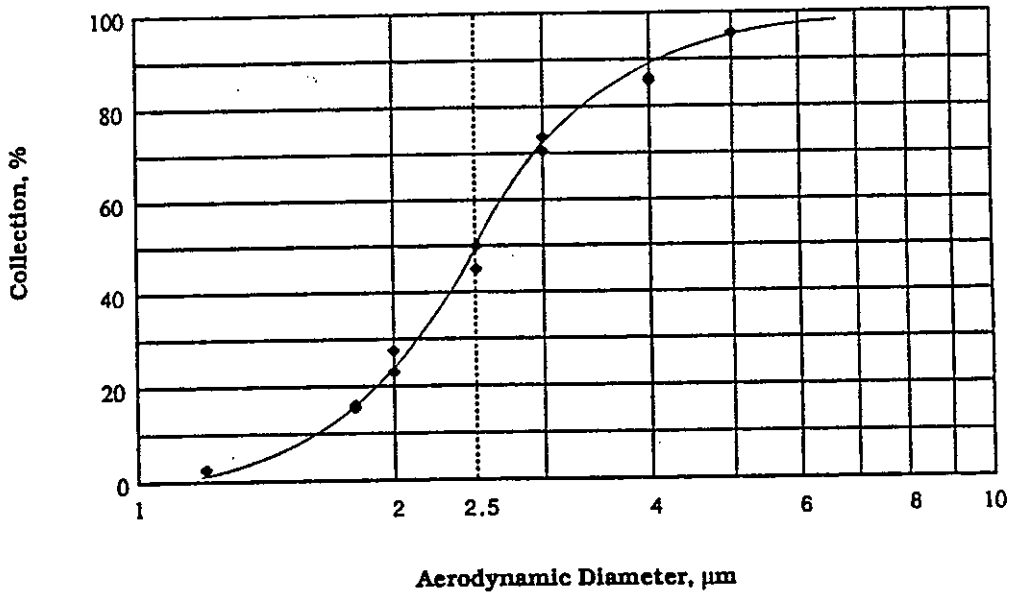


Figure 7B

Figure 7. D_{50} for Acceleration Jet (Figure 7A) and Teflon®-Coated Cyclone (Figure 7B)

731

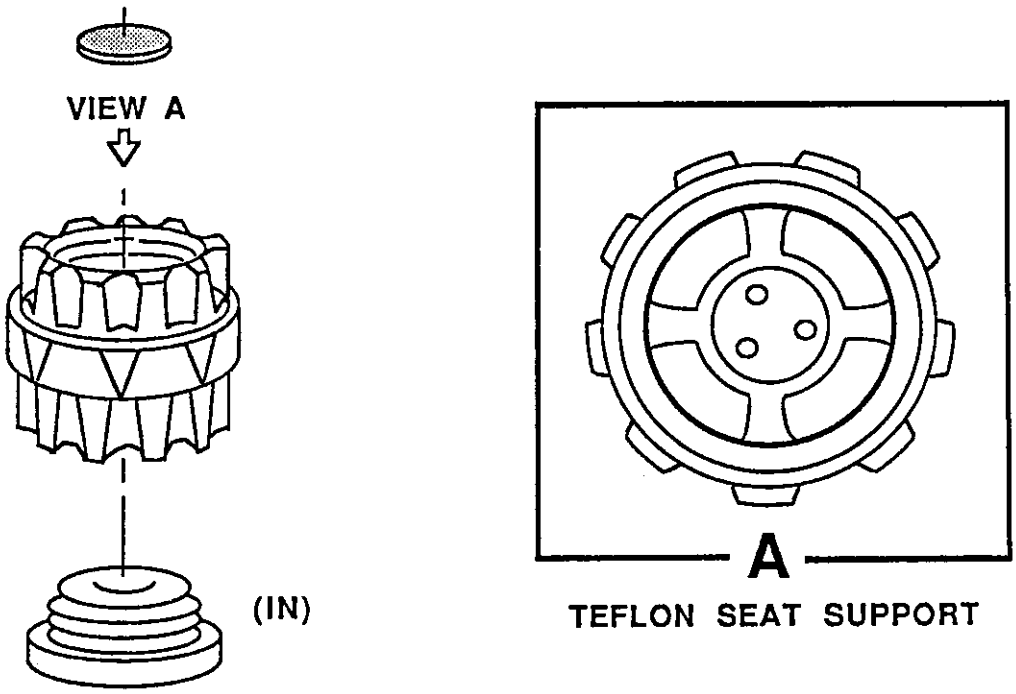


Figure 8. Side View Impactor/Coupler Assembly

732

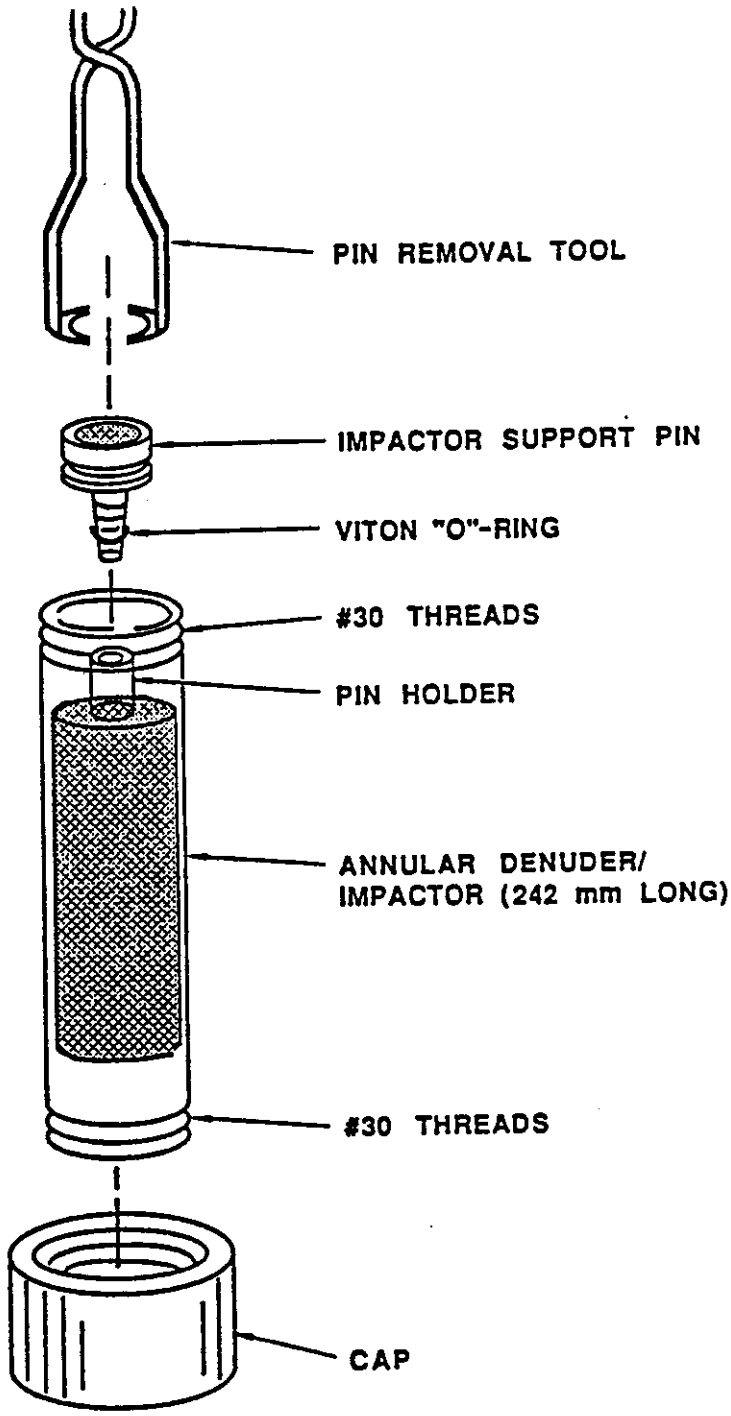


Figure 9. Glass Annular Denuder with Inset Impactor Assembly

733

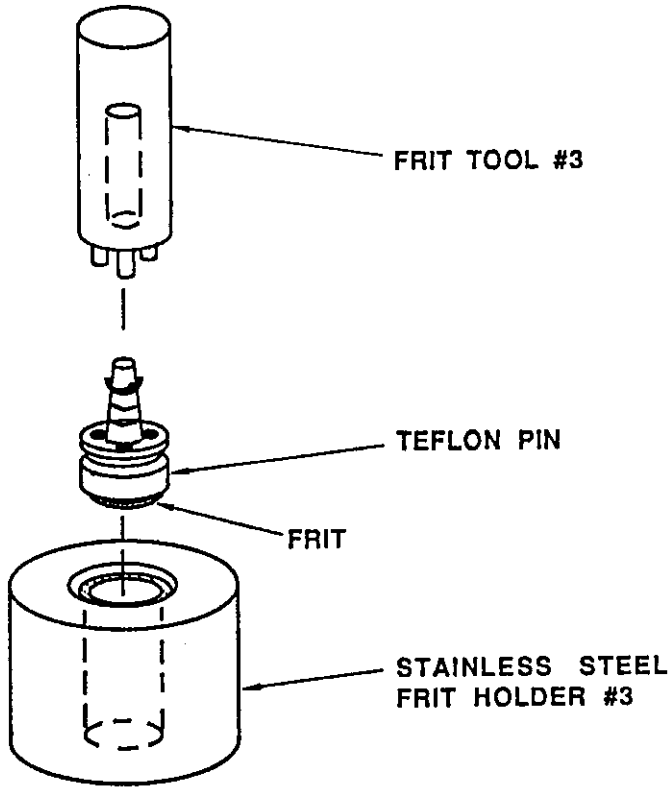


Figure 10. Frit Removal from Pin

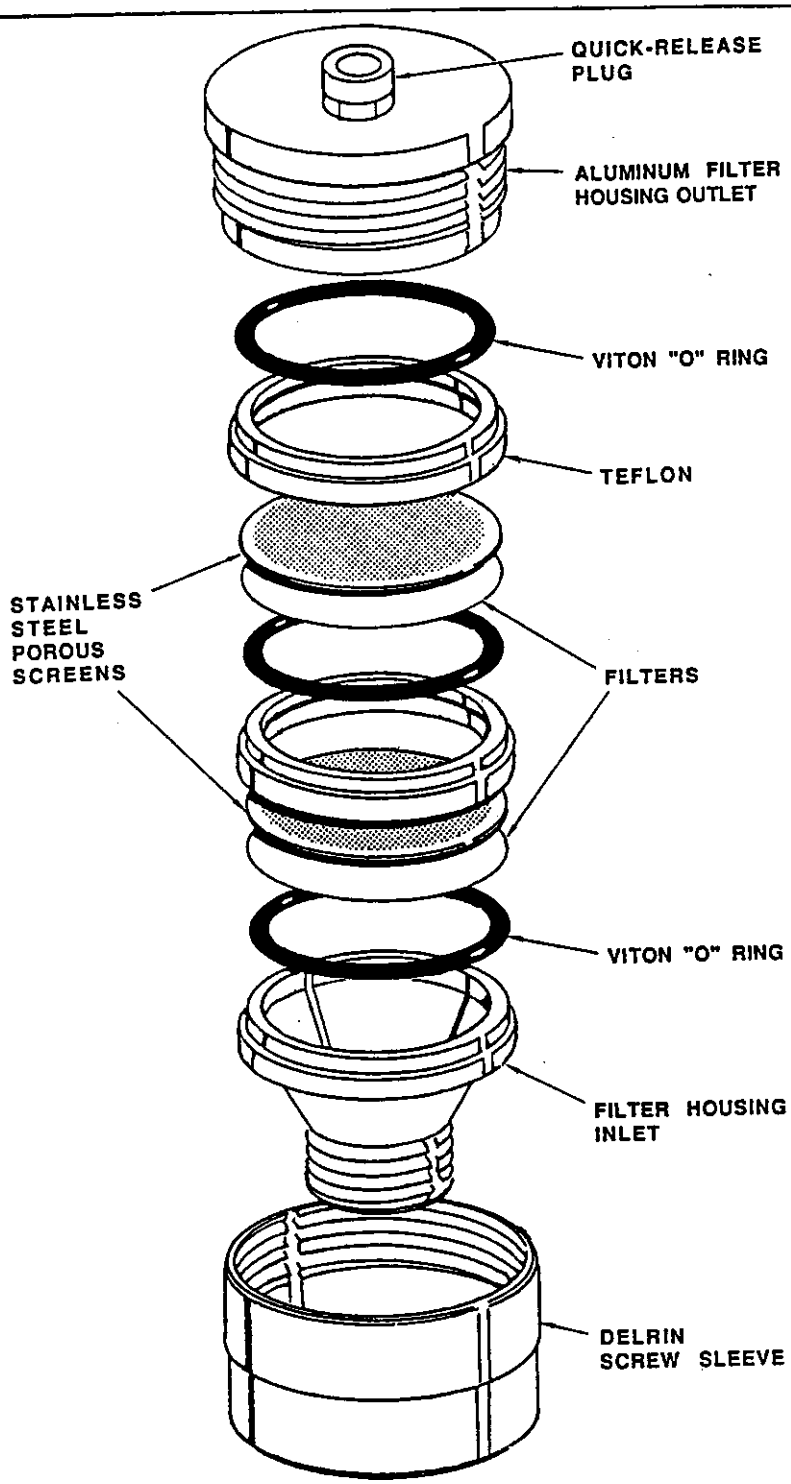


Figure 11. Filter Pack Assembly

735

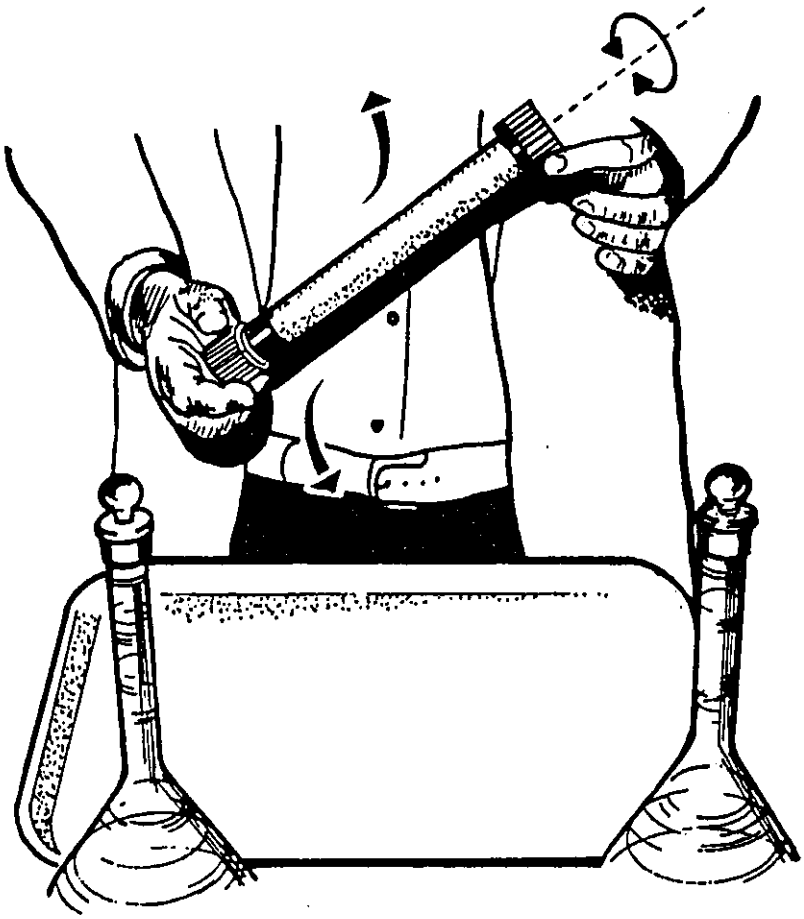


Figure 12. Annular Denuder Coating Procedure

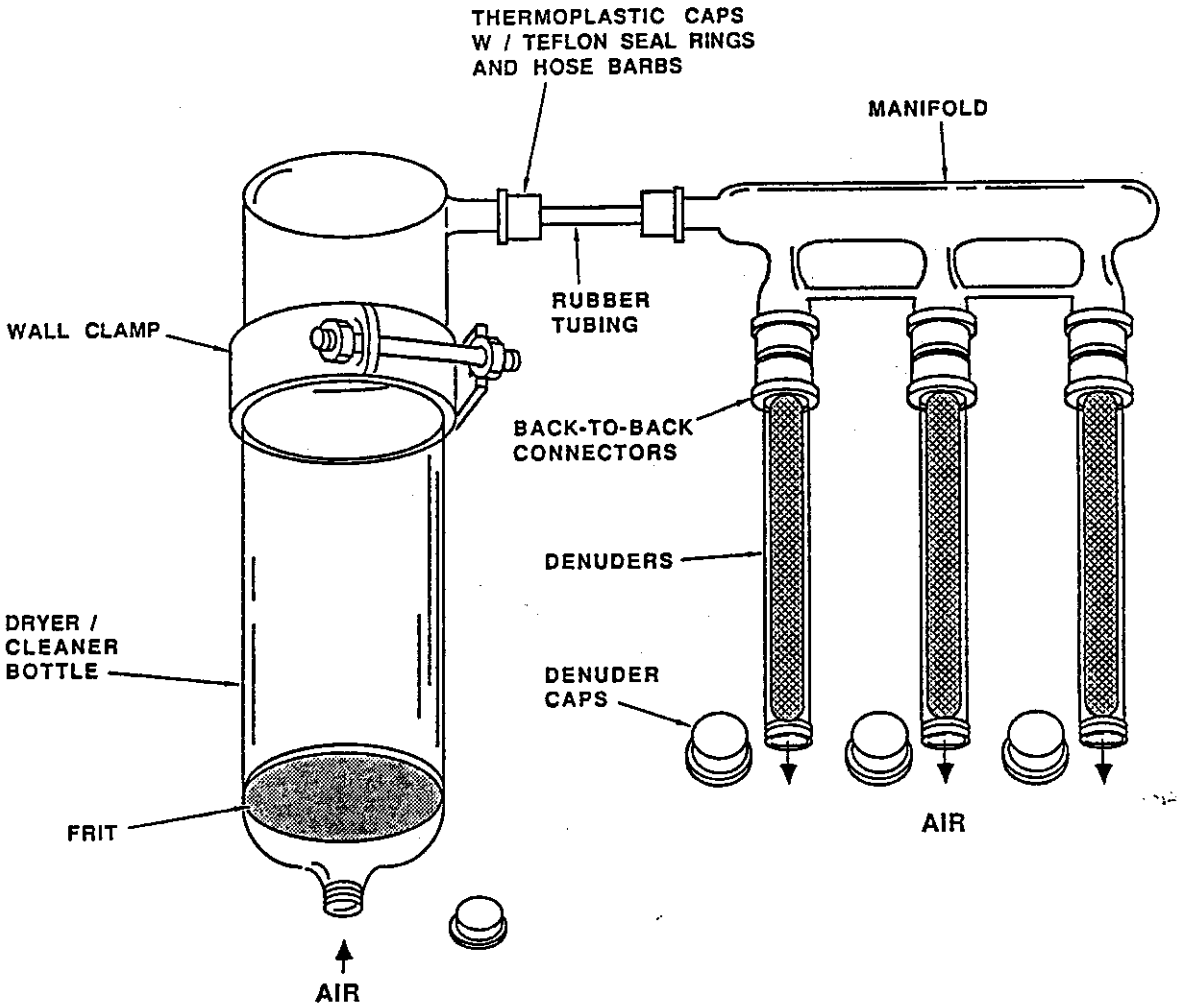


Figure 13. Drying Train and Manifold

737

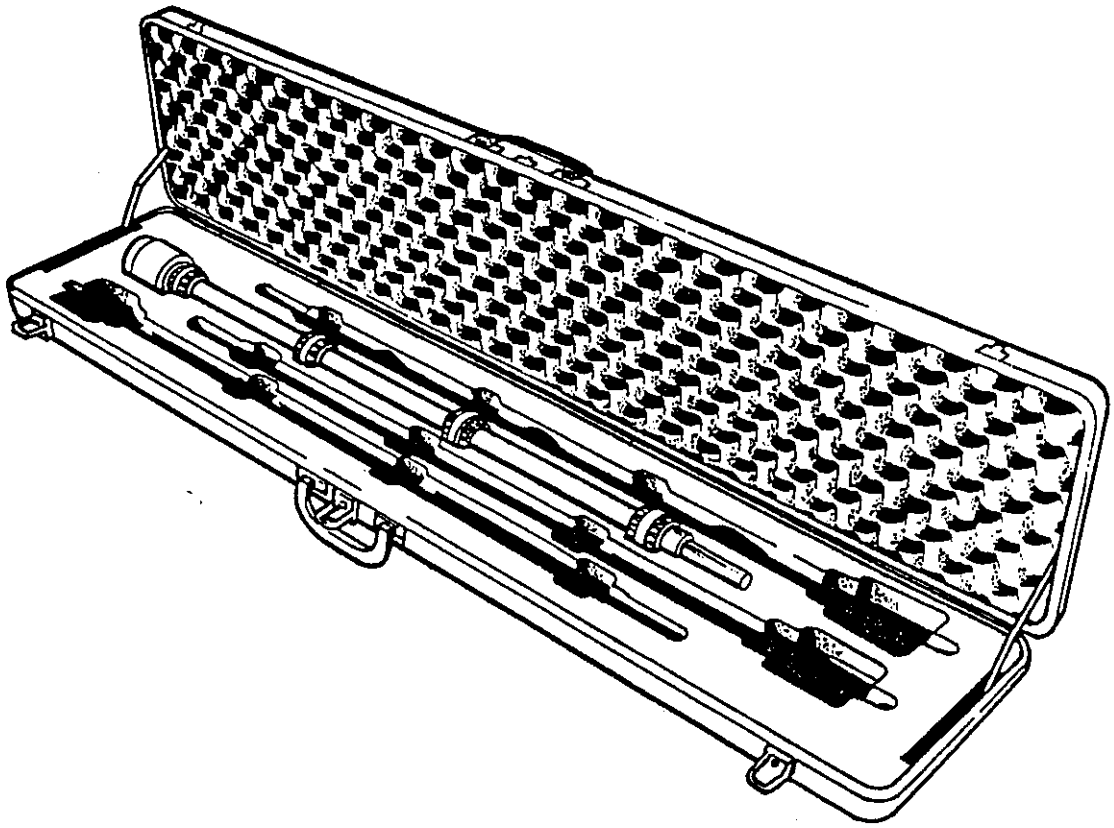


Figure 14. Annular Denuder in Field-to-Lab Case

738

DENUDER LOG SHEET

Sample Period	Date	Time Start	Time Stop	Duration	Flow L/min	Denuder Sample #A	Denuder Sample #B	Denuder Sample #C	Nylon Filter Sample #	Teflon Filter Sample #	Comments

Figure 15. ADS Field Test Data Sheet

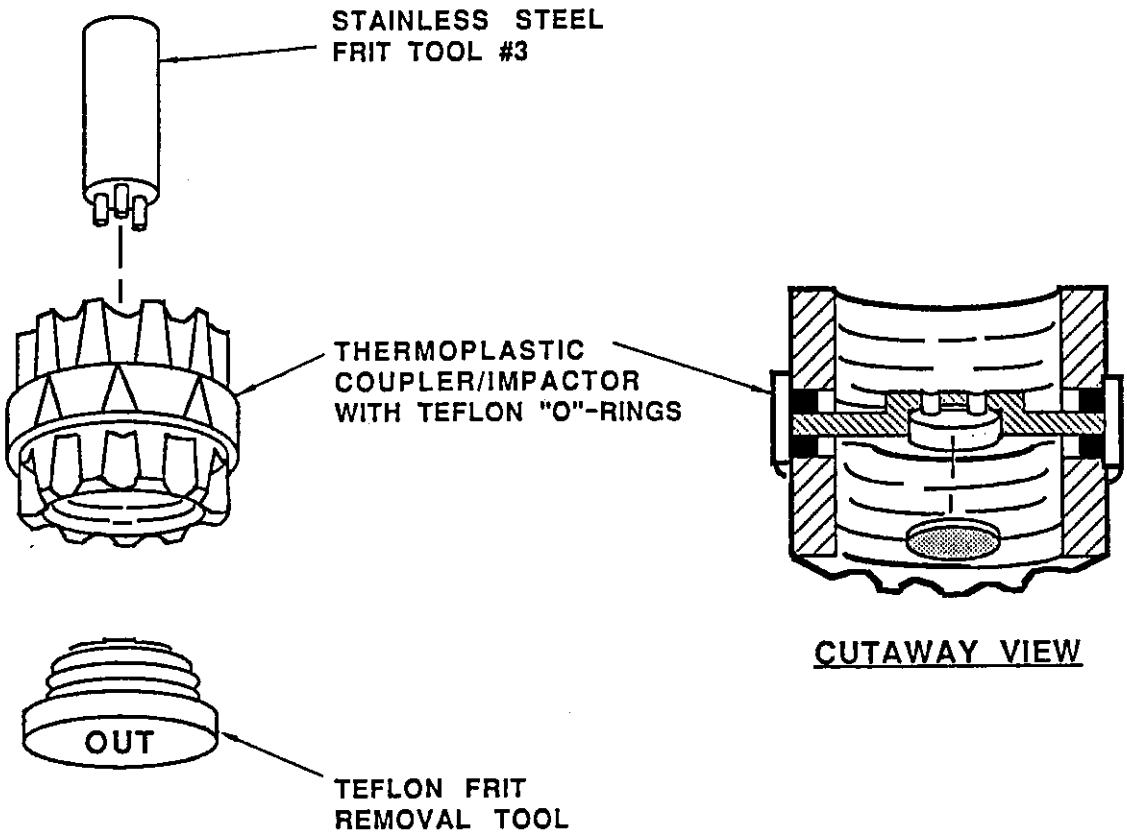


Figure 16. Side View Impactor/Coupler Assembly with Disc Removal Tools

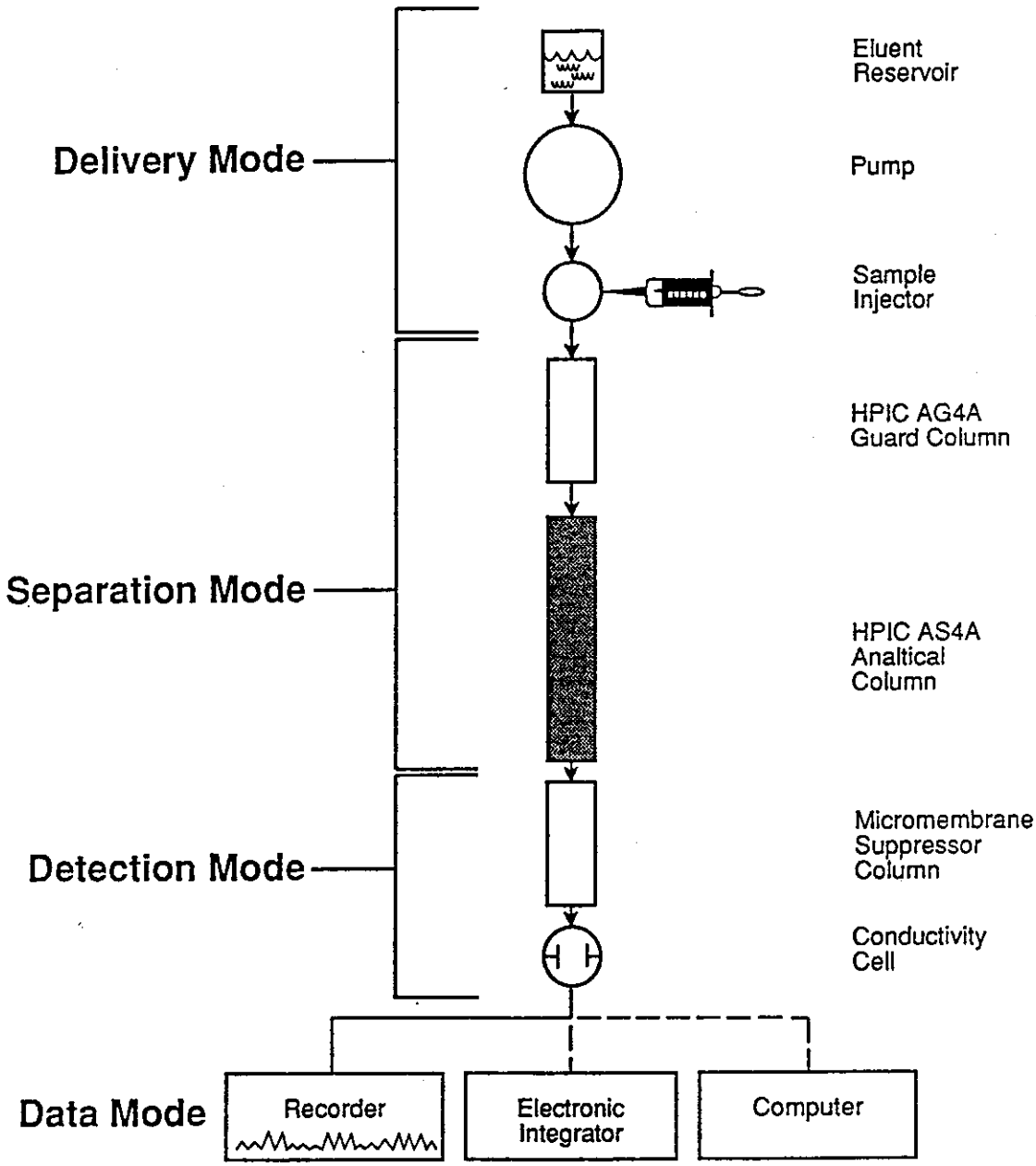


Figure 17. Major Components of a Commercially Available Ion Chromatographer

741

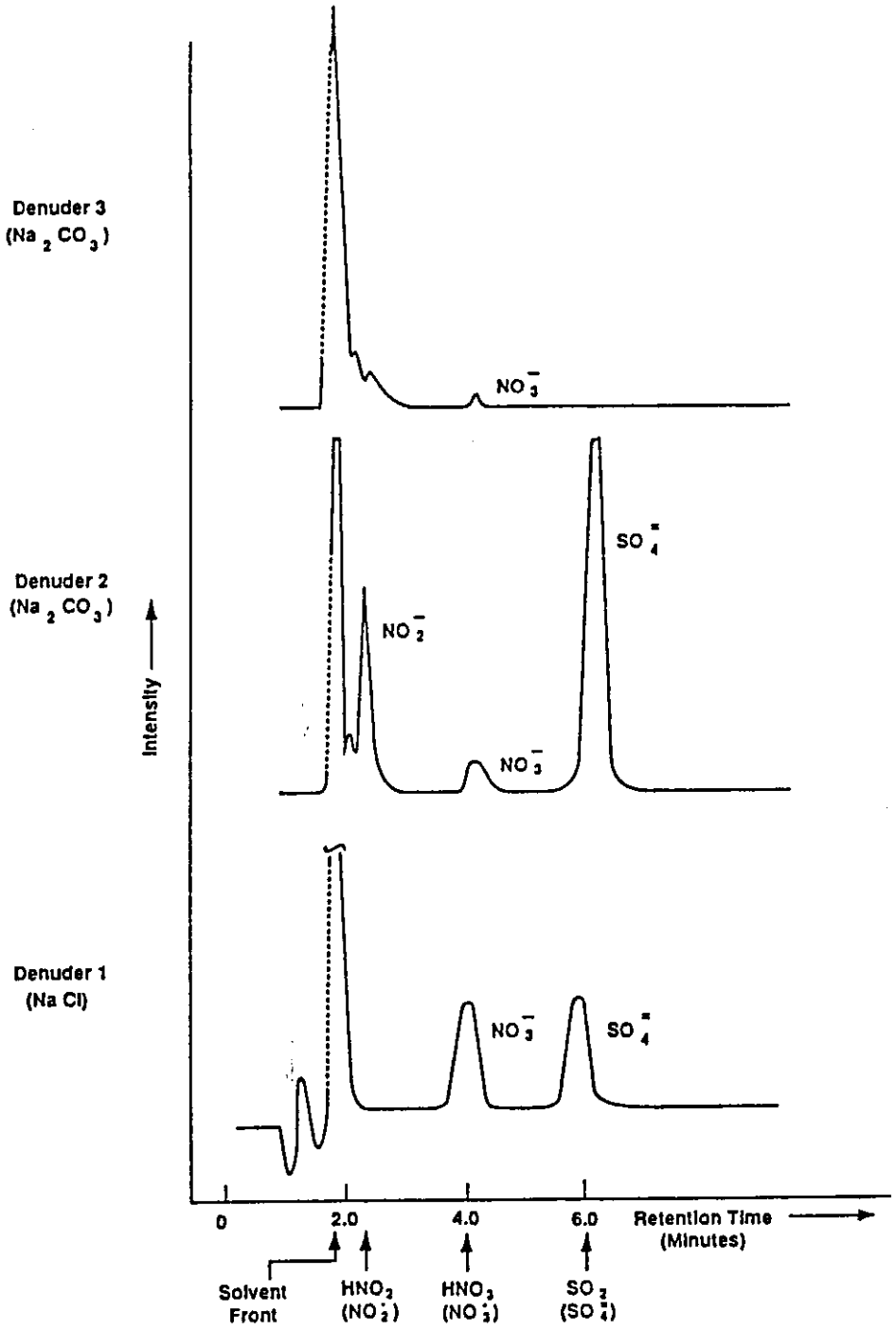


Figure 18. Chromatograms of Denuder/Filter Extract Performed by the Ion Chromatography

742

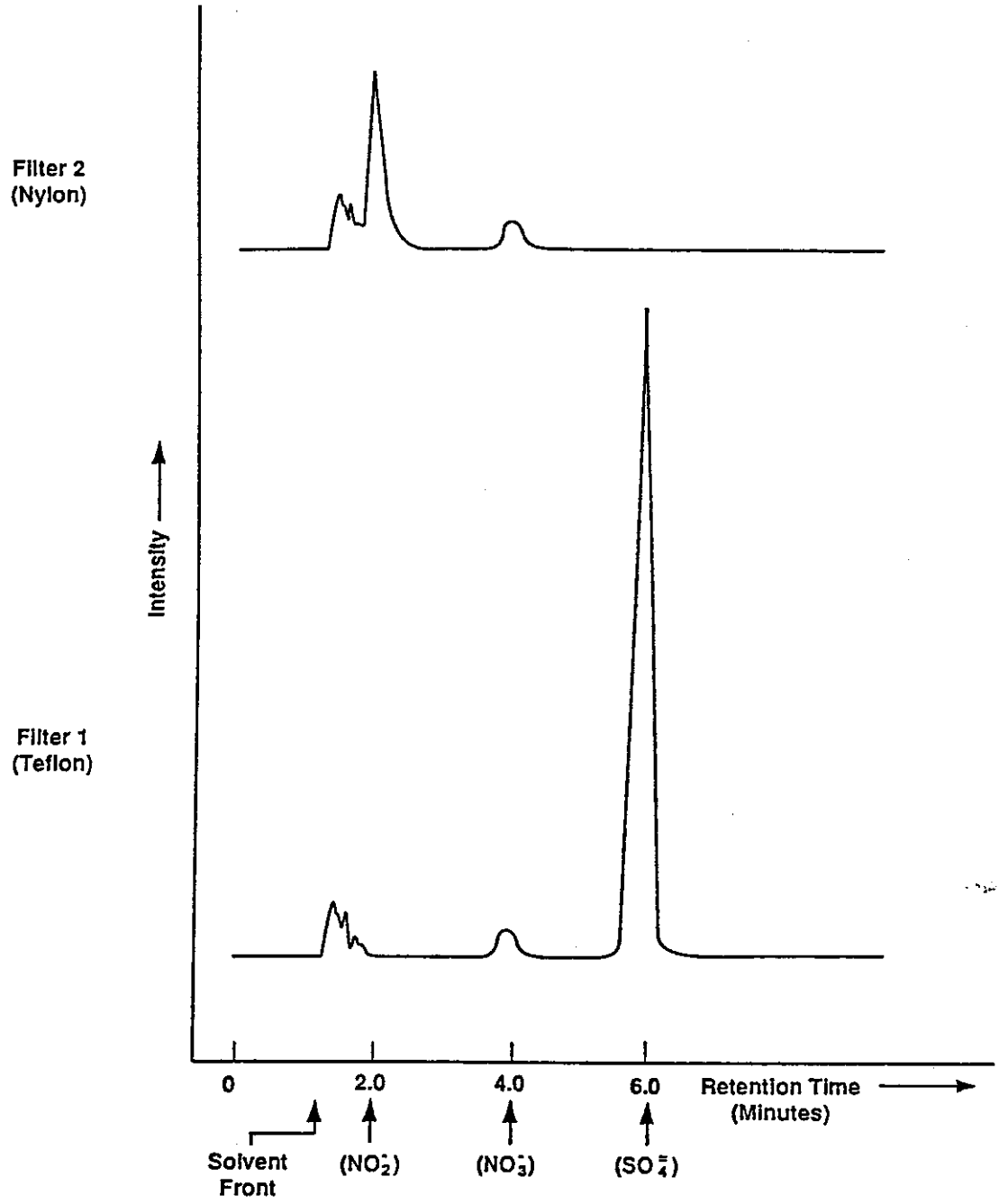
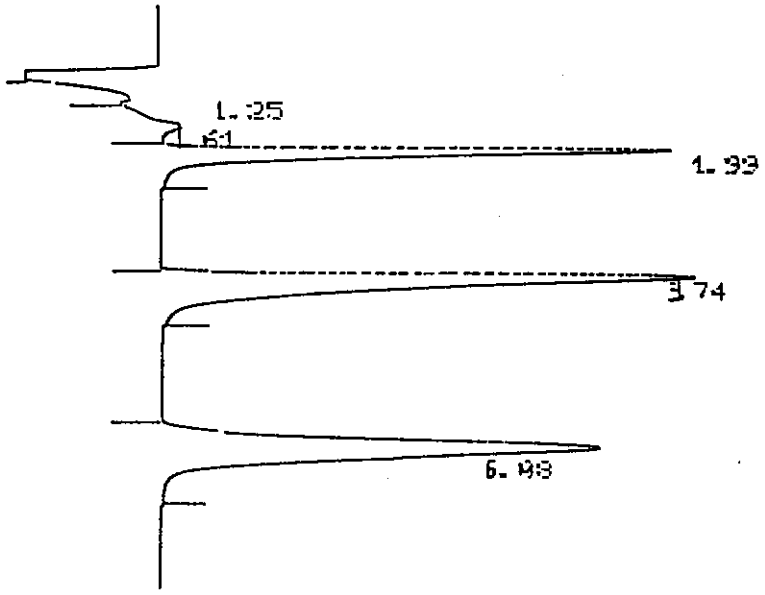


Figure 18 (Cont'd). Chromatograms of Denuder/Filter Extract Performed by the Ion Chromatography

745

DATA. CHANNEL A

SAMPLES FROM 1. TO 12. REPLICATES= 1.
CHANNEL A INJECT 05:22:17



ANIONS 05:22:17 CH= "A" PS= 1

FILE 2. METHOD 5. RUN 1 INDEX 1

ANALYST= BELL

NAME	PPM	RT	AREA	BC	RF	RRT
NO2	2.463	1.99	8009801	033252050.	75	0.327
NO3	4.829	3.74	11145516	012308038.	104	0.615
SO4	3.83	6.08	11581441	013023874.	934	1.
TOTALS	11.122		30736758			

PEAK HEIGHTS= 1 78** 2 88** 3 440** 4 437** 5 358**
RT RET 1.836 3.4884 5.3244

Figure 19. Chromatogram of a Standard with Nitrous Acid, Nitric Acid, and Sulfuric Acid

744

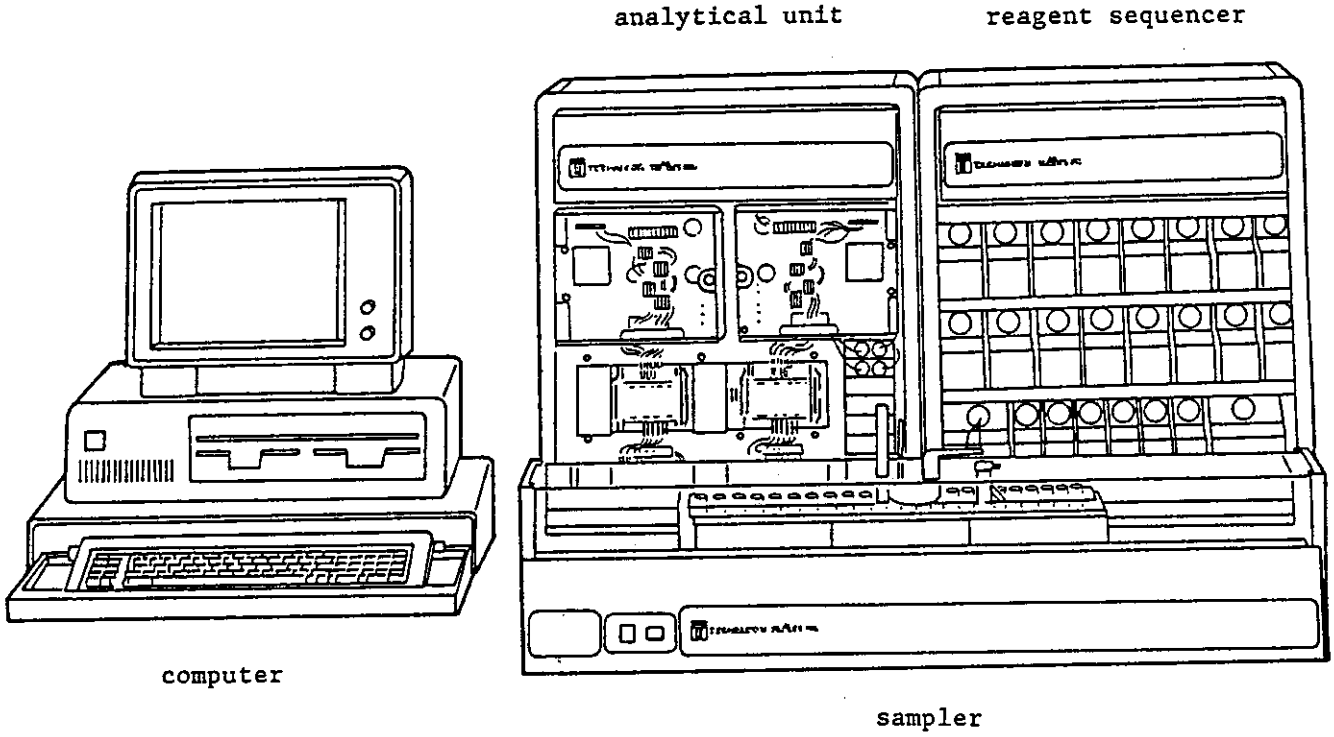


Figure 20. 4-Channel Traacs 800 System with Reagent Sequencer

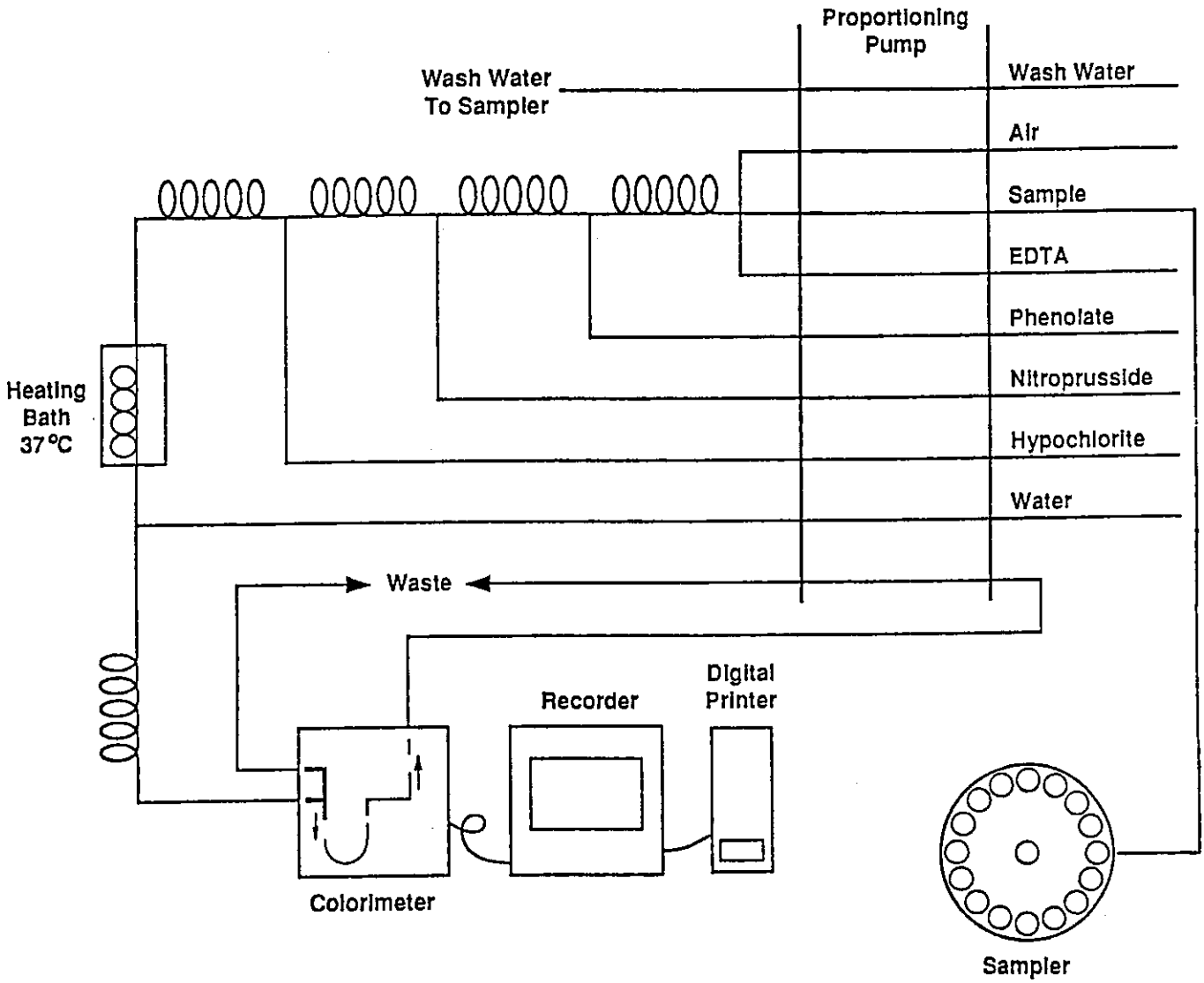


Figure 21. Technicon Autoanalyzer Flow Diagram for Ammonia Analysis

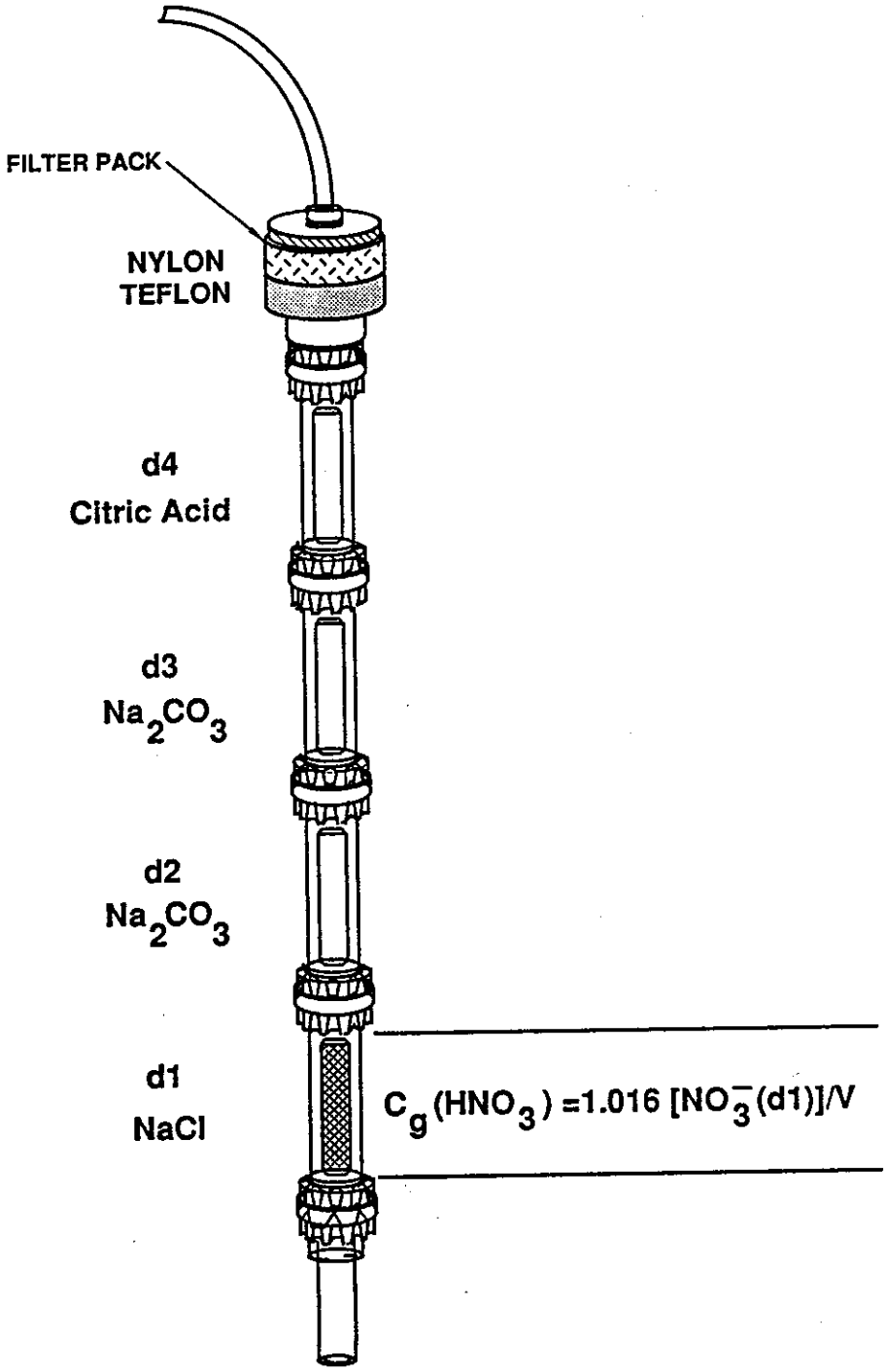


Figure 22. Nitric Acid Gas Measurement

747

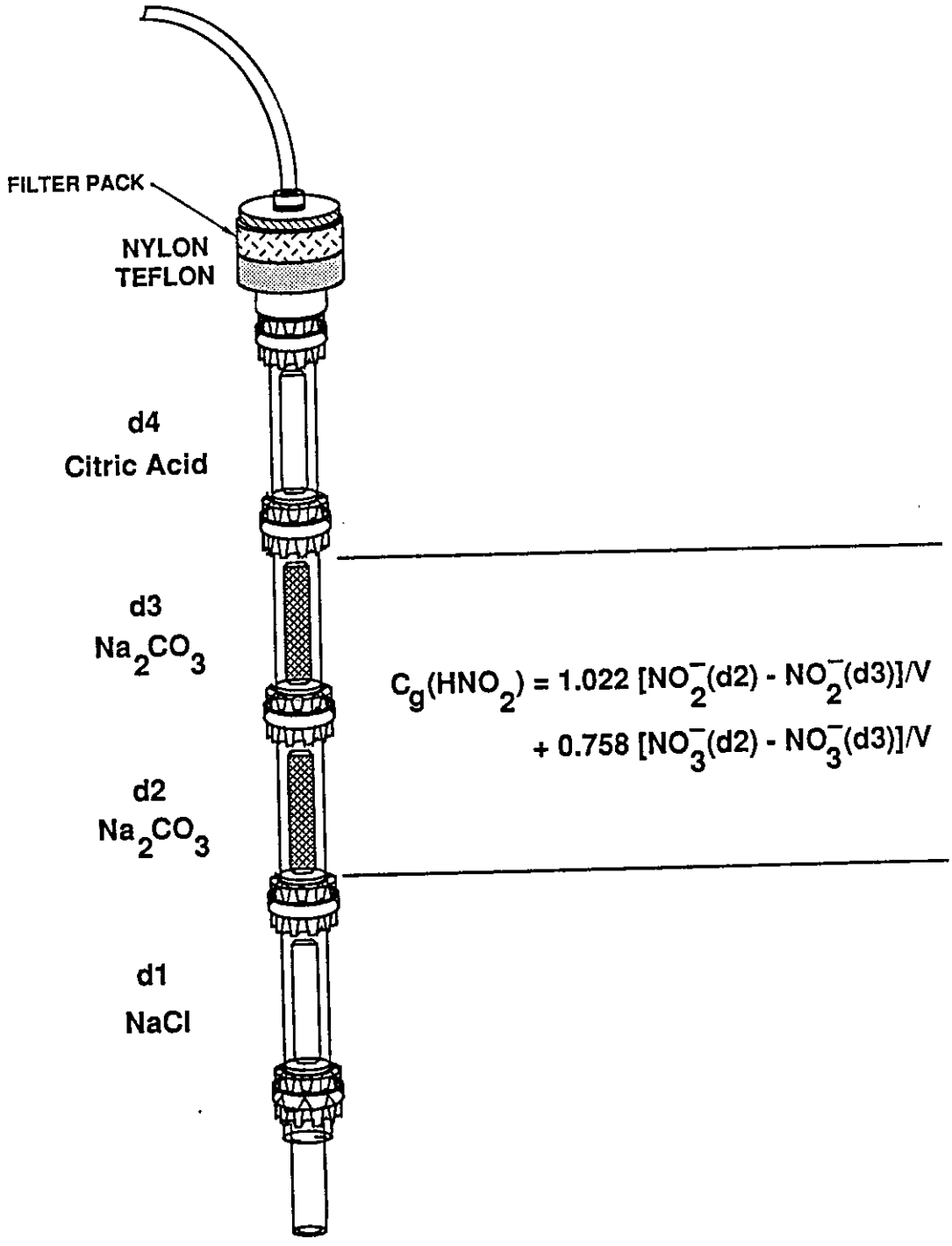


Figure 23. Nitrous Acid Gas Measurement

748

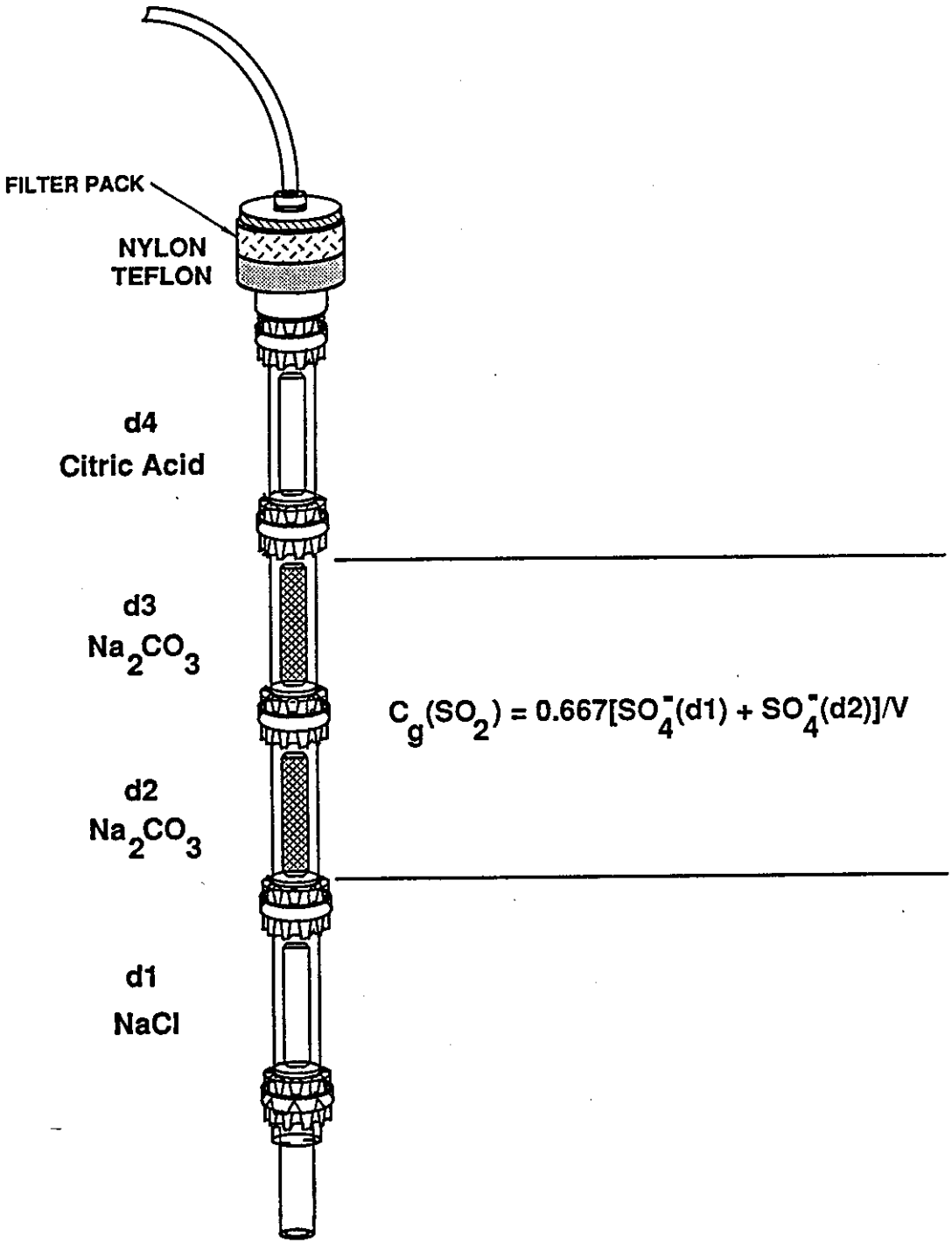


Figure 24. Sulfur Dioxide Gas Measurement

749

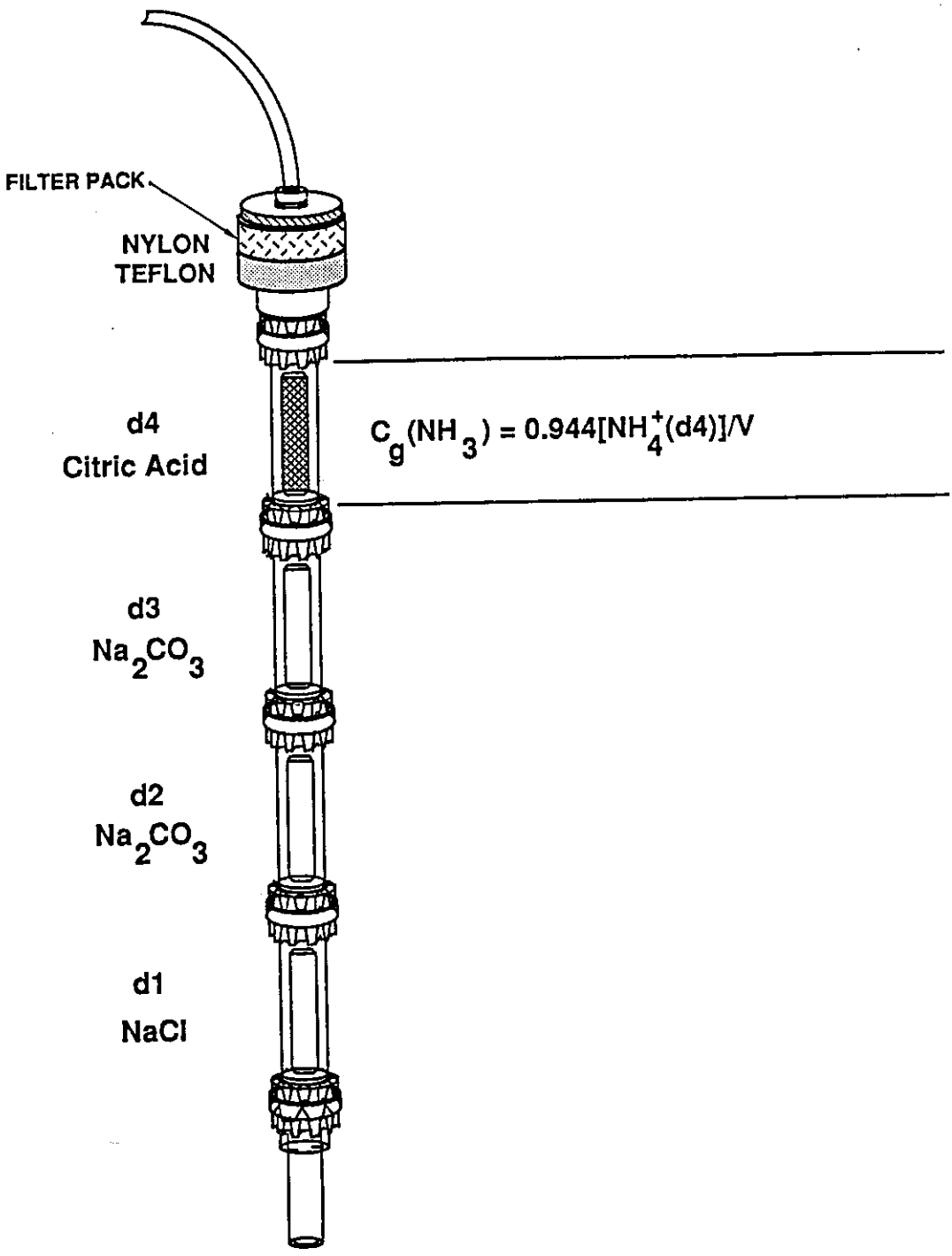


Figure 25. Ammonia Gas Measurement

750

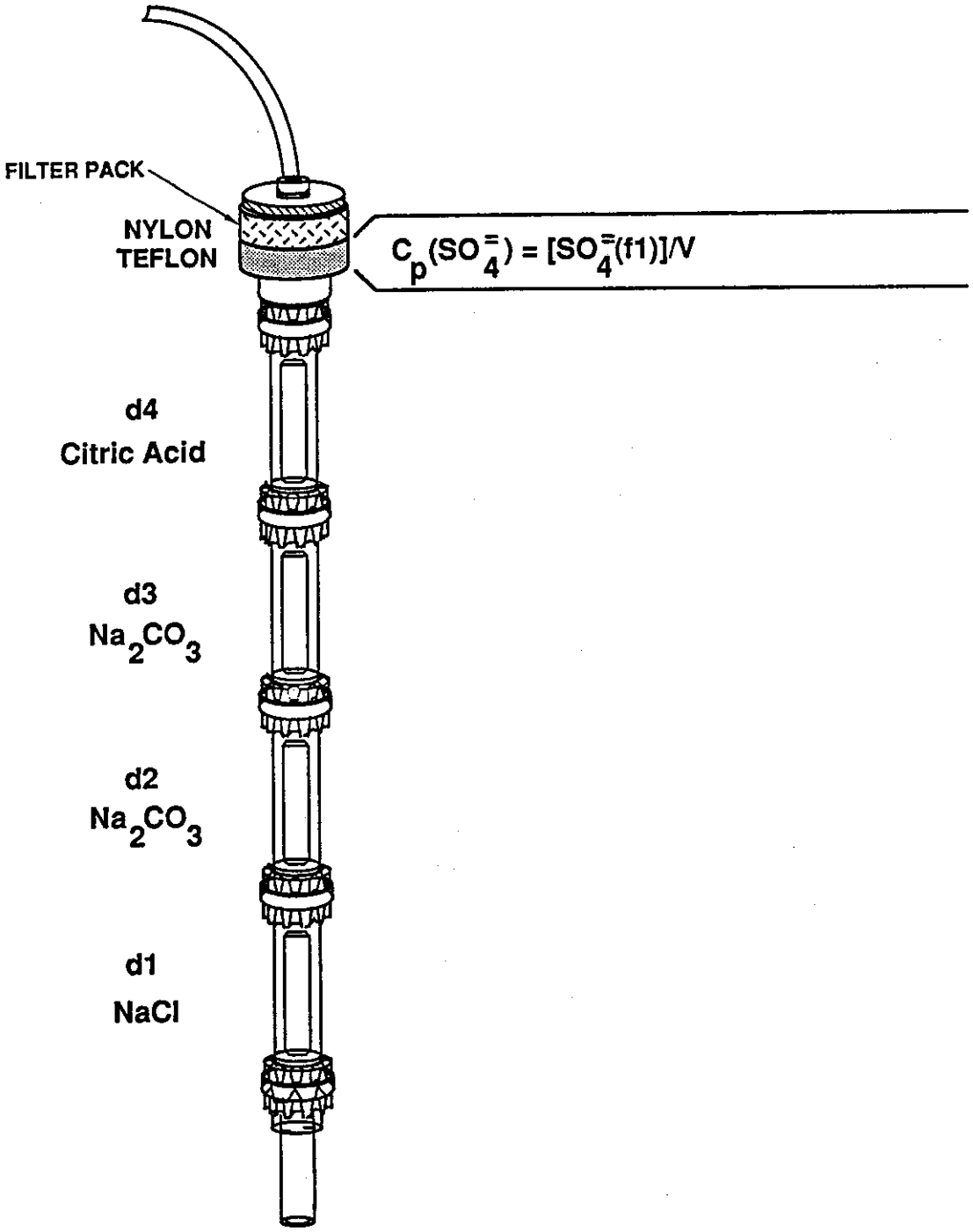


Figure 26. Particulate Sulfate Measurement

751

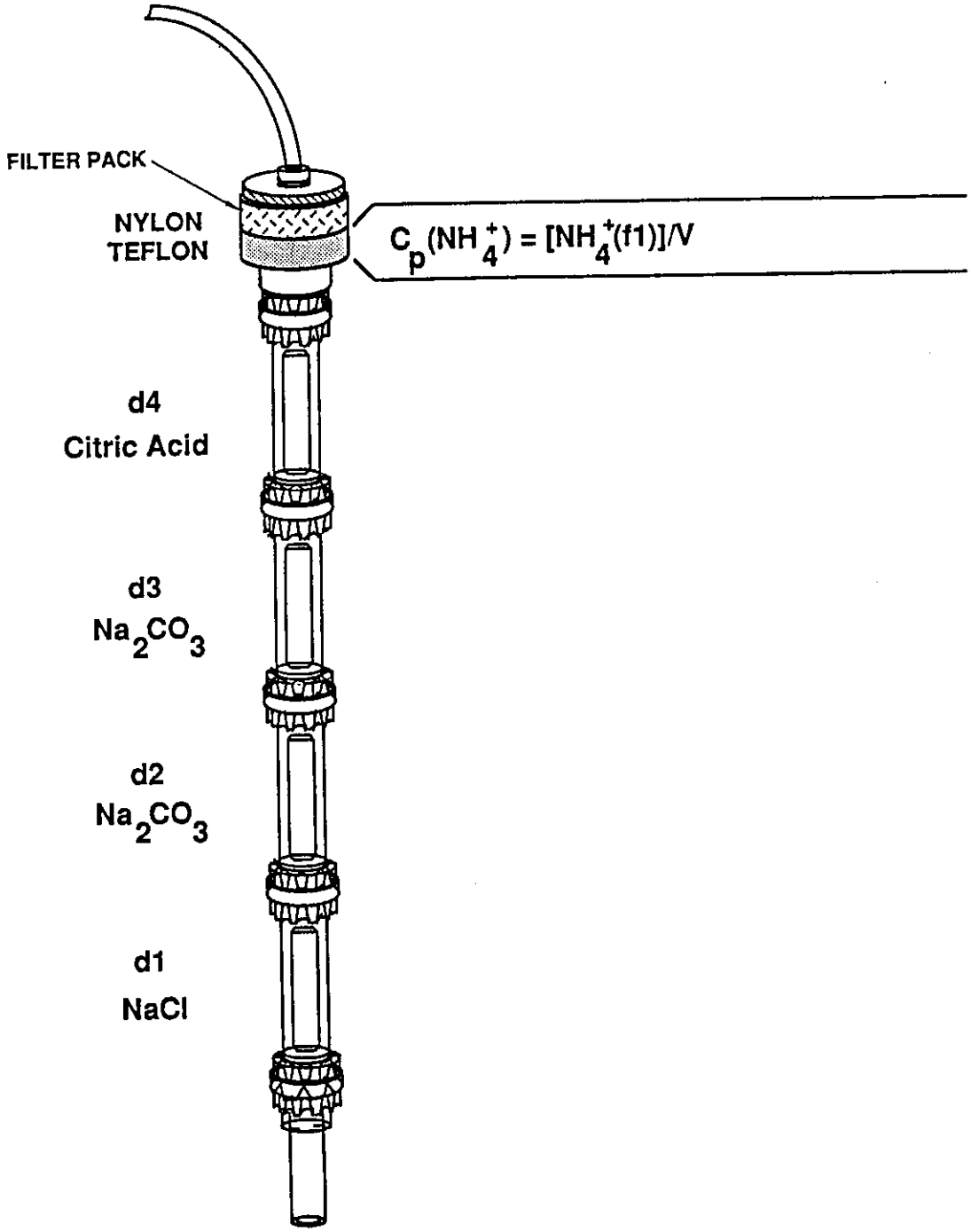


Figure 27. Particulate Ammonium Measurement

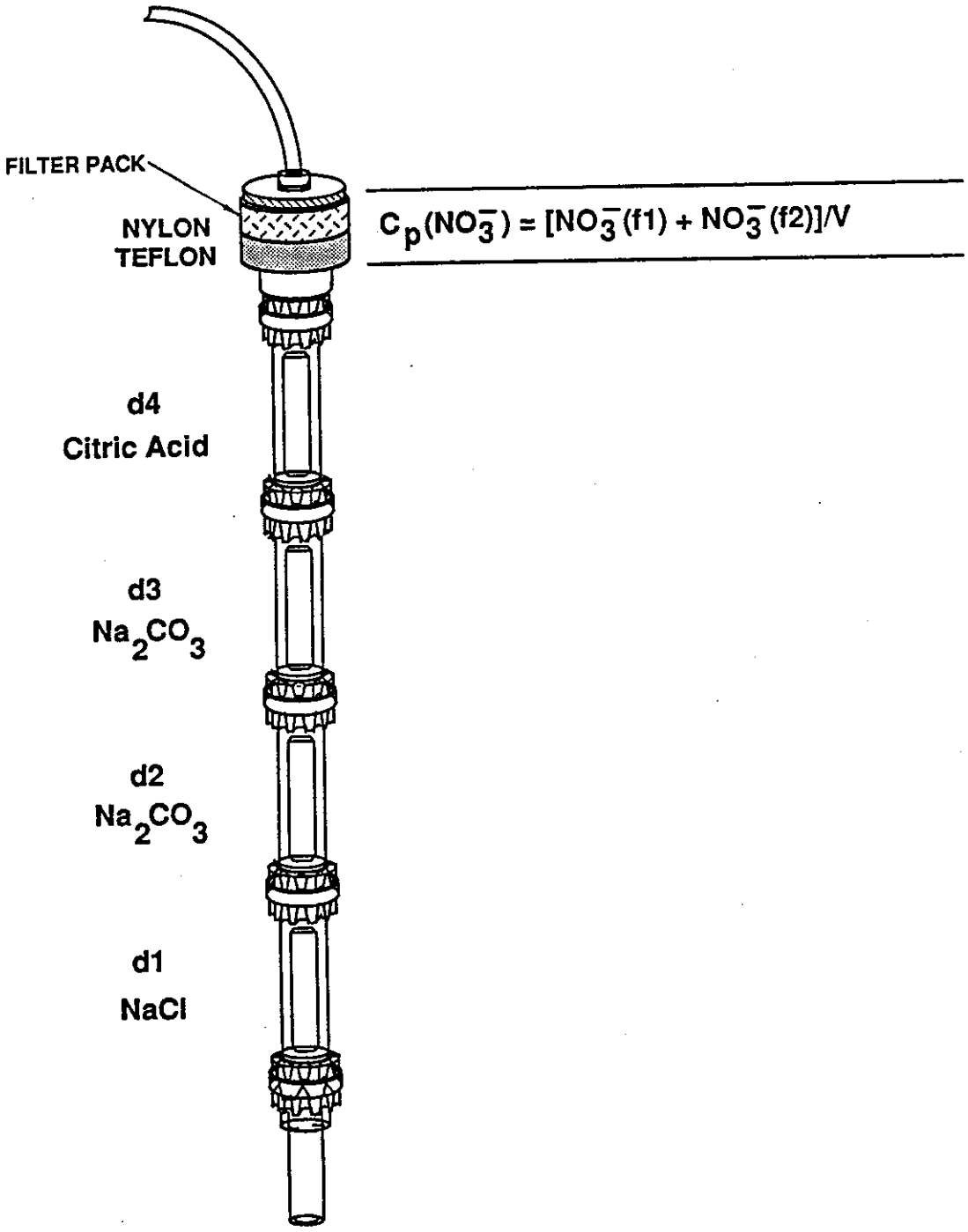


Figure 28. Particulate Nitrate Measurement

753

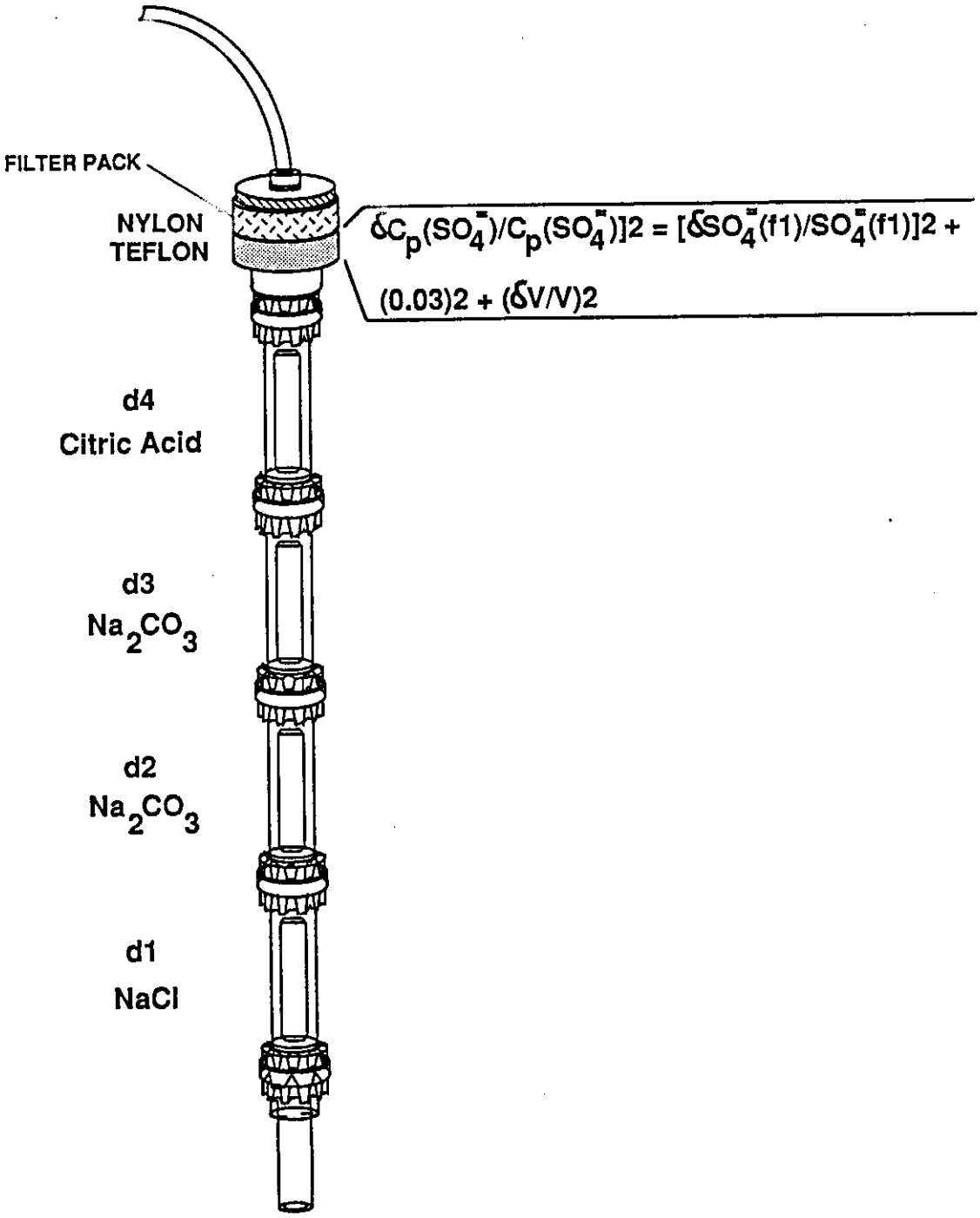


Figure 29. Particulate Sulfate Measurement and Uncertainties

754

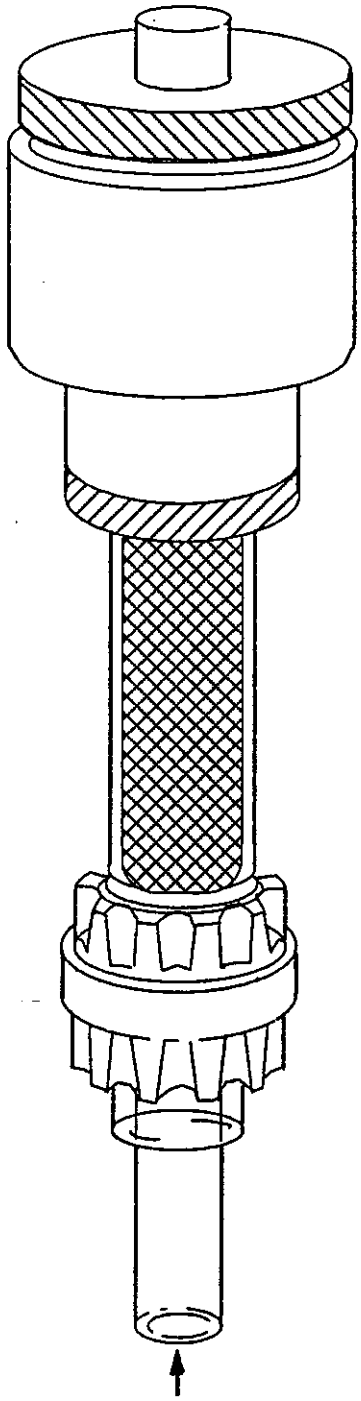


Figure 30. Annular Denuder Personal Sampler

753

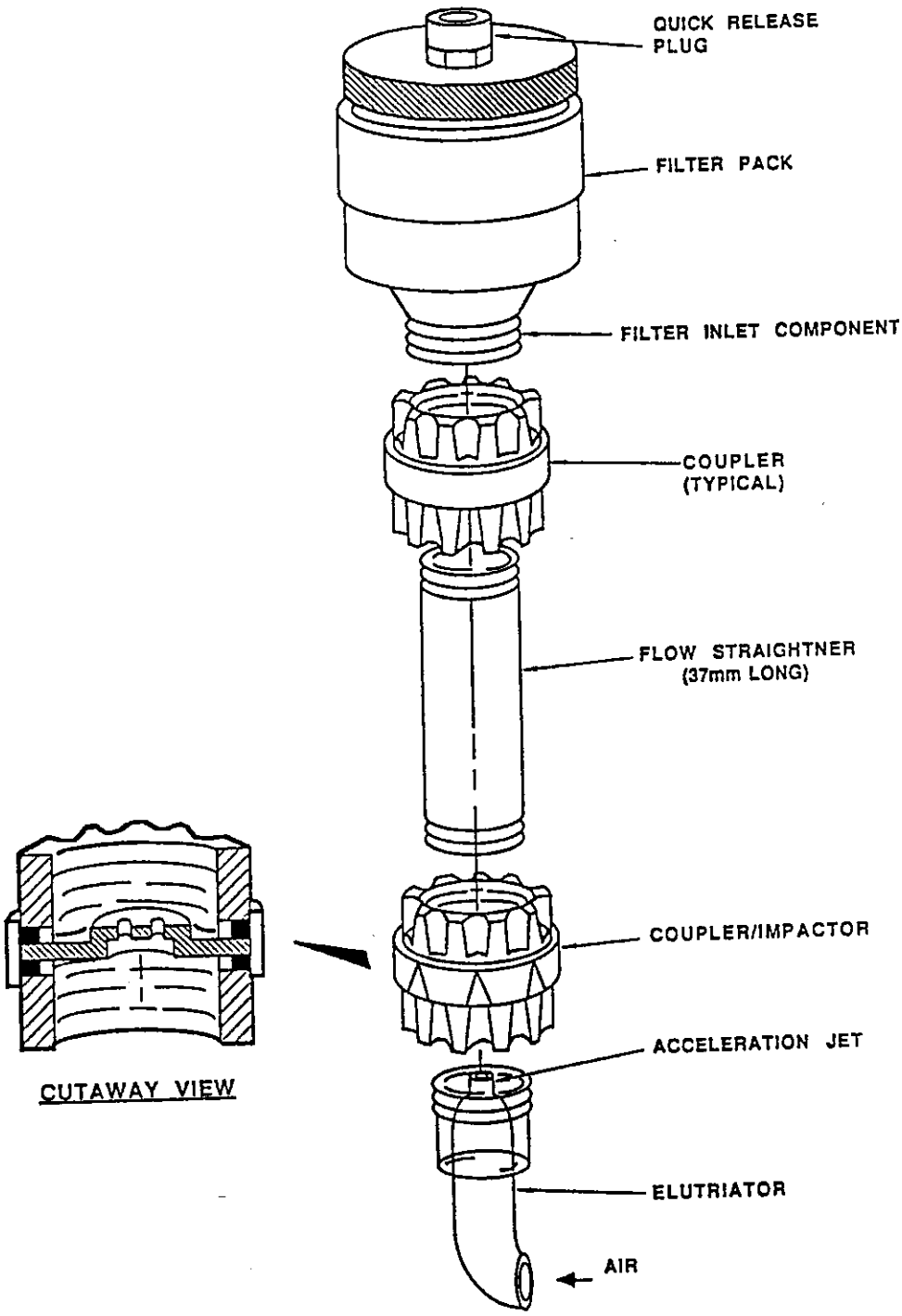


Figure 31. Fine Particle Sampler

756

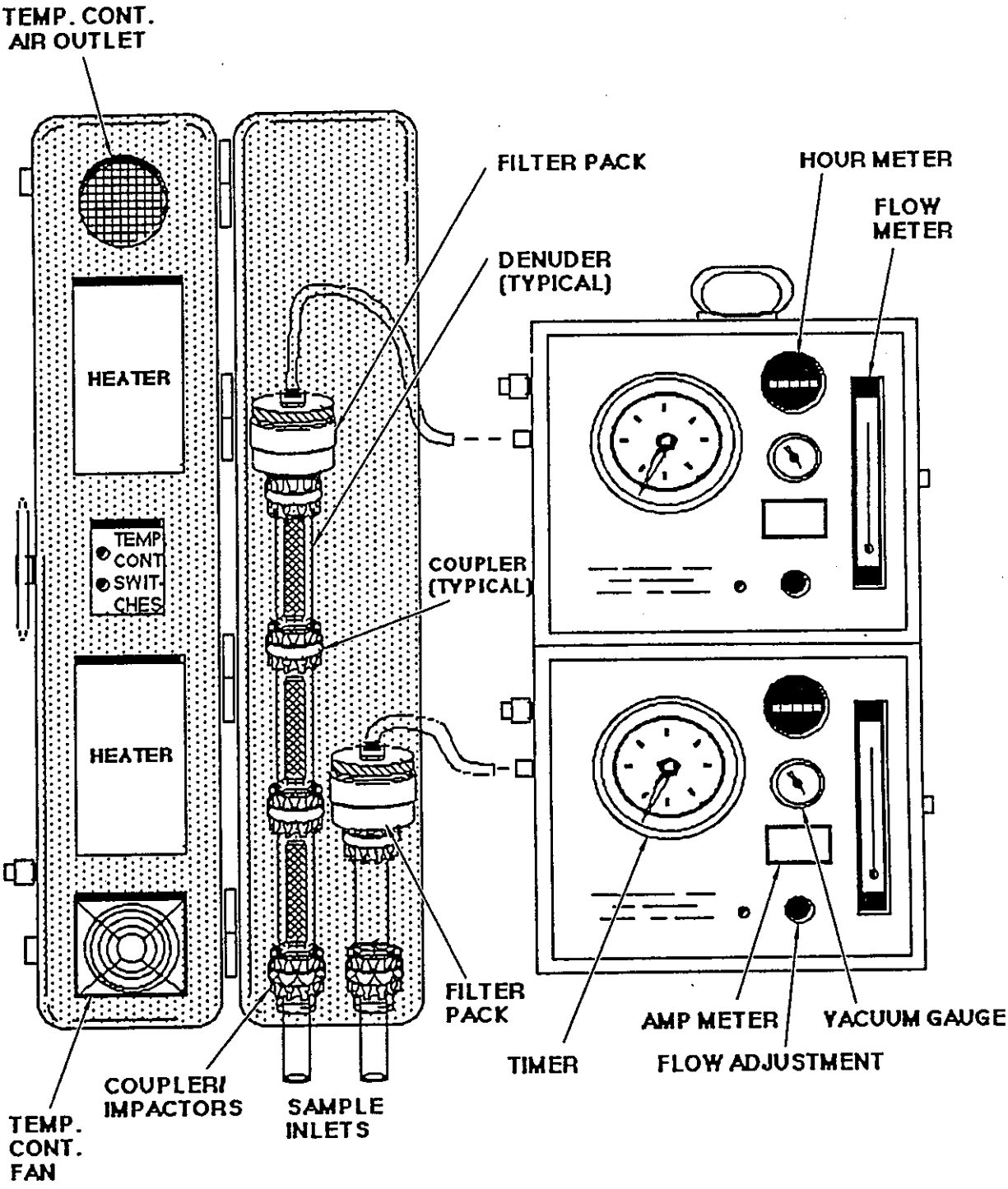


Figure 32. Annular Denuder System with Fine Particulate Sampler

757

Spectra-Physics Integrator Program
for IC Analysis

```
 2 !SKIP INJECT THEN RESTART: GO TO 99
 3 FI=2: F2=2: CA=1: !"COPY";: INPUT "V8=";: V9=1: V3=1: GO TO 25
 5 FI=5: !"USE FILE 5": GO TO 18000
 6 INPUT V4
 7 ON V4GO TO 24,65,3,5,30,255,220,229,205
24 F2=2: V3=0: CA=0
25 FI=F2: CS=1: IX=1: MC=0   : LC=1: RN=0: CW=.13: TFN"T5",1
26 T1=1.8: AC=1: AT=128 : PW=200: PT=5000: DP=3: MA=3260: MP=64
27 OF=-50: T1=.5: PW=6: PT=1000
29 !"CHECK PAPER AND CONDUCTIVITY      ";
30 GOTO 98
31 IF RC>-.01 THEN 33
32 RN=-1*RC: !"NEXT RUN IS";RN;: RN=RN-1: GO TO 30
33 IF RC=0 THEN 2074
34 IF RC=999 THEN INPUT "SCALE=";V9: GOTO 30
35 IF RC=99 THEN ABORT
36 IF RC=1 THEN RA=1: GOTO  42
40 INPUT "INJECTIONS PER STATISTICS=";RA
42 V1=RN+1
43 FI=0: !"CHECK SAMPLER FOR LOAD AND RUN";
45 PLOT OFF: PT=5000: CA=0: INJECT  : END
47 FI=F2: V2=RN+RC: !"SAMPLES FROM";V1"TO";V2"REPLICATES=";RA;
48 FI=F2: RT(4)=V8: RT(3)=V8*.87: RT(2)=V8*.62: RT(1)=V8*.34
49 CA=V3: PH=2: TT(8)=1.6+V8: TT(9)=2.9+V8: TT(10)=3.0+V8
50 PLOT A: INJECT  :  END
52 ! $10"PEAK HEIGHT(N)="PHS(1)/1000,PHS(2)/1000,PHS(3)/1000"
55 TD(RN)=AC(1):  TE(RN)=AC(2): TG(RN)=AC(3): TI(RN)=AC(4)
59 IF RA=1 THEN TD(RN)=-9.9
60 IF IX=1 THEN !;"RT SET",RT(1),RT(2),RT(3),RT(4) ELSE TD(RN)=-9.9
61 TA(RN)=LC(1): TZ(RN)=LC(2): TC(RN)=LC(3): TJ(RN)=LC(4)
62 IF RN=V2 THEN 190
64 GOTO 48
```

```
   65 FI=2: RN=V2: PLOT OFF: INJECT  : END
   98 !"TOTAL NEW INJECTIONS=";
   99 INPUT RC;
  101 IF RC=-99 THEN 2: ELSE 31
  190 V5=V5+RN : !"LOOP=75UL;COL=AS4A; S/N09317;PAST CAL=";V5
  191 !"EL=.0018 nA2co3;.0017nAhco3=12.9US;10US=1v;fLOW=1.7ML/M=1200PSI
  192 IF V3=0 THEN 210
  193 !"X=ACTUAL   +=CALCULATED":  V5=0
  194 TFN "T5",0 : ABORT: END
  205 FI=F2: INPUT "SCALE=";V9
  210 !TAB 15"PARTS PER MILLION  (UG/ML)"
  212 !" RUN NUMBER ",$9.03,CN(1),CN(2),CN(3),CN(4)
  214 FOR K=V1 TO RN: !$9 K$9.3,TA(K)*V9,TZ(K)*V9,TC(K)*V9,TJ(K)*V9
  215 IF TD(K )=-9.9   OR RA=1 THEN 218
  216 !TAB 9 "AVERAGE"  $10.3;TD(K)*V9,TE(K)*V9 ,TG(K)*V9, TI(K)*V9
  218 A=A+(TA(K)*V9): B=B+(TZ(K)*V9): C=C+(TC(K)*V9): D=D+TJ(K)*V9
  219 NEXT K: !TAB 15"SUM"$9.3 ;A,B,C,D: END
  220 FI=9: GOTO 194
  229 FI=8: F2=8: CA=1: !"COPY";: INPUT "V8=";V8;: V9=1: V3=1: GOTO 25
  233 !Y;
  255 INPUT "CHANGE END  SAMPLE TO";V2: RC=V2-RN: GOTO 47
  356 !"PEAK HEIGHTS=";: A=SIZE"PS": IF A>10 THEN A=10
  358 FOR K=1 TO A : !$2 K $5 PSH(K)/1000"**";
  360 NEXT K: ! : END
  400 FOR I=1 TO 4 : ! KA(I),KB(I),KC(I),I: NEXT I: END
  410 FOR I=1 TO 4 : !I;: INPUT KA(I),KB(I),KC(I): NEXT I: END
 2050 STOP 64: END
 2074 GOTO 6
 8340 V="XF"GOSUB 8650NEXT !!GOTO 400
18635 !$8;T;: GOTO 18640
```

Chapter IP-10

DETERMINATION OF RESPIRABLE PARTICULATE MATTER IN INDOOR AIR

1. Scope

Suspended particulate matter in air is generally considered to consist of all airborne solid and low vapor pressure liquid particles that are airborne. Suspended particulate matter in air presents a complex multiphase system consisting of a spectrum of aerodynamic particle sizes ranging from below 0.01 microns (μm) up to 100 μm and larger. Historically, measurement of particulate matter (PM) has concentrated on total suspended particulates (TSP), with no preference to size selection. Research on the health effects of TSP in ambient and indoor air has focused increasingly on those particles that can be inhaled into the respiratory system, i.e., particles of less than 10 μm aerodynamic diameter. It is now generally recognized that, except for toxic materials, it is this fraction (<10 μm) of the total particulate loading that is of major significance in health effects.

2. Applicability

2.1 Recent studies involving particle transport and transformation suggest strongly that atmospheric total suspended particulate (TSP) matter commonly occurs in two modes. The fine or accumulation mode is attributed to growth of particles from the gas phase and subsequent agglomeration, while the coarse mode is made up of mechanically abraded or ground particles. Particles that have grown from the gas phase, either because of condensation, transformation or combustion, occur initially as very fine nuclei 0.05 μm in size. Those particles tend to grow rapidly to accumulation mode particles around 0.5 μm in size which are relatively stable in the air. Because of their initially gaseous origin, this range of particles sizes includes inorganic ions such as sulfate, nitrate, ammonia, combustion-form carbon, organic aerosols, metals (Pb), cigarette smoke by-products, and consumer spray-products.

2.2 Consequently, based upon the health effects of coarse and fine particulate matter, a method has been developed to determine both continuous and speciated coarse (<10 μm) and fine (<2.5 μm) particulate matter in indoor air. A Microenvironmental Exposure Monitor (MEM) has been developed as a fixed site monitor. Similarly, Personal Exposure Monitors (PEMs) have been developed to estimate personal exposure to particles. Finally, a TEOM® continuous monitor is presented as a means of determining total mass on a real-time basis.

Method IP-10A

DETERMINATION OF RESPIRABLE PARTICULATE MATTER IN INDOOR AIR USING SIZE SPECIFIC IMPACTION

1. Scope
2. Applicable Documents
3. Summary of Method
4. Significance
5. Definitions
6. Method Limitations and Limits of Detection
7. Apparatus Description
   - 7.1 Microenvironmental Exposure Monitor (MEM)
   - 7.2 Personal Exposure Monitor (PEM)
   - 7.3 Cahn Microbalance
   - 7.4 Weighing Room Environment
8. Apparatus Listing
   - 8.1 Microenvironmental Exposure Monitor
   - 8.2 Personal Exposure Monitor
9. Filter Preparation and Initial Weighing
   - 9.1 Overview
   - 9.2 Cahn Microbalance Operational Protocol
     - 9.2.1 General
     - 9.2.2 Balance Zeroing
     - 9.2.3 Balance Calibration
   - 9.3 Initial Filter Weighing
   - 9.4 Packaging Filters
10. Preparation of the MEM Impactor Assembly
    - 10.1 General
    - 10.2 Cleaning of the Stainless Steel Impactor Plates
      - 10.2.1 Laboratory Environment
      - 10.2.2 Field Environment
11. Sampling
    - 11.1 Placement of Filters in the MEM
    - 11.2 Initial Field Flow Check of Sampler
    - 11.3 Placement of Sampler and Sampling
    - 11.4 Final Field Flow Check of Sampler
    - 11.5 Changing Impactors
12. Filter Recovery and Final Weighing
    - 12.1 24-hour Filter Equilibration
    - 12.2 Filter Inspection
    - 12.3 Final Weighing
    - 12.4 Independent Audit of Weighted Filters

13. Calculation
    13.1 Mass Calculation
    13.2 Volume Air Parcel Sampled
    13.3 Concentration of Particles in Air Parcel Sampler
14. Sampling System Calibration
15. Method Safety
16. Performance Criteria and Quality Assurance
17. References

Method IP-10A

DETERMINATION OF RESPIRABLE PARTICULATE MATTER IN INDOOR AIR USING SIZE SPECIFIC IMPACTION

1. Scope

1.1 Suspended particulate matter in air is generally considered to consist of all airborne solid and low vapor pressure liquid particles (1-3) that are airborne. Suspended particulate matter in air presents a complex multiphase system consisting of a spectrum of aerodynamic particle sizes ranging from below 0.01 microns (μm) up to 100 μm and larger. Historically, measurement of particulate matter (PM) has concentrated on total suspended particulates (TSP), with no preference to size selection (4). Research on the health effects (5-7) of TSP in ambient and indoor air has focused increasingly on those particles that can be inhaled into the respiratory system, i.e., particles of aerodynamic diameter less than 10 μm. It is now generally recognized that, except for toxic materials, it is this fraction (<10 μm) of the total particulate loading that is of major significance in health effects (8).

1.2 The two processes by which particles are formed are the grinding or atomization of matter (9-10), and the nucleation of supersaturated vapors, as illustrated in Figure 1. The particles formed in the first process are products of direct emissions into the air, whereas particles formed in the second process usually result from reaction of gases, then nucleation to form secondary particles. Particle growth in the atmosphere occurs through gas-particle interactions, and particle-particle infraction.

1.3 Recent studies (11-12) involving particle transport and transformation suggest strongly that atmospheric respirable particulate matter commonly occurs in two modes. The fine or accumulation mode is attributed to growth of particles from the gas phase and subsequent agglomeration, while the coarse mode is made up of mechanically abraded or ground particles. Particles that have grown from the gas phase, either because of condensation, transformation or combustion, occur initially as very fine nuclei 0.05 μm in size. Those particles tend to grow rapidly to accumulation mode particles around 0.5 μm in size which are relatively stable in the air. Because of their initially gaseous origin, this range of particles sizes includes inorganic ions such as sulfate, nitrate, ammonia, combustion-form carbon, organic aerosols, metals (Pb), cigarette smoke by-products, and consumer spray-products.

1.4 Coarse particles, on the other hand, are mainly produced by mechanical forces such as crushing and abrasion. Coarse particles therefore normally consist of finely divided minerals such as oxides of aluminum, silicon, iron, calcium and potassium. Coarse particles of soil or dust mostly result from entrainment by the motion of air or from other mechanical action within their area. Since the mass of these particles are normally >3 μm, their retention time in the air parcel is shorter than the fine particle fraction. Table 1 outlines the chemical constituents of the fine and coarse modes.

1.5 The composition and sources of coarse particles are not as thoroughly studied as those of fine particles. One reason is that course particles are more complex than fine particles

but similar to each other in chemical composition. It is possible, however, to recognize dozens of particle types, based on microscopical examination; these range from soil particles, limestone, flyash, oil soot to cooking oil droplets.

1.6 Outdoor concentrations of TSP, more specifically, are of major concern in estimating air pollution effects on visibility, ecological and material damage; however, people spend the majority of their time inside buildings or other enclosures.

1.7 Consequently, based upon the health effects of coarse and fine particulate matter, a method (14-17) has been developed to determine both coarse (>2.5μm to 10μm) and fine (<2.5μm) particulate matter in indoor air. A Microenvironmental Exposure Monitor (MEM) has been developed as a fixed site monitor. Similarly, Personal Exposure Monitors (PEMs) have been developed (18-20) to estimate personal exposure to particles. The PEMs can be connected to the participants lapel and are used in conjunction with personal pumps.

1.8 This method may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Applicable Documents

2.1 ASTM Standards

D1356 Definitions of Terms Relating to Atmospheric Sampling and Analysis.
D1605 Sampling Atmospheres for Analysis of Gases and Vapors.
D1357 Planning the Sampling of the Ambient Atmosphere.

2.2 Other Documents

U.S. Environmental Protection Agency Technical Assistance Document (21)
Laboratory Studies for Monitoring Development and Evaluation (22-31)

3. Summary of Method

3.1 For monitoring indoor air, two distinct samplers have been illustrated in this procedure. The Microenvironmental Exposure Monitor (MEM) has been developed as a fixed site monitor, while the Personal Exposure Monitor (PEM) has been developed to estimate personal exposure to particles. In addition, the PEMs have been used in the Particle Total Exposure Assessment Methodology (Particle-TEAM) Program underway by the U.S. Environmental Protection Agency (32). One of the objectives of the Particle-TEAM is to establish the level of human exposure to particles and relate exposure to sources of aerosol matter through the application of the PEMs.

3.2 Both systems operate on the principle of impaction. A constant flow (4 Lpm) particulate laden gas stream enters the impactor assembly. The design of the impactor allows the particulate matters to be fractionated into the desired ranges of fine respirable

[<2.5 μm] or inhalable fraction [<10.0μm]). The flow rate through the sample coupled with the impactor design characteristics enables the particulate matter to be speciated. The 4 Lpm flow rate was chosen because it was technically achievable with both a battery powered flow controlled pumping system (ideal for the PEMs) and a line powered system (ideal for the MEMs). In a typical sampling program, the flow rate will allow a total sample volume of 5.5 m^3 per day, thus facilitating improved accuracy in gravimetric measurements for a typical indoor particulate loading air parcel.

3.3 A volume of air is accurately drawn for a measured period of time through the impactor assembly to a tared filter.

3.4 The total particulate matter loading is calculated from the weight gain of the filter and the total volume of air sampled.

4. Significance

4.1 When sampling particles for subsequent chemical/elemental analysis and possible association with human health effect, characterizing reliable size separation is important. Size fractionation of deposited particles occurs in the respiratory tract during inspiration. Further, physical and chemical processes result in bi- or tri-modal distribution of suspended particles in the atmosphere.

4.2 Because alkaline particles tend to be greater than 3 μm in diameter and acidic particles tend to be less than 1 μm, a sharp size separation in this range would be desired to prevent neutralization of acidic aerosols collected on a filter. Further, the distinct separation of particle mass by size permits source resolution by multivariate statistical analysis techniques using the elemental and chemical composition of the fine fraction particle mass.

4.3 For these reasons, it is imperative that a sampling protocol addressing the sampling and analysis of speciated particulate matter in indoor air be developed.

5. Definitions

Note: Definitions used in this document and any user prepared Standard Operating Procedures (SOPs) should be consistent with ASTM Method D1356. All pertinent abbreviations and symbols are defined within this document at point of use. Additional definitions, abbreviations, and symbols are located in Appendices A-1 and B-2 of this compendium.

5.1 Particulate mass - a generic classification in which no distinction is made on the basis of origin, physical state, and range of particle size. (The term "particulate" is an adjective, but it is commonly used incorrectly as a noun.)

5.2 Dust - dispersion aerosols with solid particles formed by comminution or disintegration without regard to particle size. Typical examples include 1) natural minerals suspended

by the action of wind, and 2) solid particles suspended during industrial grinding, crushing, or blasting.

5.3 Smokes - dispersion aerosols containing both liquid and solid particles formed by condensation from supersaturated vapors. Generally, the particle size is in the range of 0.1 μm to 10 μm. A typical example is the formation of particles due to incomplete combustion of fuels.

5.4 Fumes - dispersion aerosols containing liquid or solid particles formed by condensation of vapors produced by chemical reaction of gases or sublimation. Generally, the particle size is in the range 0.01 μm to 1 μm. Distinction between the terms "smokes" and "fumes" is often difficult to apply.

5.5 Mists - suspension of liquid droplets formed by condensation of vapor or atomization; the droplet diameters exceed 10 μm and in general the particulate concentration is not high enough to obscure visibility.

5.6 Primary particles (or primary aerosols) - dispersion aerosols formed from particles that are emitted directly into the air and that do not change form in the atmosphere. Examples include windblown dust and ocean salt spray.

5.7 Secondary particles (or secondary aerosols) - dispersion aerosols that form in the atmosphere as a result of chemical reactions, often involving gases. A typical example is sulfate ions produced by photochemical oxidation of SO_2.

5.8 Particle - any object having definite physical boundaries in all directions, without any limit with respect to size. In practice, the particle size range of interest is used to define "particle". In atmospheric sciences, "particle" usually means a solid or liquid subdivision of matter that has dimensions greater than molecular radii (~10 nm); there is also not a firm upper limit, but in practice it rarely exceeds 1 mm.

5.9 Aerosol - a disperse system with a gas-phase medium and a solid or liquid disperse phase. Often, however, individual workers modify the definition of "aerosol" by arbitrarily requiring limits on individual particle motion or surface-to-volume ratio. Aerosols are formed by 1) the suspension of particles due to grinding or atomization, or 2) condensation of supersaturated vapors.

5.10 Total suspended particulate (TSP) mass - the particulate mass that is collected by the Sampler. (The system is classified in terms of the operational characteristics of the sampler).

5.11 Coarse and fine particles - these two fractions are usually defined in terms of the separation diameter of a sampler. Coarse particles are those with diameters of 2.5 μm to 10 μm and the fine particles are those with diameters less than 2.5 μm.
Note: Separation diameters other than 2.5 μm have been used.

6. Method Limitations and Limits of Detection

6.1. The limitations on the test method are a minimum weight of 20 micro grams of particles on the filter, and a maximum loading of 600 micro grams/cm^2 and minimum of 20 micro grams/cm^2 on the filter.

6.2 The test method may be used at higher loadings if the flow rate can be maintained constant (± 5%) and degradation of the aerosol preclasifier performance is not adversely affected.

6.3 The MEM and PEM samplers' limit of detection (LOD) is a function of the weighing room environment and the precision of the microbalance used to perform mass measurements.

6.4 Using the recommended equipment specified in this procedure, a 12-hour LOD of 8 $\mu g/m^3$ can be achieved for the PEM, and 4 $\mu g/m^3$ for the MEM.

6.5 Overall precision is ± 2 $\mu g/m^3$ to ±25 $\mu g/m^3$ during dust loading studies (10 to 100 $\mu g/m^3$) at a flow rate of 4 L/min. for each sampler.

7. Apparatus Description

7.1 Microenvironmental Exposure Monitor (MEM) Description

7.1.1 As illustrated in Figure 2, the MEM is subdivided into four sections: 1) an inlet section, 2) a three-piece inertial impaction section, 3) the upstream section of the filter holder; and 4) the downstream section of the filter holder.

7.1.2 Inlet section - the inlet section has four large, circumferential slots for aerosol to enter the MEM. These horizontal inlet slots prevent very large particles, perhaps those greater than 100-μm aerodynamic diameter, from entering the MEM and placing an additional particle burden on the downstream impaction plate. The inlet section also acts as a cover, preventing large particles from entering the MEM by gravity settling. The inlet section should be shown to be unbiased with respect to the particle size distribution being sampled.

7.1.3 Impaction section - the impaction section consist of three separate parts: 1) a nozzle, 2) an impaction plate(s), and 3) a part designed for mounting the impaction plate. Two versions of the impactor assembly are available. With a one stage impactor plate assembly, aerodynamic particles of <10 μm are allowed to pass around the impactor plate and subsequently collected in the lower filter. With the two stage impactor assembly, as illustrated in Figure 2, those particles <2.5 μm are collected on the lower filter. A time share option provides the capability of using two heads with one pumping system. In this way, the total sampling time can be programmed to two samplers, enabling the collection of <2.5 μm and <10 μm particulate matter in the same general environment. These features could be used to sample in two locations or to collect carbon on quartz filters or acid aerosols through a unit equipped with an ammonia denuder.

7.1.3.1 Nozzle - a single circular nozzle with a converging inlet and cylindrical throat to accelerate the aerosols through the nozzle to the filter. Two nozzle sizes are available: a nozzle with a throat diameter of approximately 8 mm, is used for removing particulate matter with an aerodynamic diameter greater than 10 μm; while smaller particles are collected on the downstream filter and saved for analysis. An approximate 3 mm diameter nozzle is used for collecting particulate matter with an aerodynamic diameter greater than 2.5 μm; while smaller particles are collected on the downstream filter and saved for analysis.

7.1.3.2 Impaction plate - a stainless-steel sintered disk is permanently mounted at the center of the impaction plate, flush with the impaction plate's surface. The pores of the sintered disk are filled with a light mineral oil in order to reduce bounce when the particles impact. The oil also wicks up through the particle deposit by capillary action so that a sticky surface continues to be available to incoming particles. The airstream containing the remaining smaller particles flows around the impaction plate through three large annular slots.

7.1.4 Upstream section of the filter holder - the upstream section of the sampler provides a flow-straightening zone directly downstream of the impactor plate so that uniform particle deposition on the filter is obtained.

7.1.5 Downstream section of the filter holder - in addition to acting as the downstream side of the filter holder, this section contains a plenum through which the filtered air exits via a side-mounted exit tube. It is also the MEM's base, which provides a surface on which the MEM can sit in the correct orientation.

7.1.6 Filter mounting and support - a 2-μm pore-size, PTFE (Teflon®), 41 mm filter disk with a polyolefin ring (Teflo #R2JO37 Gelman or equivalent) is mounted in a 2-inch x 2-inch standard Beckman-type frame and is used as the filtration medium. The downstream side of the filter is supported by a cellulose backing material (millipore AP-10 or equivalent). The two sections of the filter holder forming the filter assembly each have silicon rubber gaskets. Two draw latches hold the filter assembly together, compressing the two rubber gaskets, the cellulose backing material, and the polyolefin ring. This arrangement seals the filter assembly and prevents bypassing of the aerosol around the edge of the filter. The filter should be non-hygroscopic and should have a collection efficiency greater than 99% for the particle laden air stream of interest. The filter should be 37 mm in diameter.

Note: As an example, some glass fiber and most membrane filters with nominal pore size of 2 micrometers will nearly always fulfill this requirement. The equilibrated filter is preweighed by the user. The weight of the filter holder is not used in any determination of weight gain in this test method. The filter holder material must not contribute to any weight change of the filter.

7.1.7 Flow calibration section - to measure the volumetric flow through the MEM in the field, the inlet section is replaced with an adapter that connects via rubber tubing to a calibrated rotameter.

7.1.8 Pump - a sampling pump with a flow rate that can be determined accurately to ± 5%. Pulsation in the pump flow must be within ± 10% of the mean flow. The pump must maintain the flow constant to within ± 5% during the sampling period. The pump must be quiet enough so as to not cause undue disturbance in the area of use when being used indoors. The pumping unit has four components: a pump, a mass flow meter, a flow control circuit, and a timer. The system should be designed to provide constant flow by means of a voltage control system. The voltage control system should be designed to keep the flow at a constant 4 Lpm. The pump should be capable of maintaining up to a vacuum of 50 inches of water at the 4 Lpm flow rate. This is important since air flow to the impactor must not change as the filter loading increases. The mass flow meter should consist of a heated filament and an electrical circuit that measures the flow by determining how much heat is removed per second. If the flow is reduced (perhaps due to increased pressure drop across the impactor), the feedback circuit should apply a greater voltage to the pump to bring the flow back to the set point. A fan should be used to dissipate heat generated by the pump. The box cover should be closed and the fan running during use to maintain the accuracy of the control circuit.

7.2 Personal Exposure Monitor (PEM) Description

7.2.1 The PEM is illustrated in Figure 3 and consists of three sections: 1) an inlet-nozzle section, 2) an impactor plate, and 3) exit section.

7.2.2 Inlet and nozzle section - aerosol enters through six nozzles located on the inlet section's upstream surface, which is perpendicular to the direction of flow. Two inlet-nozzle sections are available: one has a throat diameter of approximately 1.8 mm for particulate matter cut size of <10 μm, and the other has a throat diameter of approximately 1.3 mm for particulate matter cut size of <2.5 μm.

7.2.3 Annular impactor plate - a stainless-steel sintered annulus is permanently mounted in the impaction plate, flush with the impaction plate's surface. The pores of the sintered annulus are filled with a light mineral oil in order to reduce bounce when the particles impact. The oil also wicks up through the particle deposit by capillary action so that a sticky surface continues to be available to incoming particles. The airstream containing the remaining smaller particles flows through the circular opening in the center of the impaction plate. The downstream circular edge of the impaction plate compresses the upstream face of the filter and backing material.

7.2.4 Exit section - the retaining lip of the exit section compresses the downstream face of the filter and backing material against the impactor plate edge, thereby preventing leakage and filter bypass. The exit section has an exit plenum and side-mounted exit tube, which connects by tubing to the pump.

7.2.5 Filter and support - a 2-μm nominal pore diameter, PTFE (Teflon®), 37 mm membrane filter disk with polyolefin ring (#R2JO37 Teflo, Gelman or equivalent) is used as the filtration medium. It is supported on its downstream face by cellulose backing material (Millipore AP-10 037 or equivalent).

7.2.6 Flow calibration section - to measure the volumetric flow through the PEM in the field, an adapter, which connects via rubber tubing to a calibrated rotameter, is placed over the inlet nozzle section.

7.2.7 Pump - a 145 mm x 50 mm, tough, light, alloy case, which originally housed the Casella AFC 400 pump unit, which contains the muffled double acting diaphragm pump, integral motor, and pulse dampener from the Casella AFC 400; the remainder of the components were removed and replaced with sound-deadening material.

7.2.8 Electronics section - flow should be maintained constant within a tolerance of 5% by means of an electronic control circuit using current proportional feedback. When the pressure drop across the filter increases, this system should automatically sense the rising current demand by the motor and adjust its voltage to compensate. The electronics case should also house a digital electronic elapsed timer, the LED that indicates when the pump is running, and the electronics that automatically shut off the pump if the battery is weak.

7.2.9 Battery section - the battery pack should contain 3 or 4 lithium 9-volt batteries with snap-on connectors, allowing quick battery replacement.

7.3 Cahn Microbalance

7.3.1 The Cahn Model 30 balance is capable of weighing up to 3.5 g with an accuracy of ± 0.5 μg. It operates on the principle of balancing the sample with torque motor input. The electric current flowing in the torque motor produces an equal and opposite force on the balance beam when the beam is at the reference position, identified by a photocell detection system. The current is directly related to the sample weight through the calibration process.

7.3.2 The same analytical microbalance and weights must be used for weighing filters before and after sample collection.

7.4 Weighing Room Environment

The weighing room should be a temperature and relative humidity controlled environment. Temperature should be maintained within the range of 17° to 23°C. Relative humidity should be maintained between 38% and 42%. Weekly strip chart recordings of temperature and humidity should be maintained on a hygrothermograph. Temperatures should be read from a calibrated maximum-minimum thermometer and relative humidity should be calculated from a calibrated motor aspirated psychrometer. The weighing area should be cleaned with paper towels and deionized distilled water each day before weighing. Forceps should be cleaned once a week with detergent in a sonic bath and then rinsed in deionized distilled water. Approximately once a month, the balance chamber and pans should be cleaned with diluted ammonium hydroxide and each cleaning should be noted in the weighing room log. Filters, weights, and pans should be handled only with non-serrated tip forceps. The Cahn balance should be left on continuously because it requires six hours to warm up for stable operation. Polonium 210 alpha sources should be replaced at one year intervals from date of manufacture. The replace date should be engraved on the source by

the manufacturer, and noted in the weighing room log book. The filters should be conditioned in the weighing room for at least 24 hours before they are weighed. Each filter should be passed over a deionizing unit before weighing.

8. Apparatus Listing

8.1 Microenvironmental Exposure Monitor (MEM)

8.1.1 Sampler - William Turner, Air Diagnostics and Engineering, Inc., R.R. 1, Box 445, Naples, Maine.

8.1.2 Barometer, capable of measuring atmospheric pressure to ±0.13 kPa, best source.

8.1.3 Stopwatch, capable of measuring to ±0.1 s, best source.

8.1.4 Weighing room, with temperature and humidity control to allow weighing with a micro balance to ±5 micro grams.

8.1.5 Analytical micro balance, capable of weighing to ±5 μg.

Note: Particular care must be given to the proper zeroing of the balance.

8.1.6 Buret, capacity of 1 L, used as a soap bubble meter for calibration of the sampling unit. At flows greater than 5 L/min., a transfer standard must be employed which is traceable to a primary standard. Examples of transfer standard include wet test meter, dry gas meter, mass flow meter, rotameters, and linear flow meter.

8.1.7 Plane-parallel press, capable of giving a force of at least 1000N (may be required if plastic filter holders are used that must be pressed together after insertion of the filter).

8.1.8 Tapered tube flow meter, with precision ±2% or better within the range of the flow rate used. It shall be possible to connect the suction side of the flowmeter to the inlet of a leakproof container which contains the sampling head (in order to measure the flow rate before and after sampling).

8.1.9 Thermometer, dry bulb, 0 to 50°C with divisions every 0.1°C.

8.1.10 Manometer, 0 to 250 mm of water for measuring the pressure drop across the sampling head.

8.1.11 Flexible tube, the length of the tube is dependent on how the sampling unit is placed. A length of 1 to 10 m is suitable if the pump is separated from the sampling head.

8.1.12 Inlet adapter or leakproof container (holds partial vacuum of 4 psi for 5 min.) of suitable size to contain the sampling head.

8.1.13 Impactor base - ability to hold two types of Membrana Inc., Ghia., 2" x 2" PTFE filters holders.

8.1.14 Filters - 37 mm, 2.0 μm pore size, Membrana Inc., Ghia., filters.

8.1.15 Removable filter disks, i.e., 2.0 μm pore size PTFE disks with polyolefin rings and special flat spots mounted in 2" x 2" standard Beckman frames. (Ghia #R2PJO41 with special cut). These filters have historically been used in the Beckman type automatic dichotomous sampler by the U.S. EPA.

8.1.16 A one-week timer with 84 set points in 2-hour increments and battery backup.

8.1.17 Impactor classifier - 10 and 2.5 μm cut size.

8.2 Personal Exposure Monitor (PEM)

8.2.1 Sampler - Virgil Marple, MSP Corp., 1313-5th St. SE, Suite 206, Minneapolis, Minnesota 55414.

8.2.2 Filter - 2 μm nominal pore diameter, PTFE, 37 mm membrane filter disk with polyolefin ring (#R2JO37 Teflo, Gelman or Equivalent).

8.2.3 Filter support - cellulose backing material, Millipore AP-10 037 or equivalent.

8.2.4 Pump - Casella AFC 400 pump unit or DuPont P125-A constant flow pump.

8.2.5 Analytical micro balance - refer to Section 8.1.5.

8.2.6 Buret - refer to Section 8.1.6.

9. Filter Preparation

9.1 Overview

9.1.1 All filters are conditioned in the balance room for at least 24 hr. before initial or final weighing to reduce the humidity effects on the filter weights. The 37 mm filters should be stored in individual petri dishes after initial weighing.

9.1.2 A Cahn microbalance with electronic data transfer capability should be used to weigh the 37 mm filters used in the PEM and MEM samplers. A Cahn Model 31 balance should be connected to a Compaq portable computer through a serial port. Filter numbers are printed in bar code and assigned to filter containers. In operation, the filter number are scanned with a bar code reader and the filter placed on the balance pan. A key is then pressed on the computer keyboard to indicate that the filter is in position for weighing. The computer sends the balance a request to weigh. The balance responds with weight and stability code. The operator is signaled by a tone and a message on the computer screen when weighing is completed. The operator then removes the filter and places it back in its container. The process is repeated for each filter to be weighed. The initial weight, time, and data are written to the data file by the computer.

9.1.3 After the filter has been used, it is brought back for conditioning and final weighing. The weighing procedure is the same as for initial weighing. The computer will check the data file for the initial weight entry. The final weight will be matched with the initial weight for that filter number in the data file. The computer subtracts the initial weight from the final weight to determine the particulate catch, which is used to calculate the particulate concentration (in $\mu g/m^3$) at each sampler location. After weighing, the filters are carefully returned to the petri dishes for archiving or further analyses. Because the date and time are saved in the data file with each reading, a chronological history is therefore available for additional verification.

9.1.4 The filters must be pre-weighed before use in a temperature and humidity controlled weighing room. Since the objective of the sampling system is to determine mass particle loading of the indoor air, the filters do not need to be pre-treated.

9.1.5 Insure that the weighing room meets the specifications as outlined in Section 7.4.

9.2 Cahn Microbalance Operational Protocol

9.2.1 General - initiate a weighing session by typing operator name, balance room temperature, and relative humidity into the Compaq computer. Ensure that identical stirrups are attached to the "A" hang down loop and the "tare" hang down loop of the balance beam.

Note: The maximum weight that can be measured in this range is 250 mg. Teflon® 37 mm filters should weigh in the 80-100 mg range.

Note: An ionizing, static-eliminator unit should be in the bottom of the weighing chamber.

9.2.2 Balance zeroing - after checking that the two stirrups contain no sample and are clean, close the balance door and release the pan brake by pressing the "Brake" button. Press zero (0) and then ENTER on the computer. Wait for a computer tone, which indicates that weighing is completed.

9.2.3 Balance calibration - remove a 200 mg calibration weight from its container (using plastic tweezers) and place it on the sample stirrup ("A" loop). Close the balance door. Press "200" and then ENTER on the computer. Wait for the computer tone, which indicates that weighing is completed. Repeat the above procedure with a 90 mg calibration weight. Return the 90 mg calibration weight to its container.

9.3 Initial Filter Weighing

9.3.1 Put on a clean pair of lint-free gloves. Disposable latex gloves should not be used because of possible filter contamination with talcum powder inside the gloves.

9.3.2 Select a packet of pre-conditioned (minimum of 24 hours inside the weighing chamber), clean 37 mm Teflon® filters.

9.3.3 Select a series of pre-labeled petri dishes.

9.3.4 Using Teflon® tweezers, pick up the top filters and examine them over a black surface for holes or tears. Discard any filter with a hole or tear.

9.3.5 Pass each clean filter several times over the top of the static eliminator unit in the bottom of the weighing chamber.

9.3.6 Place the clean filter on the balance stirrup and close the door. Allow the weight display to stabilize.

9.3.7 Select a pre-numbered and labeled petri dish. Scan the label with the bar code recorder. Press the "w" key (for weigh) and then ENTER on the computer. Wait for the computer tone, which indicates weighing is completed.

9.3.8 Open the balance door. With tweezers, remove the filter from the balance pan and load it into the filter support.

9.3.9 Return the filter and its support to the corresponding petri dish, close, and secure with masking tape.

9.3.10 Place the tared filter, with petri dish, in a stack ready for field sampling.

9.3.11 Complete steps 9.3.4 through 9.3.10 for each filter to be initially weighed. After every tenth filter weighing, check the balance zero. The stable electronic readout should be 00.000 ± 00.004 mg. Check the balance calibration with 200 mg and 90 mg calibration

weights as illustrated above. The stable electronic display should read 90.000 ± 00.002 mg. If the balance zero and/or 200 mg or 90 mg standard weight calibration checks fall outside the limits described above, rezero/recalibrate the balance as outlined above, and reweigh the last ten filters. If the balance zero and 90 mg check are acceptable, continue to weigh the 37 mm Teflon® filters.

9.3.12 At the end of the weighing session, enter the balance scan, relative humidity, and temperature into the computer. Recheck the balance zero and 200 mg and 90 mg standard weights as outlined above.

9.3.13 Following the completion of a weighing session, a second individual as an auditor should select 10 percent of the filters (minimum of two) for reweighing. The second person should enter his or her name into the computer and complete the above steps for each filter to be reweighed. After all the selected filters have been reweighed, compare the initial weights recorded for each filter by both the auditor and the primary operator. If the difference between the two measurements exceeds 10 μg, the session is declared invalid, and the filters must be reweighed.

9.3.14 The first filter weighed in any batch is the batch blank and is stored in a petri dish in the weighing room. The batch blank is reweighed at the end of each batch and if it differs by more than 7 μg from the first weight, all the filters must be reweighed. If by more than 5 μg but less than 7μg, then all filters back to the last zero are reweighed.

9.4 Packaging Filters

9.4.1 After weighing, the filters are placed in the frames (with the flat edge of the filter matching the flat edge of the frames). A ring is then pushed in place on top of the filter. Care should be taken that the ring does not buckle and lies flat on top of the filter.

9.4.2 The filters are recorded in the field notebook with filter type, bar number, filter identification and initial weight.

10. Preparation of the MEM Impactor Assembly

Note: The following discussion relates to the MEM impactor assembly. All instructions are applicable to the PEM impactor assembly.

10.1 General

10.1.1 The preparation of an impactor takes place in three stages: 1) all impactor plates must be cleaned before use, 2) plates must be oiled, and 3) placed into the impactor underneath the nozzles.

10.1.2 The filter backings and the filters themselves are placed inside the base of the impactor. After assembly, the impactor is now ready for use.

10.2 Cleaning of Stainless Steel Impactor Plates

Note: The following protocols are designed for both laboratory and field cleaning situations.

10.2.1 Laboratory Environment

10.2.1.1 Remove impactor plates from impactor and place in beaker or plastic tube for cleaning. Mix laboratory detergent (Liquinox or equivalent) according to manufacturer's directions in hot (40-50°C) tap water just prior to washing. Make enough to immerse all plates to be cleaned.

10.2.1.2 Add enough detergent solution to cover all plates in the beaker or tub.

10.2.1.3 Soak for 10 minutes with intermittent gentle agitation. Remove them from the beaker.

Note: Rough handling will damage plate surface. Do not put them in an ultrasonic bath.

10.2.1.4 Check for any remaining visual deposit on the surface of the plates. If deposit remains, go back to Section 10.2.1.3 and repeat washing. If still not removed, deposit may need to be brushed off from each plate in the same detergent solution with a firm bristle brush.

10.2.1.5 Place clean but soapy plates into another beaker or tub. Rinse 2 or 3 times with hot tap water or until all trace of detergent is removed.

10.2.1.6 Rinse next with distilled-deionized water. Let sit for 6 minutes. Rinse a second time with distilled-deionized water.

10.2.1.7 Drain well. Place rinsed plates in a well-ventilated container (stainless steel or aluminum cage, or screen bottom plastic tub) and dry at 50-60°C MAXIMUM for 30 minutes or until dry.

Note: Do not exceed this temperature.

10.2.1.8 Store the cleaned, dry plates in a closed container. A zip lock bag is sufficient if handled gently.

10.2.2 Field Environment

10.2.2.1 Place the plates in a tub with two scoops of a powder detergent and cover the plates with hot water, making sure that the detergent is dissolved.

10.2.2.2 Let soak for 30 minutes, agitating frequently.

10.2.2.3 Rinse the plates thoroughly, drain, and place them in a clean tub and repeat Section 10.2.2.1 and Section 10.2.2.2.

10.2.2.4 After the second washing, rinse the plates again, drain, and place them in a clean tub to rinse.

10.2.2.5 Place the tub in a sink with the faucet running, let the water fill the tub and overflow into the sink for a few hours or until there is practically no more oil on the surface of the water. The plates should be agitated occasionally and the tub checked to see that its walls have not become oily or the oil may get onto the plates.

10.2.2.6 When the water appears to be cleared of oil, drain the plates and place them in a single layer, sintered disk side up, on a large cookie sheet.

10.2.2.7 Bake them in the oven at 200°F for about 3 hours, or until none of the plates appear damp.

10.2.2.8 Turn off the oven and leave the plates to cool in the oven.

10.2.2.9 When the plates cool, place them in a clean zip-lock bag marked "CLEAN".

10.2.2.10 There should be no dirt on the plates and no water in the cintered disk. If the cookie sheets are not large enough for the number of plates chosen, the excess wet plates can be left in a sealed zip-lock bag until the first batch is out of the oven.

10.3 Oiling of Impactor Plates

10.3.1 After drying, remove the plates from the zip-lock bag.

10.3.2 Place the plates on a clean, dry surface.

10.3.3 With the aid of an eye dropper, deposit light mineral oil on the surface of the impaction plate. Apply until excess is observed.

10.3.4 Using a pair of tweezers, tilt the plate to one side to allow excess mineral oil to drain from the plate. If after proper drying and application of the oil, the oil pools up on a plate, it is permissible to wipe off all the excess oil from the plate and still use the plate.

Note: The objective is to clean the plates of dirt and excess water to coat each plate with a uniform layer of oil.

10.3.5 Place the clean, oiled plates into the MEM sampler and secure.

11. Sampling

11.1 Placement of Filters in the MEM

11.1.1 Place the tared filter and filter support in the filter holder, close firmly with the two over-center draw latches.

Note: The filter holder consists of a base and a cover that presses the plastic filter slide between two gaskets.

11.1.2 The assembly should be suitably covered to avoid contamination prior to use.

Note: If other MEM assemblies are available, replace the unit as a whole without transferring filters under field conditions.

11.1.3 Clean and inspect the interior of the preclassifier (cover). If the inside surfaces are visibly scored, replace the classifier to insure that the design characteristics of the impactor are not altered.

11.1.4 Attach sampling pump unit to the MEM.

11.2 Initial Field Flow Check of Sampler

11.2.1 Run the sampler for approximately 10 minutes to stabilize the flow rate.

11.2.2 Detach the top of the impactor and replace it with a calibration adapter. Connect the adapter, using a small piece of tubing, to the calibrated rotameter. Start pump and record initial flow rate on the Field Data Sheet.

Note: Insure flow rate is acceptable to the monitoring protocol.

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11.2.3 Disconnect the ro[illegible]ter. With the pump still running, close off the filter inlet. Flow should stop in 10 to 15 s[illegible]ds or less if the system is leak free. If not, examine all connections and flexible tubing [illegible] leaks.

11.2.4 Check the meter box assembly for proper operation.

11.3 Placement of Sampler

11.3.1 The sampling head should be located in the area in which the particulate concentration is desired. During placement of the sampling head, care should be taken to prevent any extraneous debris from entering the head during sampling. Care should also be taken to avoid any restriction of the inlet. The sampler should be placed on a flat, stable surface at least 2 to 5 feet off the floor to prevent reentrainment of settled particles.

11.3.2 Initiate sampling by turning the pump on; allowing the pump to warm-up and set the flow rate according to the manufacture's instructions.

11.3.3 Record the flow rate and the start time on the Field Data Sheet which is provided in Figure 4.

Note: If the flow rate changes during sampling by more than ± 5%, record the change and the time of change (annotating the lapsed time). Reset the flow rate. If unable to reset the flow rate to the original setting, terminate sampling and note the reason for termination.

11.3.4 At the end of the sampling period, record the final flow rate and the stop time on the Field Data Sheet. Terminate sampling by turning the pump off.

11.3.5 If the sampler has an elapsed timer, record the elapsed time on the Field Data Sheet.

11.3.6 Calculate the sampling time (Final time - Initial time) to the nearest tenth of an hour.

Note: If the standard deviation of the run time is greater than 20% of the estimated run time, during the 24 hour sampling period, record the deviation on the Field Data Sheet.

11.4 Final Field Flow Check of Sampler

11.4.1 Check the final flow rate by attaching a calibrated rotameter to the outlet of the MEM unit.

11.4.2 Turn the unit on and record final flow rate on Field Data Sheet.

Note: The initial and final flow rates should be within ± 10%.

11.5 Changing Impactors

11.5.1 Change the sampled impactor by disconnecting the hose and reconnecting to the new, clean impactor.

11.5.2 Record impactor identification number, filter identification number, base number and filter batch number on the new Field Data Sheet.

11.5.3 Once again, connect a calibrated rotameter to the impactor and record initial flow rate on the Field Data Sheet.

11.5.4 If applicable, re-set programmable timer to desired setting.

11.5.5 If you have a limited supply of impactors, you can change the filters and the impaction plates in the field. You should have a box in which to store and transport the filters. NEVER touch the filters during changing. If you touch a filter, the sample captured on it may be no longer valid.

11.5.6 The following procedures are recommended if one wishes to change filters in the field or in the laboratory.

11.5.6.1 Carefully swab the outer surface of the filter assembly with a lintless paper towel moistened with water before opening the filter holder to minimize sample contamination.

11.5.6.2 Open the filter holder and carefully remove the filter from the holder with the aid of filter tweezers. Handle the filters very gently by the edge to avoid loss of dust. Transfer the filter to a petri dish with cover or suitable holder. Do not turn the filter upside down. Record all pertinent information on the Field Data Sheet.

11.5.6.3 Return dishes to weighing room for 24 hour equilibration.

11.5.6.4 If the whole filter assembly is returned to the laboratory, it should be returned in a suitable container designed to prevent sample damage in transit.

11.5.6.5 For each set of 10 or less samples, submit a blank sample. The filters and filter holders to be used as blanks are handled in the same manner as the samples except that no air is drawn through them. Label these as blanks.

12. Filter Recovery and Final Weighing

12.1 24 hour Filter Equilibration Period

12.1.1 After sampling, filters are returned from the field as a complete batch. As the filters are unpacked, the date received and the condition of the filters are noted on the accompanying Field Data Sheet and laboratory logbook. The filter containers are then placed on a tray with the covers loosened.

12.1.2 The trays are placed in a protected area of the filter room and allowed to equilibrate for a minimum of 24 hours. Final weighing of a filter must be performed on the same balance as the original weighing. The balance is zeroed and calibrated as before, and date, relative humidity, temperature, blank mass, and tare mass are recorded on the sample weighing form.

12.2 Filter Inspection

12.2.1 Scan the bar code label on the petri dish of the first 37 mm Teflon® filter to be weighed.

12.2.2 Using Teflon® tweezers, carefully remove the filter from its container.

12.2.3 Inspect the filter for holes and tears. Enter any tear/hold or other comment in the computer or on the Filter Data Sheet.

12.3 Final Weighing

12.3.1 Place the filter on the balance stirrup and close the balance door.

12.3.2 Press "w" and ENTER on the computer key board. Wait for the computer tone, which indicates that weighing is completed.

12.3.3 Open the balance door. Using tweezers, place the filter back into the corresponding petri dish, cover, and stack for archiving.

12.3.4 Complete Sections 12.3.1 through 12.3.3 for each filter during the final weighing process. After every tenth filter weighing, check the balance zero as in Section 9.2.2. The electronic readout should be 00.000 ± 00.004 mg. Check the balance calibration with a 200 mg and a 90 mg calibration weight as in Section 9.2.3.

12.4 Independent Audit of Weighted Filters

12.4.1 Following the completion of a weighing session, a second individual as an auditor should select 10 percent of the filters (minimum of two) for reweighing.

12.4.2 After all the selected filters have been reweighed, compare the final weights recorded for each filter by the auditor and the primary operator.

12.4.3 If the difference between the two measurements for any filter exceeds 10 μg, the session is declared invalid, and the filters must be reweighed.

12.4.4 If the difference in independent final weights is less than 10 μg, the auditor should enter his or her name into the computer, indicating valid weights. The 37 mm Teflon® filters should then be archived for future evaluation.

13. Calculation

13.1 Mass of Particles found on the sample filter:

$$M_S = (m_2 - m_1) - m_3$$

where:
M_S = mass found on the sample filter
m_1 = tare weight of the clean filter before sampling, μg
m_2 = the weight of the sample-containing filter, μg
m_3 = the mean value of the net mass change found on the blank filters, μg
Note: The blank filters must be subjected to the same equilibrium conditions.

13.2 The sampled volume is:

$$V_S = Q \times t/1000$$

where:
V_S = the volume of the air sampled, m^3
Q = the mean indicated flow rate of air sampled, L/min
t = the sampling time, min
1000 = conversion from L to m^3
Note: There are no temperature or pressure corrections for changes in sampled volume since it is critical that the flow rate required for the preclassifier be set at the time and location of sampling. Additional adjustments to the tared filter weight may be necessary

to improve the method's accuracy at very low filter weights. These can be developed by re-weighing the blank tared filter weight periodically.

13.3 The concentration of the particulate matter in the sampled air is expressed in micro grams/m^3.

$$C = K \times M_S/V_S$$

where:
C = mass concentration of particulate matter, $\mu g/m^3$
K = a dimensionless correction factor for the preclassifier (supplied by the manufacturer if not equal to 1.0)
M_s = mass found on the sample filter (see Section 13.1), μg
V_S = the volume of air sampled, (see Section 13.2), m^3

14. Sampling System Calibration

14.1 The primary calibration involve the MEM or PEM samplers with sampling head, a bubble tube and pressure drop meters.

14.2 Assemble the calibration system as illustrated in Figure 5.
Note: Since the flow rate given by a pump is dependent on the pressure drop across the sampling device (filter and inlet), the pump must be calibrated while operating with a representative sampling inlet and filter.

14.3 Calibration of the sampling unit should be performed at approximately the same temperature and pressure that the sample will be collected; otherwise, appropriate temperature and pressure connections must be applied to the volume flow rate.

14.4 Place the sampling head, with the same type of filter to be used to collect the sample, in the calibration test apparatus. Connect the sampling head to the outlet of the test apparatus.

14.5 Turn on the pump and moisten the inside of the bubble meter by drawing bubbles up the meter until the bubbles are able to travel the entire length of the buret without bursting.

14.6 Adjust the sampling unit to provide the desired flow rate.

14.7 Start a soap bubble up the buret and measure with a stopwatch the time it takes the bubble to pass through a graduation of 1.0 L.

14.8 Repeat Section 14.7 at least three times, calculate the flow rate by dividing the volume of air between the preselected marks of the buret by the time required for the soap bubble to traverse the distance and average the results. If the measure flow rate is outside the specification, readjust as in Section 14.6, and repeat Sections 14.7 and 14.8.

14.9 Record the date of the calibration, the temperature, and barometric pressure at the time of the calibration on the Field Data Sheet and in the laboratory notebook.

15. Method Safety

This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the user's responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

16. Performance Criteria and Quality Assurance (QA)

16.1 Standard Operating Procedures (SOPs)

16.1.1 SOPs should be generated by the users to describe and document the following activities in their laboratory: assembly, calibration, leak check, and operation of hte specific sampling system and equipment used, preparation, storage, shipment, and handling of the sampler system, purchase, certification, and transport of standard reference materials and all aspects of data recording and processing, including lists of computer hardware and software used.

16.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the personnel conducting the monitoring.

16.2 Quality Assurance Program

The user should develop, implement, and maintain a quality assurance program to ensure that the sampling system is operating properly and collecting accurate data. Established calibration, operation, and maintenance procedures should be conducted on a regularly scheduled basis and should be part of the quality assurance program. Calibration procedures provided in Section 14, operation procedures in Sections 9-12, and maintenance procedures in Section 10 of this method and the manufacturer's instruction manual should by followed and included in the QA program. Additional QA measures (e.g., trouble shooting) as well as further guidance in maintaining the sampling system are provided by the manufacturer.

16.2.1 Sections 7.1 and 7.2 instruct the user to purchase instrumentation designed and calibrated to fractionate the particles in the gas stream.

16.2.2 Section 7.1.8 requires sampling pump to be accurate to ±5% and maintain flow to ±5% during the sampling period.

16.2.3 Section 7.4 requires the weighing room to be environmentally controlled: relative humidity maintained at 40 ±2 percent and temperature set at 20 ±3°C. In addition, a neutralizer is required to remove static charge on the filters.

16.2.4 Section 9.1.1 requires filters to be conditioned in the weighing room for at least 24 hrs. before initial and final weighing.

16.2.5 Section 9.2.2 requires the Cahn Microbalance to be zeroed and calibrated before and after a weighing session. The zero should be 00.000 ±00.004 mg, while the calibration should be within ±00.002 mg of standard.

16.2.6 Section 9.3.11 requires a check of zero after every tenth filter weighing.

16.2.7 Section 9.3.14 requires that the first filter weighed in any batch is the batch blank. The blank filter is reweighed at the end of each batch and if it differs by more than 00.007 mg from the first weight, all filters must be reweighed. If by more than 00.005 mg, then all filters back to the last zero are reweighed.

16.2.8 All filters must be recorded on the Field Data Sheet with filter type, bar number, filter identification and initial weight.

16.2.9 Section 11.2 requires an initial field flow check of the sampler.

16.2.10 Section 11.3.6 requires the run time to be within ±20% of estimated run time.

16.2.11 Section 11.4 requires a final field flow check of the sampler. The initial and final flow rates should be within ±10%.

16.2.12 Section 12.4 requires 10% of the filters (minimum of two) to be reweighed by a second, independent person. Differences between the two can not be any greater than 10 μg. If > 10 μg, session is declared invalid.

16.2.13 The Cahn Microbalancer must be audited once per month.

16.2.14 Section 14 requires the total sampling system be calibrated in the laboratory prior to field deployment.

16.2.15 The latest copy of the Quality Assurance Handbook for Air Pollution Measurement Systems (33) should be consulted to determine the level of acceptance of zero and span errors.

16.2.16 For detailed guidance in setting up a quailty assurance program, the user is referred to the code of Federal Regulations (8) and the EPA Handbook on Quality Assurance.

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Table 1. Chemical Constituents of the Coarse/Fine Mode Classification of Major Chemical Species Associated with Atmospheric Particles

Fine Fraction (<2.5 μm)	Coarse Fraction (2.5-10 μm)	Both Fine and Coarse Fractions	Variable
$SO_4^=$, C (soot), organic (condensed vapors), Pb, NH_4^+, As, Se, H^+	Fe, Ca, Ti, Mg, K, $PO_4^=$, Si, Al, organic (pollen, spores, plant parts)	NO_3^-, Cl^-	Zn, Cu, Ni, Mn, Sn, Cd, V, Sb

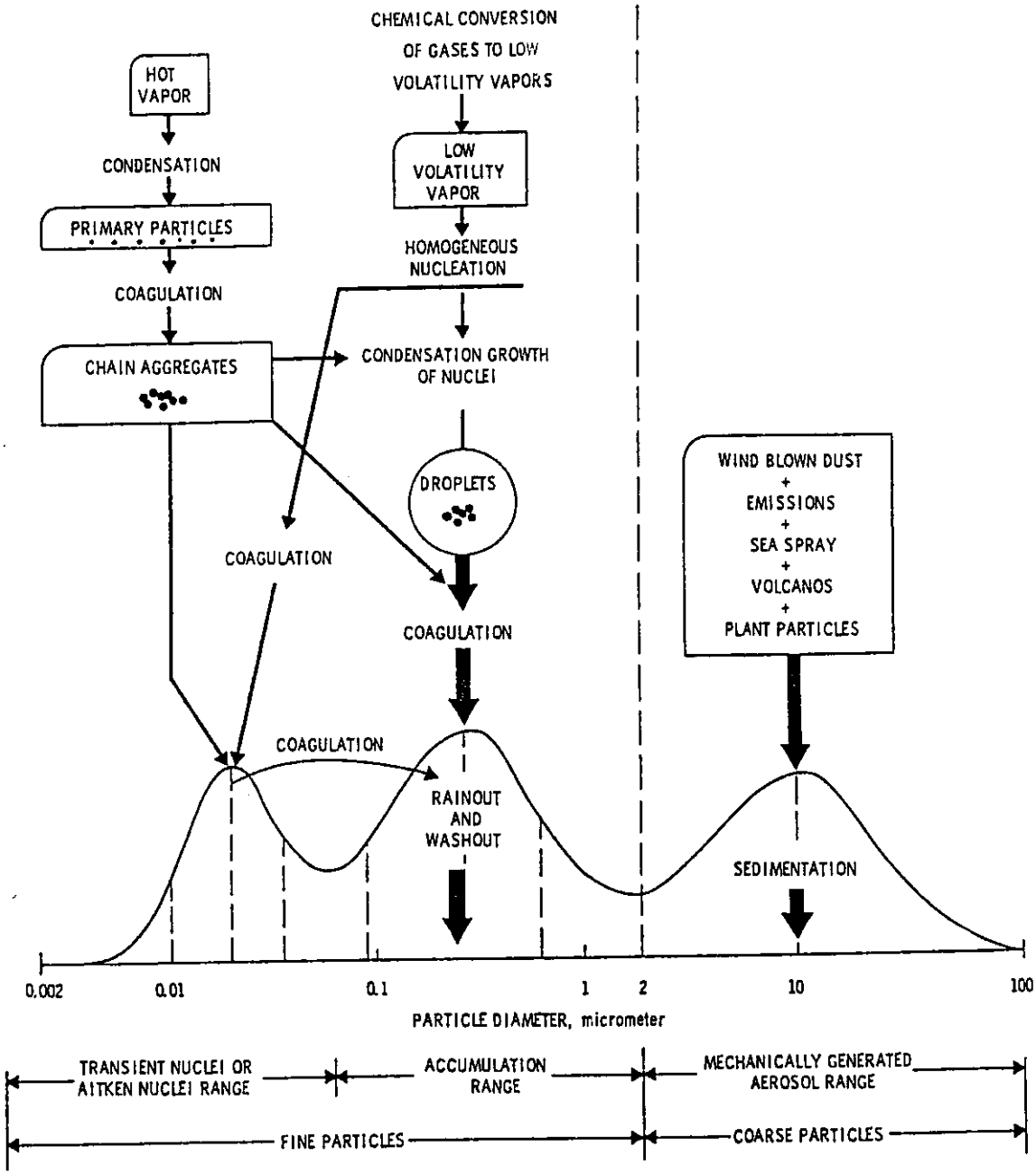

Figure 1. A Postulated Atmospheric Aerosol Formation Process

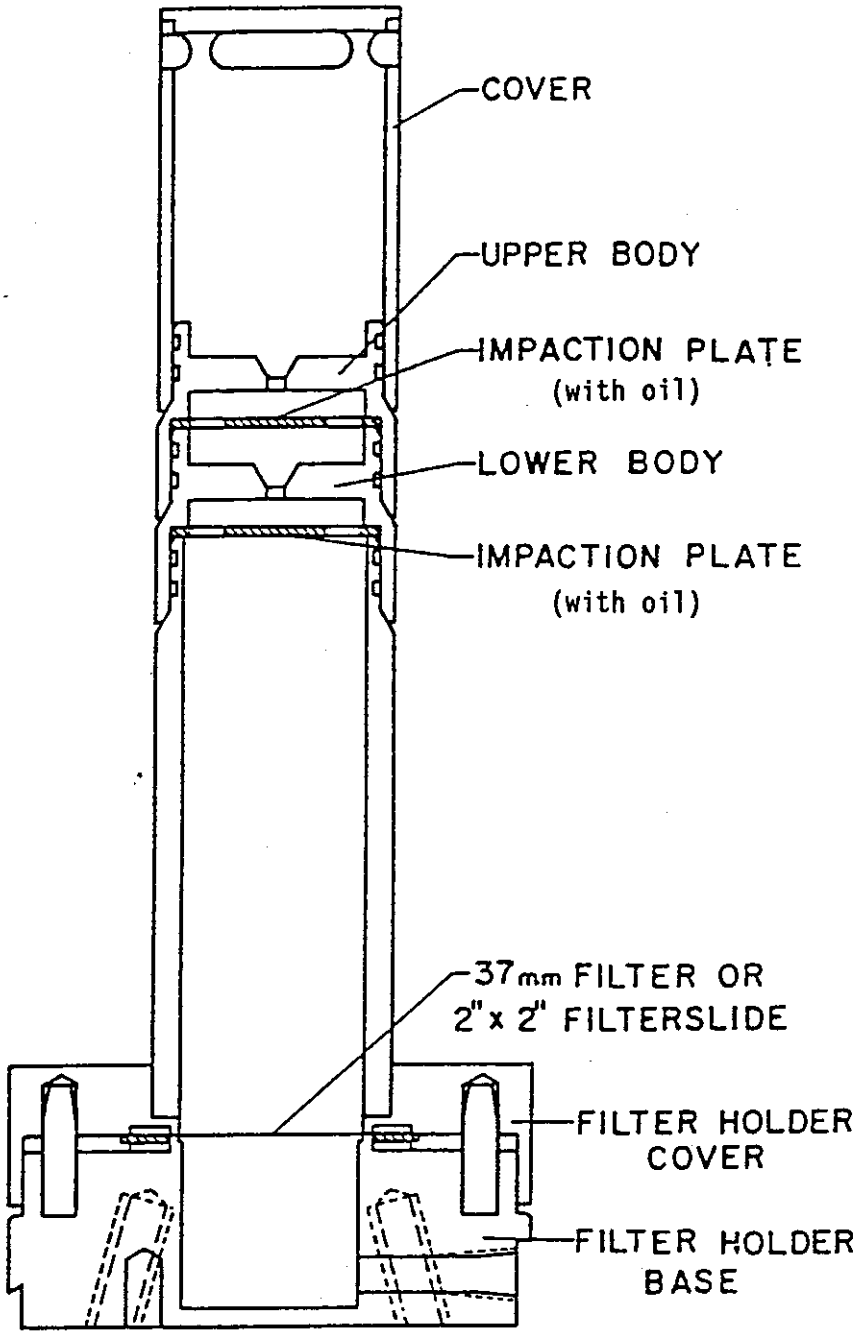

Figure 2. Schematic of Microenvironmental Exposure Monitor (MEMs)

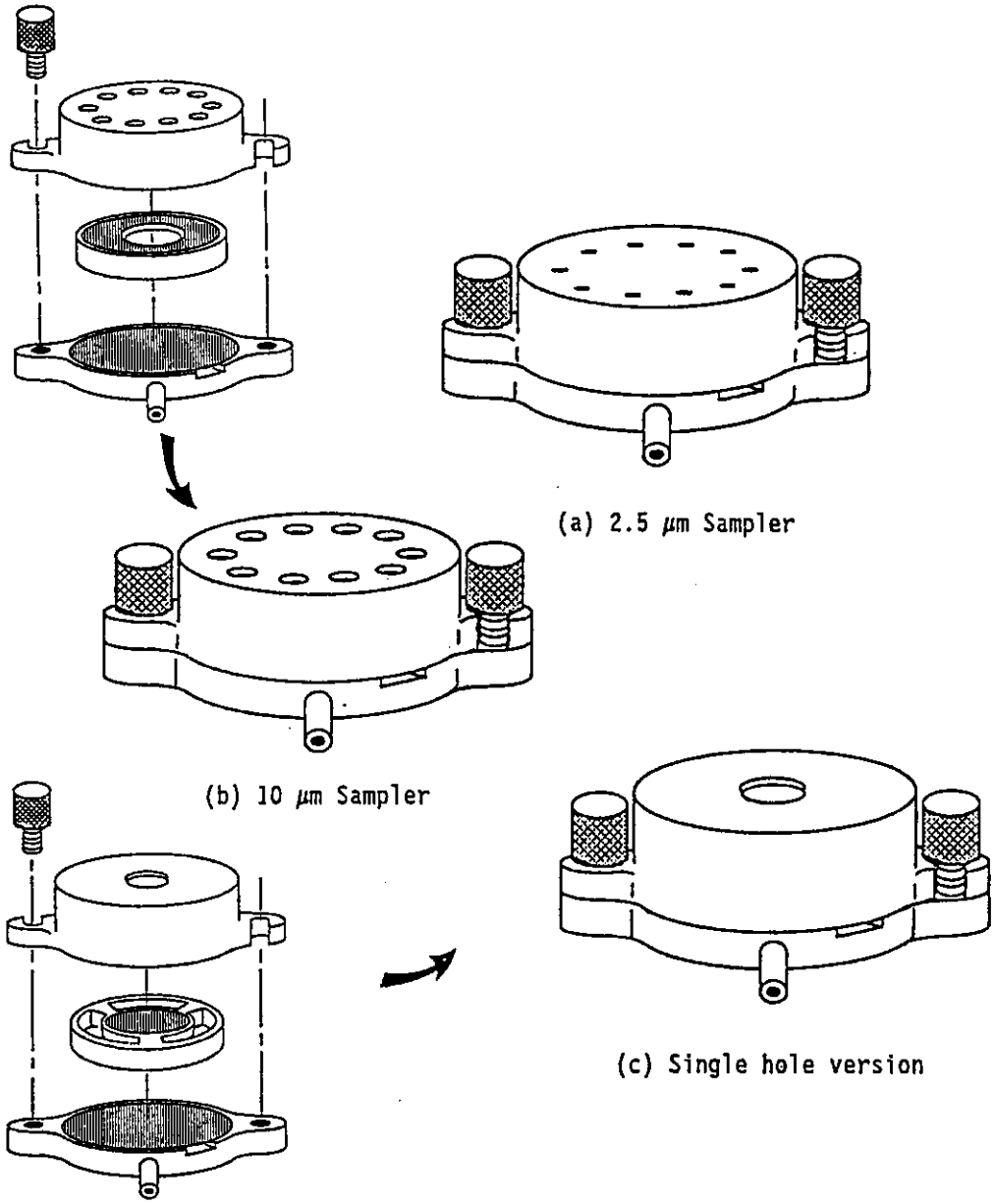

Figure 3. Schematic of Personal Exposure Monitor (PEMs)

[Examples illustrate exploded view of multiple orifices (a and b) and single orifice (c) approach. Each inlet consists of an impactor classifier, to remove particles larger than the predetermined cut size, and a filter to collect the remaining particles.]

DETERMINATION OF RESPIRABLE PARTICULATE MATTER

GENERAL

Project: _____
Site: _____
Location: _____
_____
_____
Sample Code: _____

Date: _____
Location of Sampler: _____
_____
_____
Operator: _____
_____

EQUIPMENT

Pump

Pump Model: _____
Serial No.: _____
Lab Calibration Date: _____
Flow Rate Set Point: _____
Calibrated by: _____

Sampler

Sampler: Particle fraction
MEM _____ 2.5 μm _____
PEM _____ 10.0 μm _____
Both _____

SAMPLING DATA

	Start	Stop
Time:	_____	_____
Flow Rate:	_____	_____
Temperature:	_____	_____
Pressure:	_____	_____
Avg. Flow Rate:	_____	_____

Run Time: _____ (± 20% of estimate)

Total Sample Vol.: _____
Flow Maintained Rate: _____ (± 5%)

Time	Flow Rate(Q) mL/min	Ambient Temperature °C	Barometric Pressure mm Hg	Relative Humidity,%	Comments

Figure 4. Field Sampling Data Sheet

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FILTER DATA

Filter I.D. No.: _____
Filter Bar No.: _____
Filter Case No.: _____
Filter Recorder in Laboratory Notebook: _____

WEIGHING ROOM

Atmosphere
Relative Humidity: _____ 40 ± 2%
Temperature: _____ 20 ± 3°C
Neutralizer: _____

Activity
Filters conditioned at least 24 hours: _____

Cahn Balance Zero: _____ ± 00.004 mg
After every 10th filter: _____

Cahn Balance Calibrated
- 200 mg _____ ± 00.002 mg
- 90 mg _____ ± 00.002 mg

Blank filter weight: _____

Reweigh at end: _____ ± 00.007 mg

10% of filters reweighed: _____
(no greater than 00.010 mg difference)

Cahn Balance last audited: _____ (once per month)

Figure 4 (cont'd.). Field Sampling Data Sheet

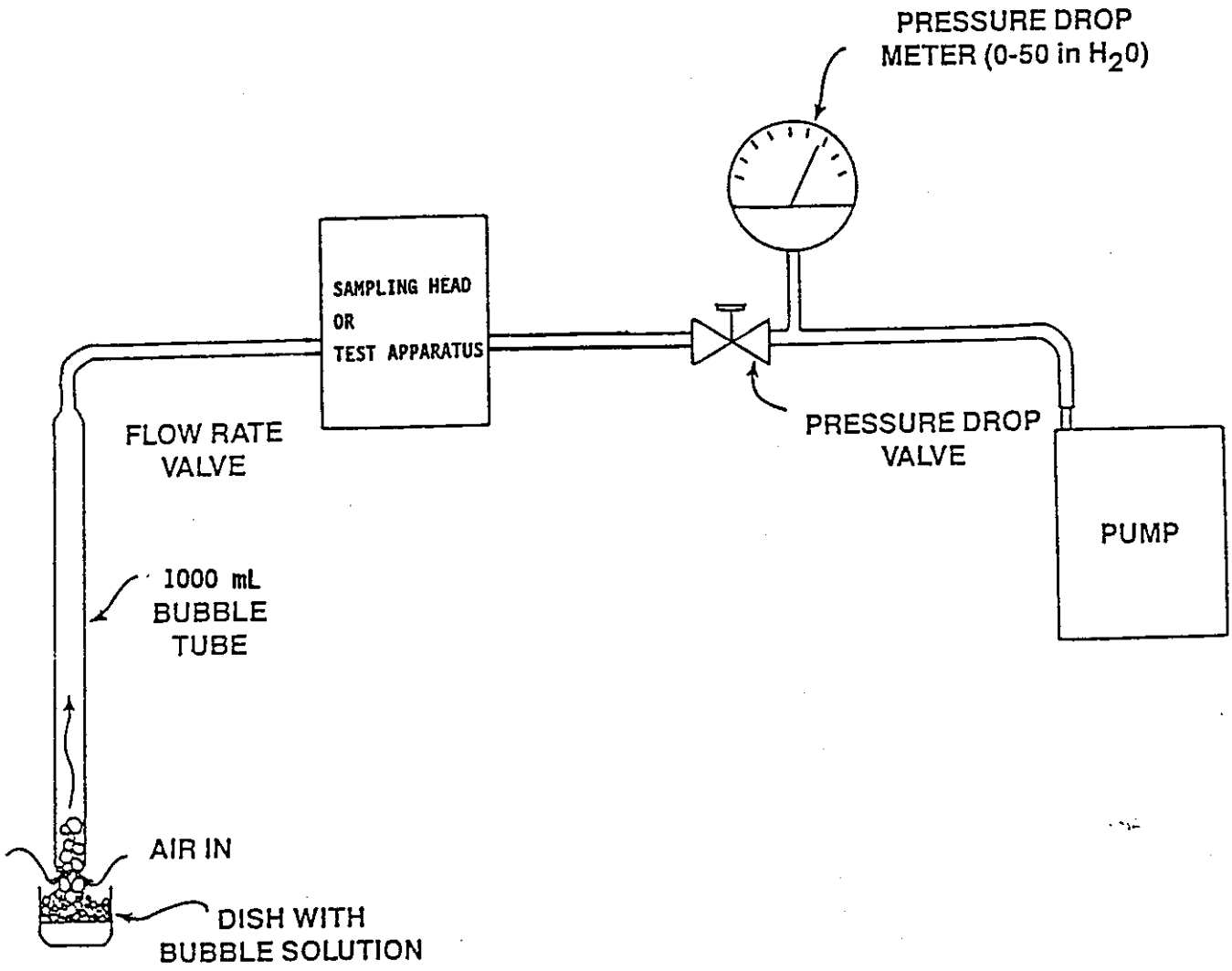

Figure 5. Calibration Assembly for Personal Sampling Pump

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Method IP-10B

DETERMINATION OF RESPIRABLE PARTICULATE MATTER IN INDOOR AIR USING A CONTINUOUS PARTICULATE MONITOR

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Appendix - TP3 Programming

Method IP-10B

DETERMINATION OF RESPIRABLE PARTICULATE MATTER IN INDOOR AIR USING A CONTINUOUS PARTICULATE MONITOR

1. Scope

1.1 This document describes the protocol for the Operation of a continuous particulate mass monitor which directly measures particulate mass at concentrations between 5 $\mu g/m^3$ and several g/m^3 on a real time basis.

1.2 The instrument calculates mass rate, mass concentration and total mass accumulation on exchangeable filter cartridges which are designed to allow for future chemical and physical analysis. In addition, the instrument provides hourly and daily averages.

1.3 The methodology detailed in this document is currently employed by such U.S. research organizations as the Argonne National Laboratory, R.J. Reynolds Tobacco Company and Philip Morris, Inc. for indoor and outdoor air quality studies, aerosol behavior studies, and cigarette smoke behavior studies.

2. Applicable Documents

2.1 ASTM Standards

D1356 Definitions of Terms Related to Atmospheric Sampling and Analysis

2.2 Other Documents

Technical Manuals (1-2)
Laboratory and Field Studies (3-12)

3. Summary of Method

3.1 Particle-laden air is drawn in through a heated air inlet followed by an exchangeable filter cartridge, where the particulate mass collects. The inlet system may or may not be equipped with the optional sampling head which pre-separates particles at either a 2.5 or 10 μm diameter.

3.2 The filtered air then proceeds through the sensor unit which consists of a patented microbalance system and an automatic flow controller.

3.3 As the sample stream moves into the microbalance system (filter cartridge and oscillating hollow tube), it is heated to the temperature specified by the software.

3.4 The automatic flow controller pulls the sample stream through the monitor at flow rates between 0.5 and 5 Lpm. The hollow tube is attached to a platform at its wide end and is vibrated at its natural frequency.

3.5 As particulate mass gathers on the filter cartridge, the tubes's natural frequency of oscillation decreases. The electronic microbalance system continually monitors this frequency.

3.6 Based upon the direct relationship between mass and frequency, the instrument's microcomputer computes the total mass accumulation on the filter, as well as the mass rate and mass concentration, in real time.

3.7 The data processing unit contains software which allows the user to define the operating parameters of the instrumentation through menu-driven routines.

3.8 During sample collection the program plots total mass, mass rate and/or mass concentration on the computer screen in the form of scales. The program allows two y-axis scales to be displayed and up to 10 variables to be plotted simultaneously. In addition, the scales and variables used in plotting the data may be changed during collection without affecting stored data. Figure 1 illustrates the assembled TEOM sensor unit and data processing unit.

4. Significance

4.1 Suspended particulate matter in indoor air is generally considered to consist of all airborne solid and low vapor pressure liquid particles. Suspended particulate matter in indoor air presents a complex multiphase system consisting of a spectrum of aerodynamic particle sizes ranging from below 0.01 microns (μm) up to 100 μm and larger. Historically, measurement of particulate matter (PM) has concentrated on total suspended particulates (TSP), with no preference to size selection. Research on the health effects of TSP in ambient and indoor air has focused increasingly on those particles that can be inhaled into the respiratory system, i.e., particles of aerodynamic diameter less than 10 μm. It is now generally recognized that, except for toxic materials, it is this fraction (< 10 μm) of the total particulate loading that is of major significance in health effects.

4.2 Particles are formed by two processes: 1) the grinding or atomization of matter (13-14), and 2) the nucleation of supersaturated vapors. The particles formed in the first process are products of direct emissions into the air, whereas particles formed in the second process usually result from reaction of gases, then nucleation to form secondary particles. Particle growth in the atmosphere occurs through gas-particle interactions, and particle-particle (coagulation) interaction.

4.3 Recent studies (15-16) involving particle transport and transformation suggest strongly that atmospheric particles commonly occur in two distinct modes. The fine or accumulation mode is attributed to growth of particles from the gas phase and subsequent agglomeration, while the coarse mode is made up of mechanically abraded or ground particles. Particles that have grown from the gas phase, either because of condensation, transformation or combustion, occur initially as very fine nuclei 0.05 μm in size. These particles tend to grow rapidly to accumulation mode particles around 0.5 μm in size which are relatively stable in the air. Because of their initially gaseous origin, this range of particle sizes includes inorganic ions such as sulfate, nitrate, ammonia, combustion-form carbon, organic aerosols, metals (Pb), cigarette smoke by-products, and consumer spray-products.

4.4 Coarse particles, on the other hand, are mainly produced by mechanical forces such as crushing and abrasion. Coarse particles therefore normally consist of finely divided

minerals such as oxides of aluminum, silicon, iron, calcium, and potassium. Coarse particles of soil or dust result from entrainment, by the motion of air or from other mechanical action within their area. Since the mass of these particles is normally > 3 μm, their retention time in the air parcel is shorter than the fine particle fraction.

4.5 The composition and sources of coarse particles are not as thoroughly studied as those of fine particles. One reason is that coarse particles are more complex and similar in chemical composition. It is possible, however, to recognize dozens of particle types, based on microscopical examination; these range from soil particles, limestone, flyash, oil soot to cooking oil droplets.

4.6 Outdoor concentrations of TSP, more specifically, are of major concern in estimating air pollution effects on visibility, ecological and material damage. However, people spend the majority of their time inside buildings or other enclosures; they breath indoor air and therefore, indoor concentrations dominate average exposure. To the extent that indoor concentrations are different from the outdoors, population exposures are different from those estimated by outdoor monitors.

4.7 Consequently, based upon the health effects of coarse and fine particulate matter, a continuous particulate monitor has been developed to allow mass measurement of particulate concentration on a real-time basis.

4.8 The monitor utilizes the filter-based measurement system for providing real-time mass monitoring capability.

5. Definitions

Definitions used in this document and any user prepared SOPs should be consistent with ASTM D1356. All abbreviations and symbols are defined with this document at the point of use.

6. Interferences

6.1 The instrument's primary operating mechanism is the microbalance system which relies upon changes in the frequency of an oscillating tapered element to determine changes in the particulate mass collected. Because of this characteristic, the instrument should be isolated from mechanical noise as much as practical. It should be located in the area to be measured so that external objects are not likely to contact or jar the instruments enclosure or the air sampling tube. Additionally, the instrument should be located in an environment with minimal temperature fluctuations. The units can operate effectively in environments with temperatures ranging between 7.2°C and 52°C.

6.2 Although the instrument may retrieve a sample from indoor or outdoor environments, it is important that the sample stream temperature is maintained within as narrow bounds as possible. Large abrupt temperature fluctuations (7-8°F/minute) of the sample stream may cause measurement accuracy to decrease due to the inlet systems inability to adjust the temperature of the sample to that specified by the software before travelling to the microbalance system. Sample temperature can range from ambient to 60°C.

Note: For aerosols such as cigarette smoke that may contain substantial fractions of dissolved semivolatiles, heatin e aerosol may decrease the apparent mass and may introduce errors into subseque emical analyses. As a precaution the TEOM may be operated at low inlet temperatu -30°C to 35°C).

7. Apparatus

The TEOM® Ambient Particulate Monitor is comprised of two main components (see Figure 1): the TEOM® Data Processing Unit and the TEOM® Sensor Unit. However, when purchased, these units are not fully assembled. Therefore, the following section describes the components contained in these two main units which are available separately as needed.

7.1 Enclosure cabinet - the enclosure cabinet (see Figure 2) houses a mass flow controller with an inline filter cartridge and silicone tubing, an electronic circuit chamber with the appropriate wiring for electricity and frequency signal output (inside left-covered by a plexiglass board).

7.1.1 Located on the outside right panel are the power, signals, microcomputer input/output and vacuum connections. The front of the metal door houses the ON/OFF switch and the pressure gauge which controls the mass flow controller. The inside of the door holds the silicone tubing which connects to the flow controller. The top wall of the enclosure cabinet contains a square hole (~3 in.) in the left side into which the sensor/preheater assembly fits.

7.1.2 The inside right side holds a toggle restraining clamp which secures the sensor/preheater unit when moving the unit small distances (R&P proprietary product).

7.2 Sensor/preheater assembly - the sensor/preheater assembly (see Figure 3) consists of the inlet and the microbalance.

7.2.1 The inlet consists of two concentric hollow (black) metal tubes. The outer tube is ~12" long and ~3" in diameter. The tip of the outer tube is configured to accommodate a 1/2" tubing for sampling or an additional sampling head, which separates particles by diameter allowing either $\leq$2.5 μm diameter or $\leq$10 μm diameter particles to enter the system. The base of the outer tube is welded to a rectangular metal mounting plate which is fixed to the top outside wall of the enclosure cabinet. The inner tube is connected to the outer tube at only one location to allow the microbalance to be suspended in the enclosure cabinet. The base of the inner tube is connected to the microbalance top outer wall. The connection accommodates an air temperature probe assembly which controls the temperature of the inner tube of the inlet.

7.2.2 The microbalance is a rectangular metal enclosure which houses a metal cylinder (the sensor head) the size of the inner inlet tube. The metal cylinder contains an oscillating tapered element, an electronic feedback system, and a filter cartridge. The tapered element is attached to a platform at its wide end (bottom) and has a small metal tip onto which the filter cartridge sits. The electronic feedback system consists of an amplifier board which maintains the elements oscillation and the electronics which allow frequency signals to be

transcribed to mass units. At the bottom of the microbalance, a silicone tube, which is connected to the mass flow controller, carries the air sample. Also attached to the bottom is the electrical cord. When purchased the whole unit is accompanied by a hardware manual which describes in detail the assembly and use procedures.

7.3 Filter cartridge - the filter cartridge (see Figure 4) is a half-inch diameter thin aluminum base (foil-like) assembly. The foil is crimped around the filter edges to contain it. Attached to the aluminum base is a water-resistant plastic cone which fits onto the metal tip of the oscillating element.

7.4 Filter exchange tool - the filter exchange tool (see Figure 4) is a four-inch long aluminum tube. The lower part of the tool has two perpendicular connections. The top connection is an aluminum disc which is slightly smaller than one-half inch in diameter. It is made to fit over the filter face when assembling and disassembling. The bottom connection is a "U-shaped" fork. The tines of the fork straddle the cone of the filter cartridge during assembling and disassembling.

7.5 Inline filter cartridge - standard filter cartridge, available from Fisher-Scientific.

7.6 Carbon-vane vacuum pump - oil-free pump with constant vacuum, available from Fisher-Scientific.

7.7 Microcomputer and keyboard - recommended IBM-compatible. The software should be able to plot real-time data on the screen and should give the user a number of options for saving data on disk, printing data, or transmitting information to other devices using analog or digital signals. The use of both hard disk and floppy disk systems should be available.
Note: The TEOM® is marketed and manufactured by Rupprecht and Patashnick Co., Inc., 8 Corporate Circle, Albany, NY, 12203. The following discussion addresses the receiving and setting-up of the monitor.

8. Assembly of Sensor Unit

The TEOM® Sensor Unit consists of two components: 1) the enclosure cabinet, and 2) the sensor/preheater assembly.

8.1 Remove both components from their shipping boxes. Set the enclosure cabinet upright in the designated location for the required sampling. Try to locate the enclosure cabinet at the source of the sample if possible (see Figure 2 for cabinet configuration).
Note: If the use of a sampling line cannot be avoided, keep its length to an absolute minimum and avoid sharp bends. Sampling line will cause some reduction in particulates reaching the microbalance. This, in turn, causing an underestimation of the sample content to be made.

8.2 Lay the sensor/preheater assembly flat on a table so that the shipping brace (the angle bracket painted red) faces upward. Remove the screws holding the shipping brace. When this bracket is removed, the air preheater tube flexes and allows the TEOM® Sensor Head

to drop until the air preheater tube touches the outer (3" ID) tube. Figure 3 illustrates the sensor/preheater assemblies.

8.3 Replace the cable and tubing support that connect the two side plates of the TEOM® Sensor Head using the 8-32 x 1/2" screws that were removed.

8.4 Make sure the TEOM® Sensor Head restraining clamp (the small orange handled toggle clamp) connected to the bottom right of the Sensor/preheater assembly is in its open (unclamped) position.

8.5 Carefully lift the sensor/preheater assembly. Hold it so that the air preheater tube is vertical and above the TEOM® Sensor Head. The long flange of the mounting plate should face left (i.e., the handle for opening the TEOM® microbalance should face toward you).

8.6 Carefully lower the sensor/preheater assembly through the square opening in the top of the enclosure cabinet (see Figure 3), making sure that the ribbon cable and vacuum tube precede the TEOM® Sensor Head through the opening. Line up the holes in the mounting flange with the threaded holes in the top of the enclosure, and secure with provided #10-32 x 3/8" screws.

8.7 Route the ribbon cable over the top of the power supply cover, which is behind the plexiglass printed circuit board cover, and plug its end (3-pronged) connector into the mating 25 pin connector (P12) at the printed circuit board cover.

8.8 Push the 1/4" vacuum tubing into the two support clips on the side of the large acrylic guard. Push the end of the hose over the free end of the inline filter which precedes the mass flow controller.
Note: Observe that the sensor unit contains an inline filter cartridge to protect the mass flow controller from being contaminated or blocked by particles contained in unfiltered air.

8.9 Check that the TEOM® Sensor Head is free to move in all directions--left, right, and forward and back. This is necessary to isolate the Head from any outside vibrations (i.e. it should be completely suspended within the enclosure cabinet). The only connection of the Sensor Head is in the heated air inlet where the inner tube is connected to the outer tube (see Figure 3).

9. Assembly of the Sensor Unit and the Data Processing Unit

9.1 Examine the front and side panels of the TEOM® Sensor Unit. Ensure that the power switch located on the front panel (door) is off. This switch should not be turned on until the TEOM® hardware is set up and Section 2 of the TEOM® Software Manual has been read (see Appendix).
Note: The black panel on the right side of the sensor unit contains all the external connections needed for power, signals, and vacuum pumps. Examine, also, the input/output connectors located on the side and back of the microcomputer.

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9.2 Attach the black coaxial cable between the BNC connector on the TEOM® Sensor Unit labelled "Freq Sig" and the BNC connector on the R&P Counter Board (see Figure 5).
Note: The BNC connector marked "Freq Sig" transmits the frequency output from the TEOM® Sensor Unit. The expansion card in the highest numbered slot (slot 4 in the Compac II personal computer) is the R&P Counter Board. This board contains a BNC connector for receiving the frequency signal from the TEOM® Sensor Unit. The nine-pin connector located on this board is not used in the TEOM® Series 1200 Ambient Particulate Monitor.

9.3 Attach the analog cable between the two 9-pin connectors on the TEOM® Sensor Unit and the 37-pin connector on the analog board in the microcomputer.
Note: The nine-pin connectors allow analog data to pass between the TEOM® Sensor Unit and the microcomputer. The expansion card in the next-to-highest numbered slot (slot 3 in the Compac II personal computer) is an analog input/output board with digital input/output capabilities.

9.4 Attach a 3/16" (inside diameter) hose from the barbed hose connector on the right side panel of the TEOM® Sensor Unit to the port of a suitable oil-free vacuum pump.
Note: The pump should be capable of maintaining approximately 20" Hg vacuum at a 4 Lpm flow rate. Pulsations from the vacuum line should be kept at a minimum. A small carbon vane pump of 1/10 hp or greater is suitable. Place the sample pump away from the TEOM® Sensor Unit to minimize the coupling of pump vibrations into the TEOM® Sensor Unit.

9.5 Attach the printer (optional) to the microcomputer with a parallel printer cable.
Note: The 15 pin "D" connector provides the user with analog input/output capabilities for user defined functions. Three channels of analog input and output are available for definition by the user. All analog signals are scaled from 0 to 5 VDC. For example, the user may choose up to three variables (such as mass concentration or total mass) to be output to a chart recorder or data acquisition system by entering the appropriate value in the Configuration Definition Routine (see Appendix or Section 6 of the TEOM® Software Manual). It is also possible to input three independent signals (for instance humidity and ambient temperature) into the TEOM® Sensor Unit. These inputs may be changed into engineering units, and plotted and/or saved on disk simultaneously with the TEOM® data.

9.6 Attach the power cords to the TEOM® Sensor Unit and microcomputer. Plug the power cords of the TEOM® Sensor Unit, microcomputer and optional printer into electric sockets with the appropriate voltage. Contact R&P, your distributor or representative if you have any questions about the voltage for which your instrument is configured. Do not apply power until instructed to do so in Section 11.1 or in Section 2 of the TEOM® Software Manual.

10. Exchanging the Filter Cartridge

Upon arrival of a new TEOM® series 1200 Ambient Particulate Monitor, the sensor/preheater unit will not be equipped with a filter cartridge. Therefore, it is necessary to follow the filter exchange procedures outlined below to prepare the instrument for operation. The new instrument comes with a box of 20 blank filter cartridges. Before proceeding with the exchange, some special precautions must be taken:

- Do not exchange filter cartridges when the TEOM® Series 1200 Ambient Particulate Monitor is taking data, i.e. when it is in the Collection Mode. Filter cartridges should be exchanged either when the instrument is in the Initialization Mode, or when both the TEOM® Sensor Unit and microcomputer are turned off.
- Do not handle new TEOM® filter cartridges with fingers. Use the filter tool provided with the instrument to exchange filters.
- Keep the sample pump running to facilitate filter exchange.

10.1 Loading the Filter Cartridge

10.1.1 Locate the TEOM® microbalance lever with the black ball in the down position (see Figure 3). Carefully rotate this lever upward. The TEOM® Sensor Head will swing forward into its filter changing position, exposing the filter cartridge.
Note: When the TEOM® Sensor Head is in this open position, the tapered element automatically stops vibrating to facilitate filter exchange.

10.1.2 Remove a clean filter cartridge from its shipping/storage box using the filter exchange tool. The tools upper metal disc should cover the filter's surface while the lower tines of the fork should straddle the hub of the filter base.

10.1.3 Hold the filter exchange tool in line with the tapered element and lightly insert the hub of the filter cartridge onto the tip of the tapered element. Ensure that the filter is seated properly. The tools metal disc should be centered over the filter before pressure is applied. Apply downward pressure to set it firmly in place. This will reduce the chances of distorting the crimped filter (see Figure 4).

10.1.4 Remove the filter exchange tool by retracting it sideways until it clears the filter. Do not disturb the filter.

10.1.5 Gently move the ball-ended lever to the down position to close the head. Allow the springs to pull it closed for the last centimeter so that the distinct sound of a metal-to-metal contact is heard.
Note: Do not let the TEOM® microbalance slam closed from the full open position.

10.1.6 Close and latch the door to the instrument enclosure cabinet. Keep the door open for as short a time as possible to minimize the temperature upset to the system.

10.1.7 Allow the unit to stabilize for one half-hour before taking data.

10.2 Removing the Filter Cartridge

Note: Filter lifetime depends upon the flow rate used, and the nature and concentration of the particulate sampled. The lower the flow, the longer the filter life. The filter lifetime is determined by the pressure drop across the filter, as shown by the vacuum gauge on the front panel of the TEOM® Sensor Unit. TEOM® filter cartridges must be exchanged when

the pressure drop reaches 15" Hg. This generally corresponds to a total mass accumulation of 5 to 10 mg. The automatic flow controller inside the TEOM® Sensor Unit cannot maintain the flow rate desired by the user when the pressure drop exceeds this level.

10.2.1 Using the filter exchange tool (see Figure 4), remove the filter cartridge from the sensor head. Carefully insert the lower fork of the tool under the filter cartridge so that the tines of the fork straddles the hub of the filter cartridge. The tool's upper metal disc should be centered over the filter's surface but not touching it. Gently lift the filter from the tip of the tapered element with a straight pull upwards.
Note: Never twist the filter cartridge to remove it or apply sideways force to the tapered element (see Figure 4).

10.2.2 Store the used filters or discard as necessary.

10.2.3 Remove a clean filter cartridge from its shipping/storage box using the exchange tool. Grasp the clean filter as instructed in Section 10.1.2. Do not touch the filter cartridge with your fingers - use only the exchange tool.

10.2.4 Follow the procedures detailed in Section 10.1.3 through Section 10.1.7 to insert the clean filter cartridge onto the sensor head and restore the instrument back to the operation mode.

11. Instrument Operation

Before the instrument start-up procedures are implemented, follow the instructions detailed below or those through Section 2.5 of the TEOM® Software Manual.

11.1 Preparation of Computer

11.1.1 Hard disk systems - make sure that diskette drive A does not contain a diskette. Remove any diskette that resides in diskette drive A.

11.1.2 Floppy disk systems - insert the TEOM® Program Diskette in diskette drive A. Insert the TEOM® Data Diskette or any formatted diskette with free storage capacity in diskette drive B.

11.1.3 When TP3 is not automatically executed, then it can be executed through MS-DOS.

11.1.3.1 For hard disk systems choose the proper disk drive: C: <Enter>; select the appropriate subdirectory: CD \TP3 <Enter>; start program execution: TP3/Instrument Name <Enter> - where InstrumentName is the model number of the TEOM® monitor, such as 1200. For example, type TP3 /1200 to start executing TP3 for the TEOM® Ambient Particulate Monitor.

11.1.3.2 For floppy disk systems choose the proper diskette drive: A: <Enter>; start program execution: TP3/Instrument Name <Enter> - where InstrumentName is the model number of the TEOM® monitor, such as 1200. For example, type TP3 /1200 to start executing TP3 for the TEOM® Ambient particulate Monitor.
Note: If an improper instrument name is entered, the instrument informs the user with a special screen. In this case, the program halts execution and waits for the user to press any

key before re-entering MS-DOS. If this condition is encountered, refer to Section 11.1.3.1 and/or 11.1.3.2 for instructions to re-start the program.

11.1.4 Once TP3 has begun execution, it displays a message for several seconds indicating that it is loading additional files. The system screen is then displayed. This screen gives information on the vendor. The next screen displays a copyright notice to the user.

11.1.5 After this input, the computer shows the main display screen [see Figure 6(a)]. The precise layout of this screen can vary from one type of TEOM® instrumentation to another. The main display screen is displayed by the computer during nearly every phase of instrument operation. All real-time data plotted and displayed by the instrument appear on this screen. Figure 6(b) illustrates the components of the main display screen.
Note: Do not turn on power to the TEOM® Sensor Unit unless the preceding steps have been taken and the TP3 software is running on the computer. Operating the instrument while not under computer control may lead to overheating and damage.

11.2 Instrument Start-up

11.2.1 Turn on the TEOM® Sensor Unit at the power switch located in the lower right-hand corner of the unit's front face.

11.2.2 Turn on the sample pump. Allow 2 hours (24 hours for highest accuracy) for the TEOM® monitor to warm up to its user-defined temperature set points and achieve its flow rate before beginning data collection. Pre-filtered (Ballston Filter 9933-05-CQ) air should be drawn through the instrumentation during the initial warm-up period. These filters are the same diameter as the inlet of the outer metal tube and are very similar to the inlet filter which precedes the mass flow controller. Each pre-filter fits directly onto the silicone "sampling" tubing which covers the outer metal inlet. Other filters which are similarly made can be used as long as they are demonstrated equivalent.
Note: The baseline performance of the TEOM® monitor in terms of mass concentration is shown in Figure 7. These data were taken after the device had operated continuously for a long period of time, with pre-filtered air drawn through the system and under stable ambient and sample stream temperatures. The data file shown in this figure is 1200BASE.PRN, which is provided as part of the instrument's software.

11.2.3 Turn on the optional printer.

11.2.4 When a baseline is achieved similar to that of Figure 7, remove the pre-filter from the heated air inlet-silicone tubing assembly while the vacuum is still being applied. This initiates sampling.

11.3 Instrument Shut-Down and Shipping

11.3.1 Turn off the TEOM® Sensor Unit at the power switch located in the lower right-hand corner of the unit's front face.

11.3.2 Turn off the sample pump and the optional printer.

11.3.3 When sampling at another location nearby, the sensor/ preheater assembly must be secured before moving the Sensor Unit. Close the sensor head restraining clamp located at the lower right side of the microbalance unit and inside right side of enclosure cabinet.

This secures the sensor head to the side of the enclosure cabinet to prevent damage during transport.
Note: Do not transport the assembled sensor unit large distances or by commercial carrier in the assembled condition.

11.3.4 Transport the assembled sensor unit by hand or cart to the new sampling location. Open the restraining clamp when the instrument is set up at its new location.

11.3.5 When transporting by commercial carrier, the sensor/preheater assembly must be removed from the instrument enclosure cabinet. The reverse of the assembly instructions should be followed to disassemble the sensor unit components (see Section 8). Each component should be packed separately in the original containers using suitable packing materials such as foam or bubble wrap.

12. Instrument Variable Settings

12.1 Setting Sampling Parameters

The software provided with the TEOM® Series 1200 Ambient Particulate Monitor contains three pre-defined configurations:

- plots mass concentration on the computer monitor during data collection
- plots mass concentration and 24-hour averaged mass concentration on the computer monitor during data collection
- plots mass concentration and total mass on the computer monitor during data collection

All of these configurations store the date, time, mass concentration and total mass on disk when data files are created by the program.
Note: Configurations U to Z are reserved for the TEOM® demonstration software. Do not create configurations with these names. These configurations may be changed and new configurations may be added by the user in the Configuration Definition Routine (Section 6 of the TEOM® Software Manual). Slots 13 to 18 of the Configuration Definition Routine allow the user to change the values for operating temperatures and flow rate (See Appendix or Section 6 of the TEOM® Software Manual). Since these settings are unique for each type of TEOM® instrumentation, they are defined below specifically for the TEOM® Series 1200 Particulate Mass Monitor:

Configuration Line	Description	Permissible Range
13	Sample Flow Rate	-5.0 to 0, 0.5 to 5.0 L/min
14	TEOM® Housing Temp	0, 25 to 60°C
15	Air Tube Temperature	0, 25 to 60°C
16	TEOM® Cap Temperature	0, 25 to 60°C
17	Enclosure Temperature	0, 25 to 50°C
18	Not Defined	

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The values of these settings are recorded in data files sorted on disk, and are also included in the numeric printouts of data files enabled by the F9 key.

12.1.1 Sample flow rate (slot 13) - the sample flow rate is the rate (Lpm) at which the particulate-laden sample is drawn through the TEOM® monitor. A negative value causes the flow controller to open its valve fully, allowing for external control of the flow rate. In this case the instrument computes mass concentration based upon the absolute value of the negative number entered. A value of 0 closes the valve of the flow controller, stopping the sample flow through the system. A positive value between 0.5 and 5.0 L/min automatically sets the flow controller to the entered flow rate.

12.1.2 Housing temperature (slot 14) - the value of this slot determines the temperature at which the TEOM® housing in the Sensor Unit is to be maintained. A value of 0 specifies that the temperature of the TEOM® housing is not to be controlled. A value between 25 and 60°C automatically causes the instrument to control the TEOM® housing temperature at the indicated temperature.

12.1.3 Air tube temperature (slot 15) - the value of this slot determines the temperature at which the sample air flow is maintained, as measured by a probe in the air stream. A value of 0 specifies that the temperature of the air tube is not to be controlled. A value between 25 and 60°C automatically causes the instrument to control the temperature of the air at the indicated temperature.

12.1.4 Cap temperature (slot 16) - the value of this slot determines the temperature at which the cap of the TEOM® microbalance is maintained. A value of 0 specifies that the temperature of the cap is not to be controlled. A value between 25 and 60° C automatically causes the instrument to control the temperature of the cap at the indicated temperature. This value is normally set to be the same as the TEOM® housing temperature (Slot 14)

12.1.5 Enclosure temperature (slot 17) - the value of this slot determines the temperature at which the interior of the enclosure is maintained. It should normally be set to 45° C. A value of 0 specifies that the temperature of the enclosure is not to be controlled. A value between 25 and 50° C automatically causes the instrument to control the temperature of the enclosure at the indicated temperature.

12.2 Instrument Frequency Clipping

Because the TEOM® Series 1200 Particulate Mass Monitor is ordinarily used to measure relatively long term changes in particulate concentrations, the instrument's clipping capability is normally turned on.

12.2.1 The instrument's clipping routine is used to lessen the effects of outlying frequency values (isolated "bad" data points) on mass calculations that can be caused by mechanical or electrical disturbances. When the clipping capability is turned on, a "window" is formed around the average frequency value (adjusted for slope).

12.2.2 If the next raw frequency value lies within the window, the frequency value is not affected and the span of the window is decreased by the decimal percentage prescribed by "inclip" (see below).

12.2.3 If the next raw frequency value lies outside the window, the frequency value is given the maximum or minimum value of the window, depending upon whether the raw frequency point was high or low. In addition, the span of the window is increased by the decimal percentage prescribed by "outclip" (see below). "Inclip" and "outclip" are assigned the following values in the TEOM® Series 1200 Particulate Mass Monitor:

Inclip 0.02
Outclip 0.02

13. Confirmation of Instrument Calibration

Note: The procedure below enables the user to confirm the calibration of the TEOM® microbalance set by the manufacturer. There is no need for frequent calibration checks, as the mass detection characteristics of the TEOM® system's tapered element do not change over time. Following is a description of the method used at the Automotive Emissions Laboratory of the New York State Department of Environmental Conservation for checking the calibration of the TEOM® monitor. The procedure allows the user to check the accuracy of the instrument's calibration constant, K_o, calculated by the manufacturer. It involves a comparison of the mass indicated on a gravimetric balance with that indicated by the TEOM® monitor for a given calibration mass. The calibration mass is a circular disk of Pallflex filter material 3 mm (1/8") in diameter. The instrument used to punch out circular Pallflex disks is a vacuum tweezer assembly which is also used to transport the Pallflex discs.

13.1 Punch circular discs out of Pallflex filter paper (type T60A20) using the disc punching instrument. A calibration dot 3 mm in diameter weighs approximately 100 mg.

13.2 Determine the mass of the calibration dot on a gravimetric laboratory balance that has microgram sensitivity.

13.3 Establish a baseline for total mass on the microcomputer screen with the sample flow rate set, for example, at 3 Lpm.

13.4 Drop the calibration dot onto the center of the TEOM® filter cartridge. This is done by decreasing the suction of the vacuum tweezer. Close the TEOM® microbalance to restart the vibration of the TEOM® monitor.
Note: Do not touch the TEOM® filter cartridge with the vacuum tweezer. As is the case with TEOM® filter cartridges, the calibration dot must never be touched by hand.

13.5 The TEOM® monitor will indicate the change in total mass that results from the calibration dot being placed on the filter cartridge.

13.6 Remove the calibration dot from the TEOM® filter cartridge using the vacuum tweezer. Do not touch the TEOM® filter cartridge with the vacuum tweezer. Make sure that the total mass reading returns to its original base line (to within a fraction of a microgram).

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13.7 Compare the masses determined gravimetrically and by the TEOM® system, and calculate a revised calibration constant, K_o, if necessary:

K_o (revised) = K_o (original) x Mass (gravimetric)/Mass (TEOM® Monitor)

13.8 If desired, revise the calibration constant, K_o, stored in the TEOM® monitor.

14. Main Display Screen

Note: This section describes the commands that manipulate the information shown on the main Display Screen. An understanding of this Section is important for the effective use of the TEOM® monitor. Figure 8 identifies the components of the main display screen.

14.1 Top Line of the Main Display Screen

14.1.1 Current configuration - each configuration has a single-letter name ranging from A to Z. When the computer is turned on, configuration A is automatically loaded into memory (see Figure 8). If a listing of the current configurations is desired or if a different configuration is to be loaded, consult Appendix for the correct procedures.

14.1.2 Operating mode - the operating mode indicates the current operating status of the TEOM® monitor. The instrument runs in the following modes.

14.1.2.1 The instrument is in the Initialization (INIT) Mode when it is first turned on, and after the main display screen has been cleared and the Initialization Mode chosen by pressing F3.

14.1.2.2 The instrument collects, plots and displays mass rate, mass concentration and total mass data when in the Collection Mode. Press F1 when in the Initialization Mode to enter the Collection Mode.

14.1.2.3 The instrument enters the Stop Mode after data collection has been stopped with the F2 key. The image on the main Display Screen may be printed while in the Stop Mode by pressing F9.

14.1.2.4 In the Replot Mode the user may replay data files stored on disk. Enter this mode by pressing F7 either in the Stop Mode (to replot the newest data file) or in the Initialization Mode (to replot any data file stored on disk).

14.1.2.5 The F9 key is used in the Stop and Replot Modes to print the image on the main display screen. When the F9 key is pressed while in the INIT Mode, the user may choose to print the numeric contents of any data file stored on disk.

Note: Because of the time required to print a screen image or the contents of a data file, the heating circuits in the TEOM® Sensor Unit are turned off during printing. The user may have to allow for temperatures to stabilize again before resuming data collection.

14.1.3 Data file name - all data file names have a .PRN extension even though this is not shown on the main display screen. This built-in program feature ensures file compatibility with all versions of Lotus 1-2-3® spreadsheet software. A listing of data files currently stored on disk may be obtained by entering ALT+D (hold down the ALT key and press D) when in the INIT Mode.

14.1.4 Current time - this part of the screen displays the current time of day. If this clock time is incorrect, exit from TP3 into MS-DOS by pressing F10. Then type TIME followed by <Enter>. The computer then displays the current time and gives the user a chance to enter a new time. Re-enter TP3 from MS-DOS by entering the commands shown in Section 11.1.

14.1.5 Current date - this part of the screen displays the current date. If this date setting is incorrect, exit from TP3 into MS-DOS by pressing F10. Then type DATE followed by <Enter>. The computer then displays the current date and gives the user a chance to enter a new date. Re-enter TP3 from MS-DOS by entering the commands shown in Section 11.1.

14.2 Bottom Line of the Main Display Screen

14.2.1 Y1-axis label - the Y1-axis Label displays the name of the variable whose scale is shown on the left-hand Y-axis. The abbreviations used to designate variables are listed in Table 1.

14.2.2 Error code - this field indicates whether a hardware malfunction has been detected by the instrument. An error code 0 represents no malfunction. The instrument detects the following types of error conditions:

Error Code	Description
0	No error condition
1	Error condition on R&P Counter Board
2	Error condition on analog input board
4	Error condition on analog output board
8	Error condition on digital input board
16	Error condition on digital output board
32	Unsupported programming feature used
64	Tapered element not oscillating or improper cable attachment

In the case of multiple simultaneous errors, the error code consists of the sum of the current error conditions. For example, the error code 65 indicates that an error condition has been detected on the R&P Counter board (code 1) and that the computer is not receiving a frequency signal from the TEOM® microbalance (code 64). Pressing F3 resets the error code to 0.

14.2.3 Status code - the status code conveys information about the calculation of data and the amount of disk space available for saving data. This field is blank under most operating conditions. A status code display most commonly occurs just after F1 has been pressed in the INIT Mode to begin data collection (codes M and R). In this case, the status display gives the user feedback that data collection has begun and indicates when the computer has calculated the first valid data point. Because total mass, mass rate and mass concentration calculations are based upon averaged raw data, a certain time elapses between the start of data collection and the calculation of the first valid data point. Total mass data are plotted and displayed as 0 until a sufficient number of raw frequency data

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points have been collected for calculation. Likewise, mass rate and mass concentration data are plotted and displayed as 0 until the appropriate number of total mass data points have been processed.

Status Code	Description
M	Total mass, mass rate and mass concentration are plotted and displayed as 0--data not yet valid.
R	Total mass values are valid. Mass rate and mass concentration are not yet valid, and are plotted and displayed as 0.
D	The data disk has reached its maximum capacity. The current data file has been closed in an orderly fashion but data are no longer being stored on disk.
blank	Normal condition. If the instrument is in the Collection Mode, total mass, mass rate and mass concentration data are valid.

14.2.4 Y-axis selection - the arrow in this field indicates which Y-axis is the current Y-axis, i.e., which axis is influenced by commands that change the display of Y-axes. If the arrow points to the left, the Y1-axis (left) is the current Y-axis and is affected by Y-axis commands. Conversely, if the arrow points to the right, the Y2-axis (right) is the current Y-axis and responds to Y-axis commands. Press F5 to change the current Y-axis. This command toggles between the Y1-axis and Y2-axis. The following Y-axis commands act only upon the current Y-axis.

Command	Results
Shift+Fn	Display the selected Y-axis scale
Up Arrow, Down Arrow	Shift Y-axis up/down by one division
PG UP, PG DN	Shift Y-axis up/down by one page
2, 5, 0	Expand Y-axis scale by factors 2, 5, 10
ALT+2, ALT+5, ALT+0	Contract Y-axis scale by factors of 2, 5, 10
Home	Reposition Y-axis scale to center next Y-point

14.2.5 User input field - the user input field displays prompts and accepts inputs from the user. A number of function key commands, such as F1, F3, F4, F6, and F10 require input from the user. When a prompt appears in the User Input Field, the instrument awaits the user's input before continuing its operation. All user inputs must be followed by <Enter> in order to be accepted by the computer. Prompts which include the message "(Y or N)" require that a Y or N be entered by the user followed by <Enter>. The F6 command allows the user to change the variables shown in the Main Numeric Display and Short Numeric Display at any time. After F6 is pressed the computer displays the message

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"Command:". In response, enter the location at which the variable is to be displayed (explained below), followed by <Enter>. The computer then prompts the user with the message "Entry:". Then type the Program Register Code for the desired variable (see Table 1), followed by <Enter>. The location of the desired variable is determined by the following codes:

Code	Description
0	Short Numeric Display
1-42	Main Numeric Display. The Main Numeric Display may contain up to 14 lines of information, with three variables displayed per line. The locations are numbered from bottom to top in the following manner:

Code	Description		
Top Line	•	•	•
	•	•	•
	10	11	12
	7	8	9
	4	5	6
Bottom Line	1	2	3

Note: Certain models of TEOM® instrumentation do not have a Main Numeric Display. For example, the following key sequence causes raw frequency data to be displayed in the Short Numeric Display:

F6 0 <Enter> 86 <Enter>

14.2.6 Short numeric displays - this field displays the current value of a variable selected by the user. Variables may be displayed at this location in two ways:

- follow the procedure described above in Section 14.2.5, or
- If the variable to be shown in the short numeric display is represented by a function key, enter CTRL + Fn (hold down CTRL and press the desired function key). For example, enter CTRL + F5 to show real-time mass rate values in the short numeric display.

14.2.7 Y2-axis label - the Y2-axis Label displays the name of the variable whose scale is shown on the right-hand Y-axis. The abbreviations used to designate variables are listed in Table 1.

14.3 X-Axis and Y-Axis Scales

Figure 8 identifies the location of the X-axis and Y-axis scales of the main display screen.

14.3.1 X-axis scale - the X-axis scale always displays time. By making the appropriate selection in the configuration definition routine (see Appendix), time can be displayed as either the elapsed time of data collection or time-of-day. The format may be either hh:mm:ss (hours:minutes:seconds) or dd:hh:mm (days:hours:minutes). The span of the

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X-axis scale may be changed in the Initialization, Collection and Replot Modes in the following manner:

Command	Result
Left Arrow, Right Arrow	Decrease/increase span by a factor of 2.
CTRL + Left Arrow, CTRL + Right Arrow	Decrease/increase span by increments determined by the program.

These commands may be entered in any order and as often as desired. When they are used in the Collection or Replot Modes, the graphical display area is cleared.

14.3.2 Y-axis scales - the main display screen can display as many as two Y-axis scales at the same time. The Y1-axis is located to the left and the Y2-axis to the right of the bottom line of the main display screen (Section 14.2.4). A number of commands may be used to change the current Y-axis scale (see 14.2.4). These commands all function in the Initialization, Collection and Replot Modes.

14.3.2.1 For examples, follow these steps to display the scale for mass concentration on the Y2-axis: 1) press F5, if necessary, to point the Y-axis selector toward the Y2-axis, and 2) enter SHIFT + F6 to display the mass concentration scale. This command is a toggle switch. Executing it again turns off the current Y-axis scale.

14.3.2.2 The Up Arrow, Down Arrow, PG UP and PG DN commands allow the user to reposition variables vertically by shifting the scale of the current Y-axis either up or down. These keystrokes may be pressed in any order and repeated as often as desired.

14.3.2.3 The 2, 5, 0, ALT +2, ALT +5 and ALT + 0 commands change the scaling of the current Y-axis by factors of 2, 5, and 10. They may be executed in any order, and as often as desired.

14.3.2.4 The Home command is useful when a plotted variable such as total mass is about to go off the screen. Pressing Home in this case repositions the current Y-axis scale so that the next data point is plotted in the middle of the screen.

14.4 Variables Selected for Plotting

The variables currently selected for plotting in the Collection and Replot Modes are shown directly above the graphical display area (see Figure 8). These settings may be turned on and off any time the main display screen appears on the monitor. Variables may be added to or deleted from the list of plotted variables by entering an appropriate ALT +Fn command. For example, press ALT + F6 to add or subtract mass concentration from the list of plotted variables.

14.5 Main Numeric Display

This field displays the current values of selected variables. Its format varies from one model of TEOM® instrumentation to another. The main numeric display may be scrolled

up and down using the CTRL + UP and CTRL + DN commands. The variables shown here may be changed by the user according to instructions in Section 14.2.5.

14.6 Automatic Execution Setting

The operation of the TEOM® monitor may be directed from a remote location using the digital input capability of the computer. When the automatic execution setting is on, the TEOM® monitor executes the steps of the instrument cycle according to the values of digital inputs 0 and 1. The instrument's automatic collection capability may be turned on and off only in the Initialization Mode. Enter ALT + A to toggle this remote operation ability on and off (see Figure 9). The value of digital inputs 0 and 1 cause the instrument to execute the following steps of the instrument cycle when the automatic execution capability is turned on:

Digital Input 0	Digital Input 1	Description
0	0	The instrument awaits a digital input
1	0	Corresponds to F1: Begin data collection, enter Collection Mode
0	1	Corresponds to F2: Stop data collection, enter Stop Mode
1	1	Corresponds to F3 (when in Stop Mode): Clear screen, enter INIT Mode or Corresponds to F2 and F3 (when in Collection Mode): Stop data collection and clear screen, enter INIT Mode.

Generally, a digital input of 0 corresponds to ground, while an input of 1 refers to 5 VDC. Allow up to 5 seconds for the instrument to respond to the above digital input commands. These settings and the locations of the inputs can vary from one type of TEOM® monitor to another. Refer to the TEOM® Hardware Manual, or consult with R&P or your distributor, to determine the location and proper handling of these digital inputs.

15. Method Safety

This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the user's responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

16. Performance Criteria and Quality Assurance (QA)

Required quality assurance measures and guidance concerning performance criteria that should be activated within each laboratory are summarized and provided in the following section.

16.1 Standard Operating Procedures (SOPs)

16.1.1 SOPs should be generated by the users to describe and document the following activities in their laboratory:

- assembly, calibration, leak check, and operation of the specific sampling system and equipment used;
- preparation, storage, shipment, and handling of the sampler system;
- purchase, certification, and transport of standard reference materials; and
- all aspects of data recording and processing, including lists of computer hardware and software used.

16.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the personnel conducting the monitoring work.

16.2 Quality Assurance Program

The user should develop, implement, and maintain a quality assurance program to ensure that the sampling system is operating properly and collecting accurate data. Establish calibration, operation, and maintenance procedures should be conducted on a regularly scheduled basis and should be part of the quality assurance program. Calibration verification procedures provided in Section 13, operation procedures in Section 11,, and the manufacturer's instruction manual should be followed and included in the QA program. Additional QA measures (e.g., trouble shooting) as well as further guidance in maintaining the sampling system are provided by the manufacturer. For detailed guidance in setting up a quality assurance program, the user is referred to the code of Federal Regulations (18) and the EPA Handbook on Quality Assurance (19).

17. References

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17. Nagda, N. L., Rector, H. E., and Koontz, M. D., *Guidelines for Monitoring Indoor Air Quality*, Hemisphere Publishing Corporation, New York, NY, 1987.

18. 40 CFR, Part 58, Appendix A, B.

19. *Quality Assurance Handbook for Air Pollution Measurement Systems*, Volume II - Ambient Air Specific Methods, EPA 600/4-77-0272, May 1977.

Table 1. Program Register Codes

Code	Title	Description	Comments
Mass Rate			
80	MR00	Mass Rate (g/sec)	Use for disk storage
83	MR	Mass Rate (g/sec or selectable)	Use for plotting and printing
Mass Concentration			
81	MC00	Mass Conc (mg/m^3)	Use for disk storage
84	MC	Mass Conc (mg/m^3 or selectable)	Use for plotting and printing
Total Mass			
82	TM00	Total Mass (g)	Use for disk storage
85	TM	Total Mass (g or selectable)	Use for plotting and printing
TE Frequency			
86	FR00	Raw Frequency (Hz)	Use for disk storage
87	FR01	Clipped Frequency (Hz)	
88	FR	Average Frequency (Hz)	
89	SD	Std Dev of Frequency (10 sec)	Indicates stability of instrument
Clipping			
97	CLIP	Clipping Indicator	0 = Inactive; 1= Active
98	CLWI	Size of Clipping Window (Hz)	
Time and Date			
90	XTIM	Current Experimental Time (sec)	Automatically saved on disk
91	REPS	Calculation Repetitions	Number of program loops
92	CTIM	Clock Time	Format: 0.HHMMSS (hours, min, sec)
93	CDAT	Clock Date	Format: 0.MMDDYY (month, day, year)
Diagnostics			
95	ERR#	Current Error Code	
Automatic Instrument Operation			
148	DI00	Digital Input 0	With instrument in Automatic Setting, these inputs control operation
149	DI01	Digital Input 1	

TP3 refers to variables (such as mass concentration) by numbers called Program Register Codes. These Program Register Codes are common to all TEOM® instrumentation. Certain TEOM® monitors make use of additional codes. Consult Appendix A of the TEOM® Hardware Manual for a complete listing of codes applicable to your particular TEOM® instrument model.

Table 2. Description of Stored Data Files

Line(s)	Description
1	The time and date at which the data collection cycle was begun, expressed in the following format: 1 + mmddyyhhmm (1 + month, day, year, hour, minute).
2	The unique calibration constant for the TEOM® monitor. It is used during replotting to calculate total mass, mass rate and mass concentration from raw frequency data stored on disk. This calibration does not change during the lifetime of the instrument.
3	The rate at which the computer gathers raw frequency data from the TEOM® Sensor Unit. Typical instrument settings are one data point every 1.68 and every 0.21 seconds.
4	The rate at which data are saved to disk in seconds.
5	The length of time over which raw frequency data are averaged to compute total mass values.
6	The length of time over which total mass values are averaged to compute mass rate and mass concentration.
7-12	Instrument settings such as the sample flow rate and temperatures. The definition of these settings can vary from one type of TEOM® instrument to another.
13-20	The Program Register Codes (see Table 1) and names of the variables stored in columns 1-8 of the data file.

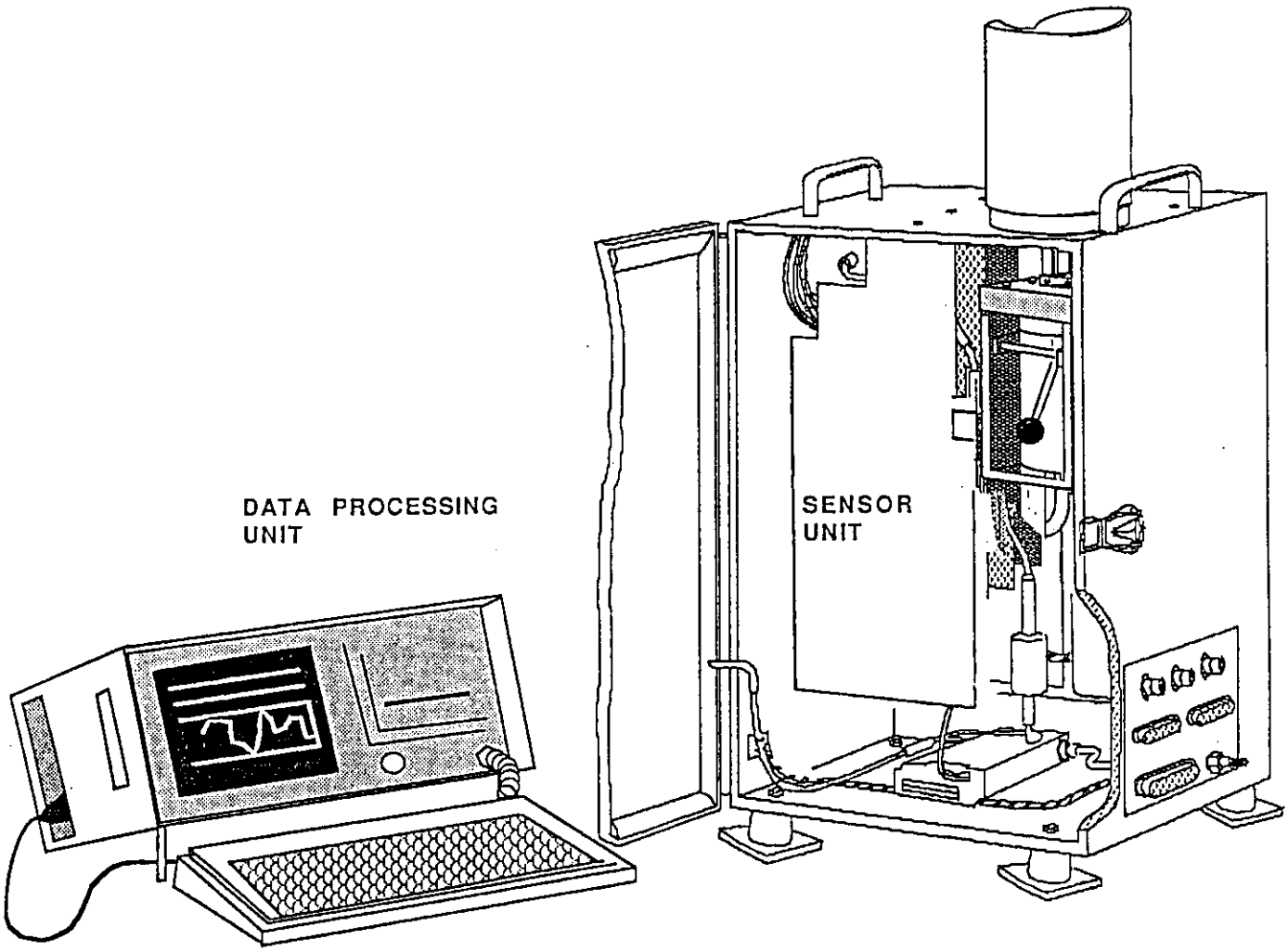

Figure 1. Assembled TOEM® Continuous Particulate Monitor

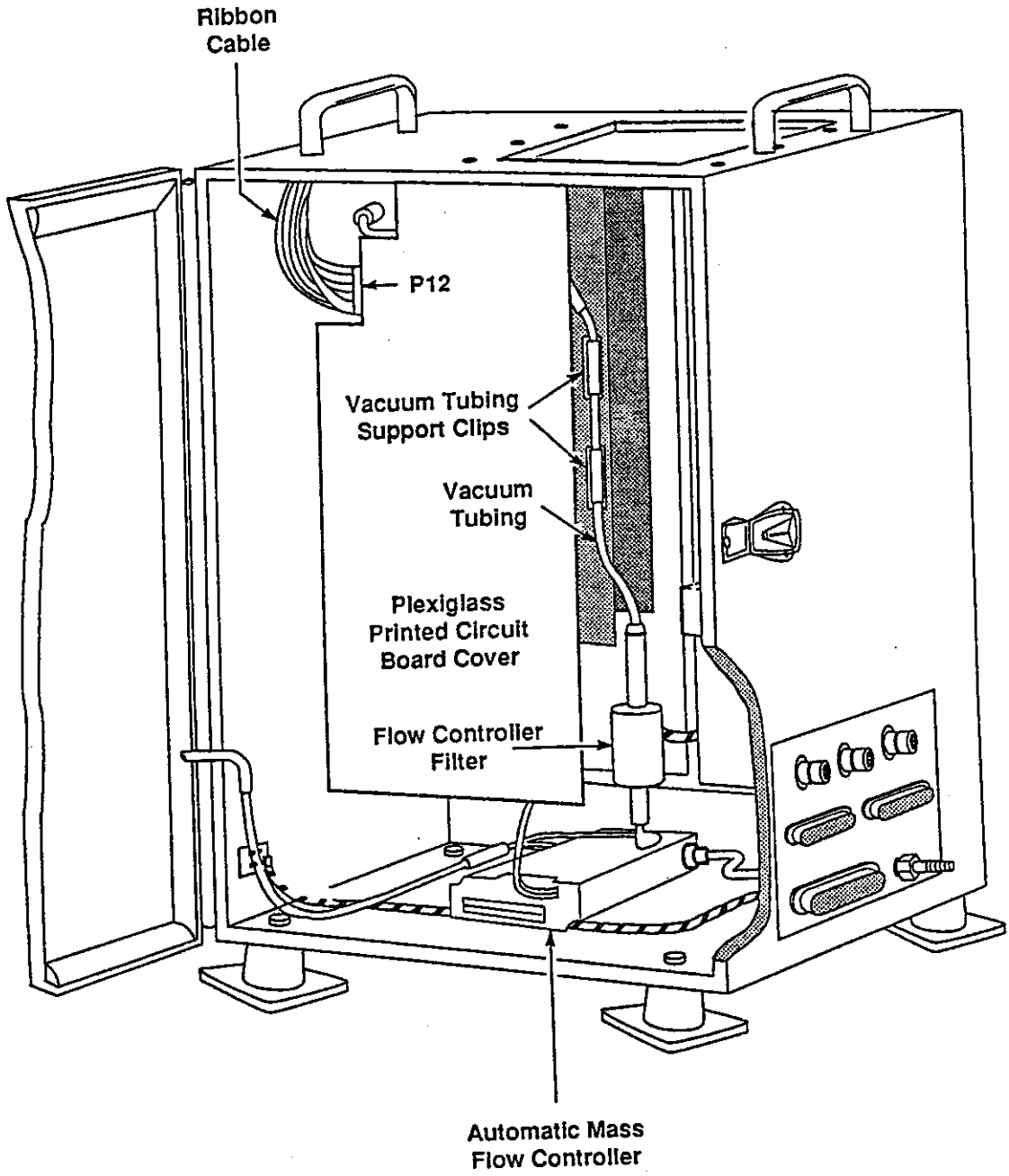

Figure 2. Enclosure Cabinet

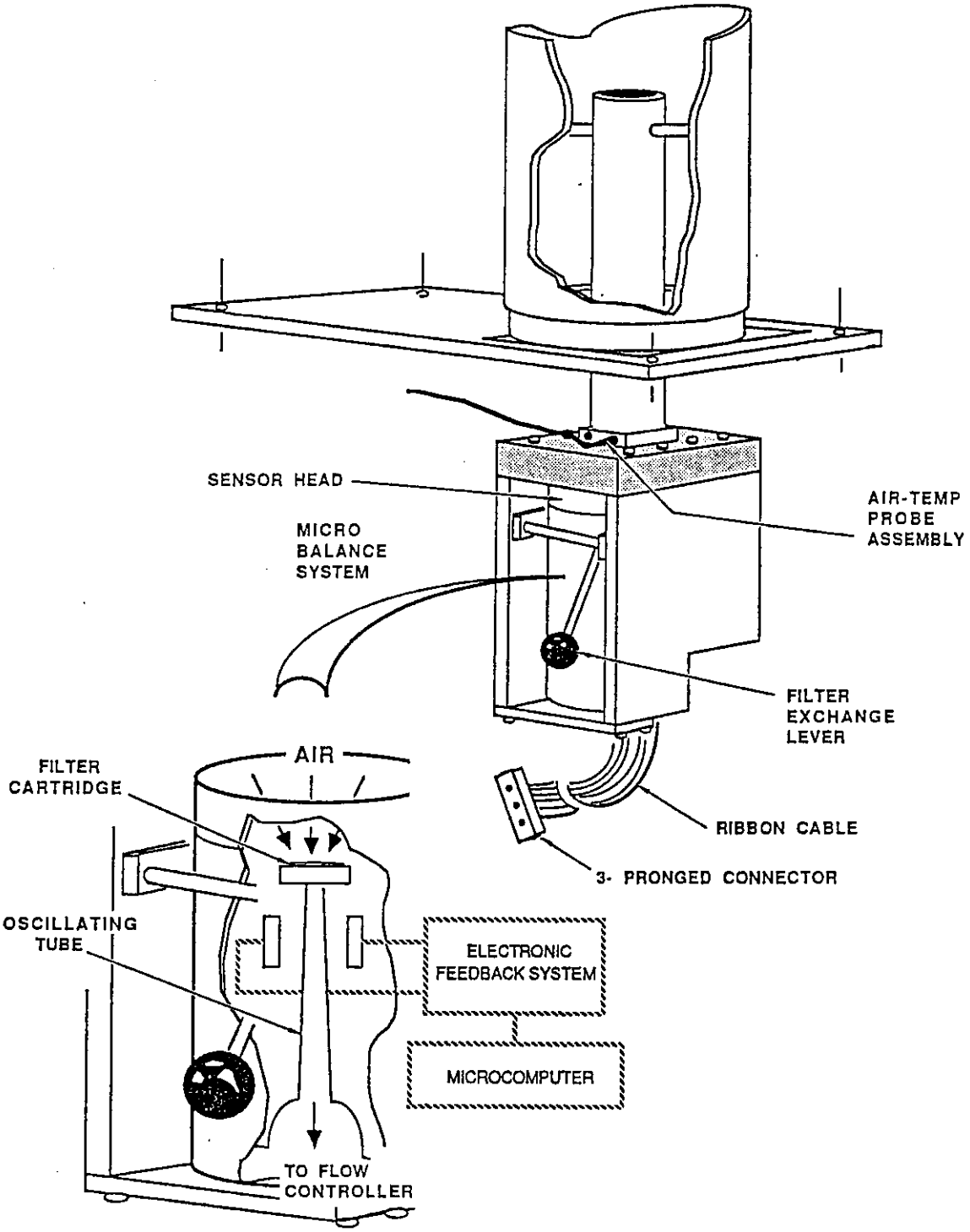

Figure 3. Sensor/Preheater Assembly

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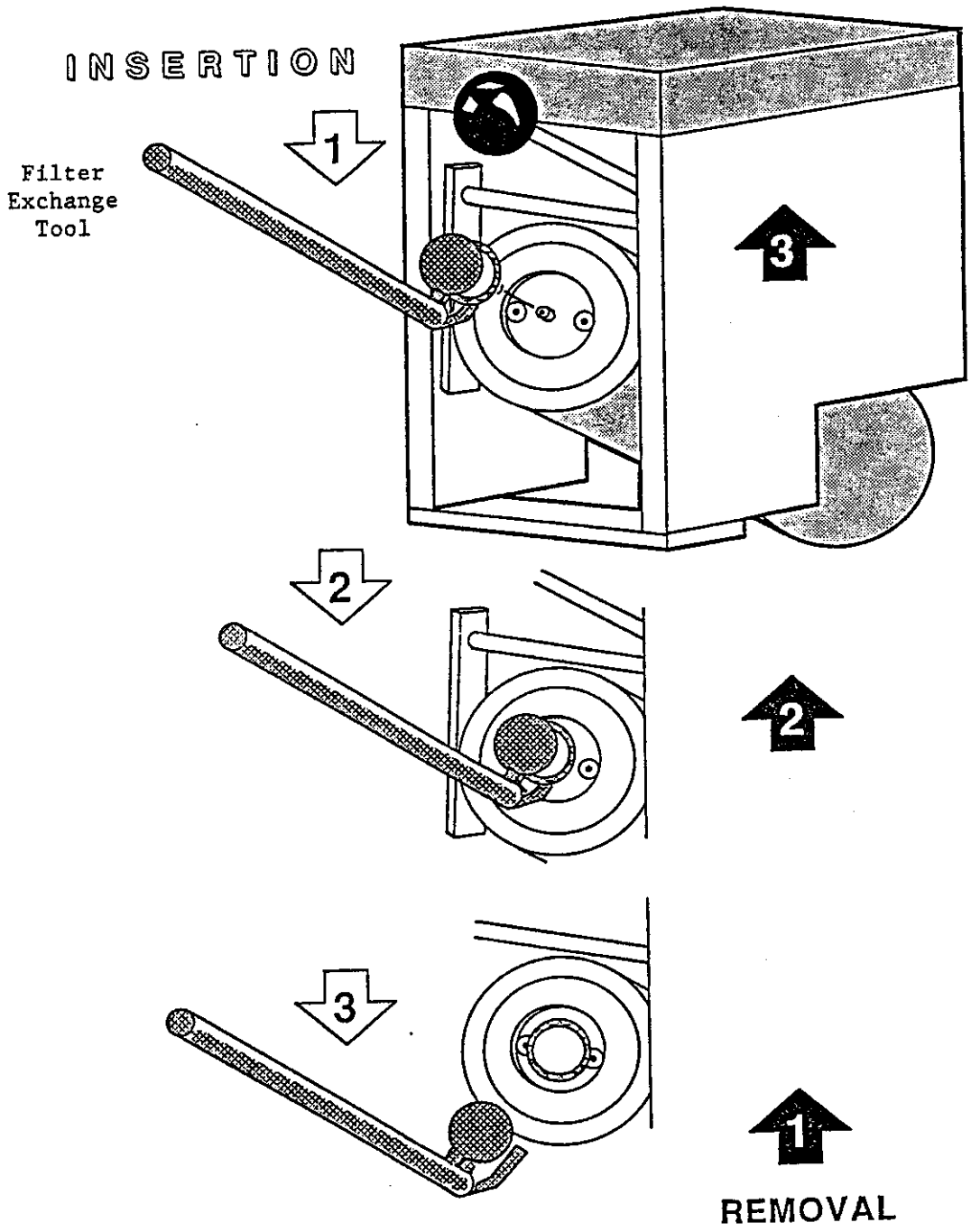

Figure 4. Loading/Removing Filter Cartridge Assembly

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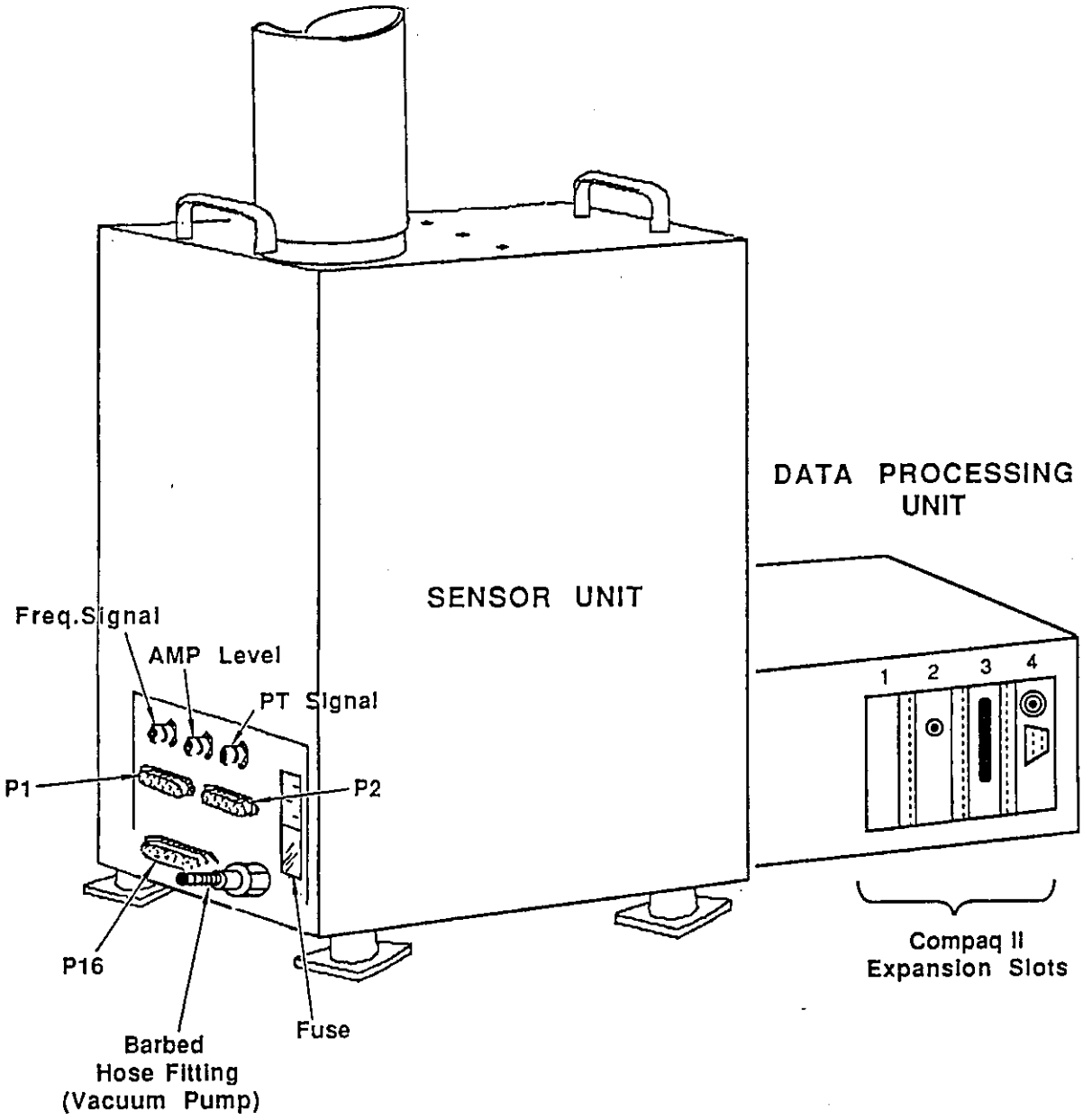

Figure 5. Electrical Connections Associated with the TEOM® Sensor Unit and Data Processing Unit

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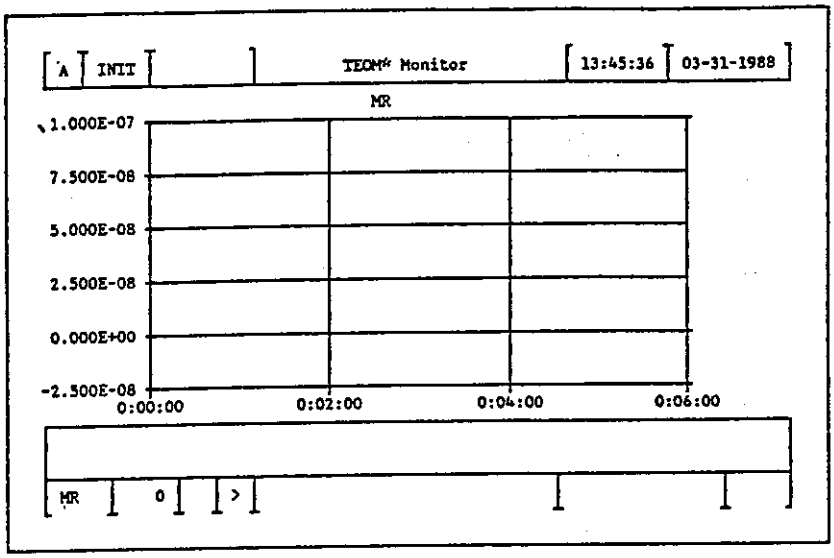
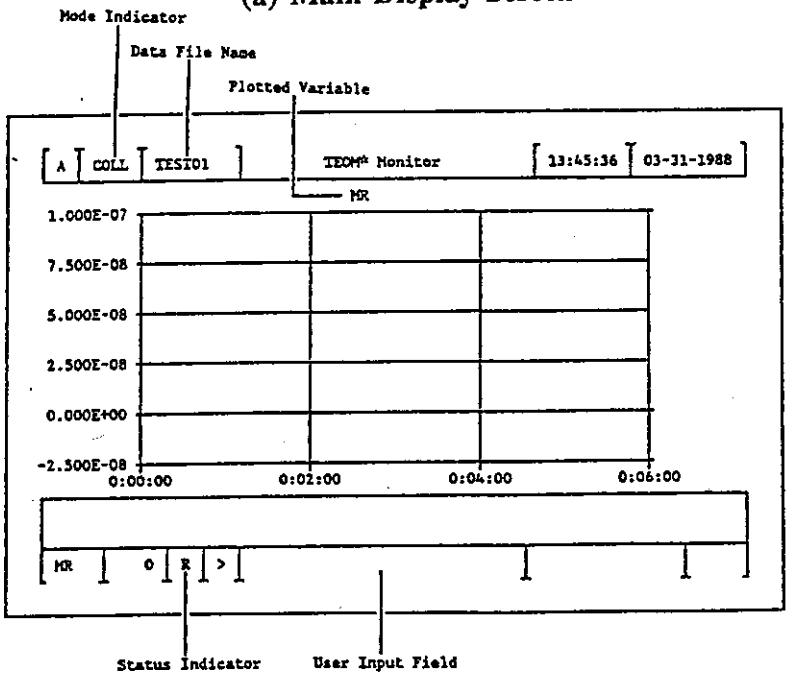


(a) Main Display Screen



(b) Components of the Main Display Screen

Figure 6. TEOM® Display Screen

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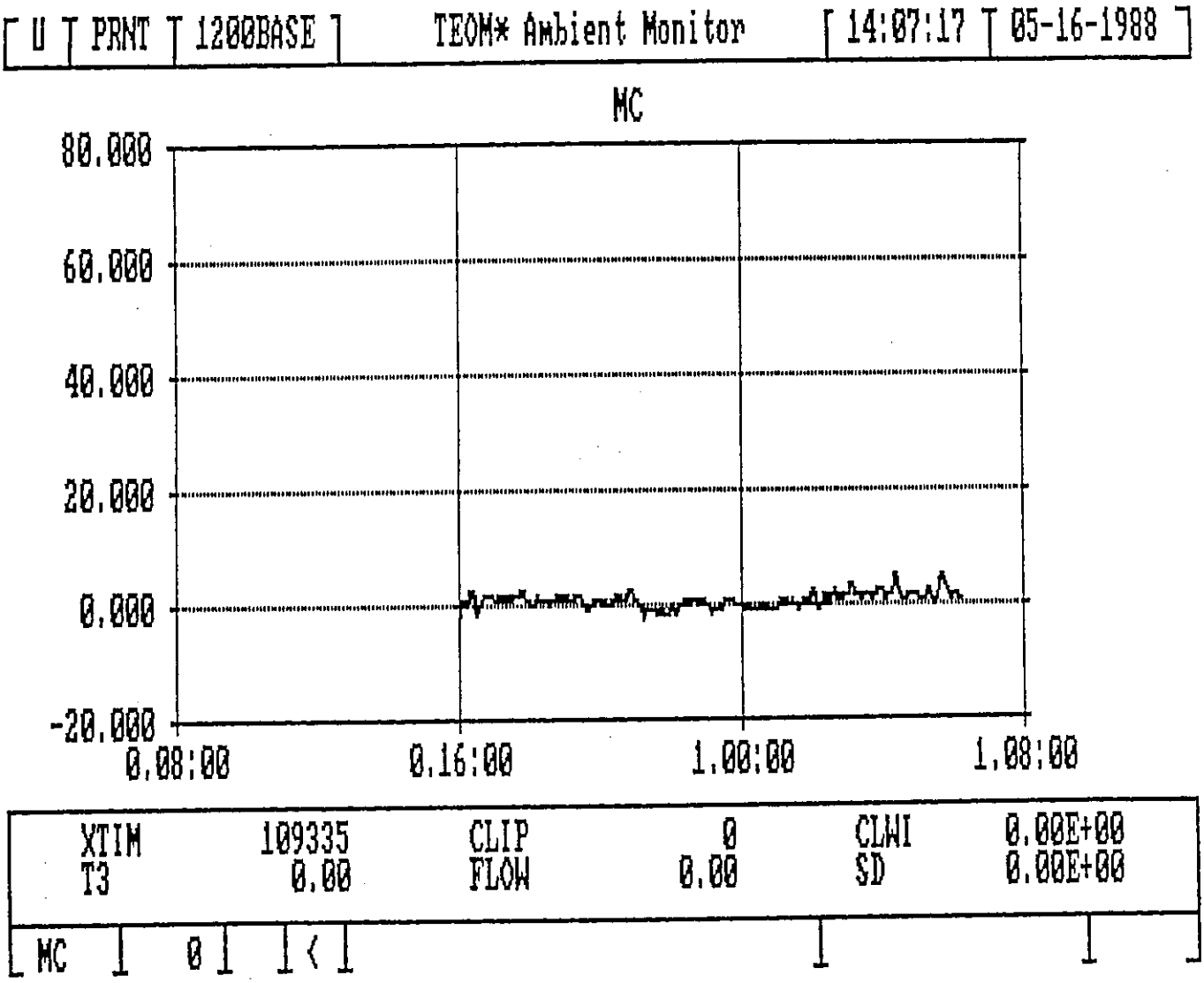

Figure 7. Baseline Performance of the TEOM® Monitor

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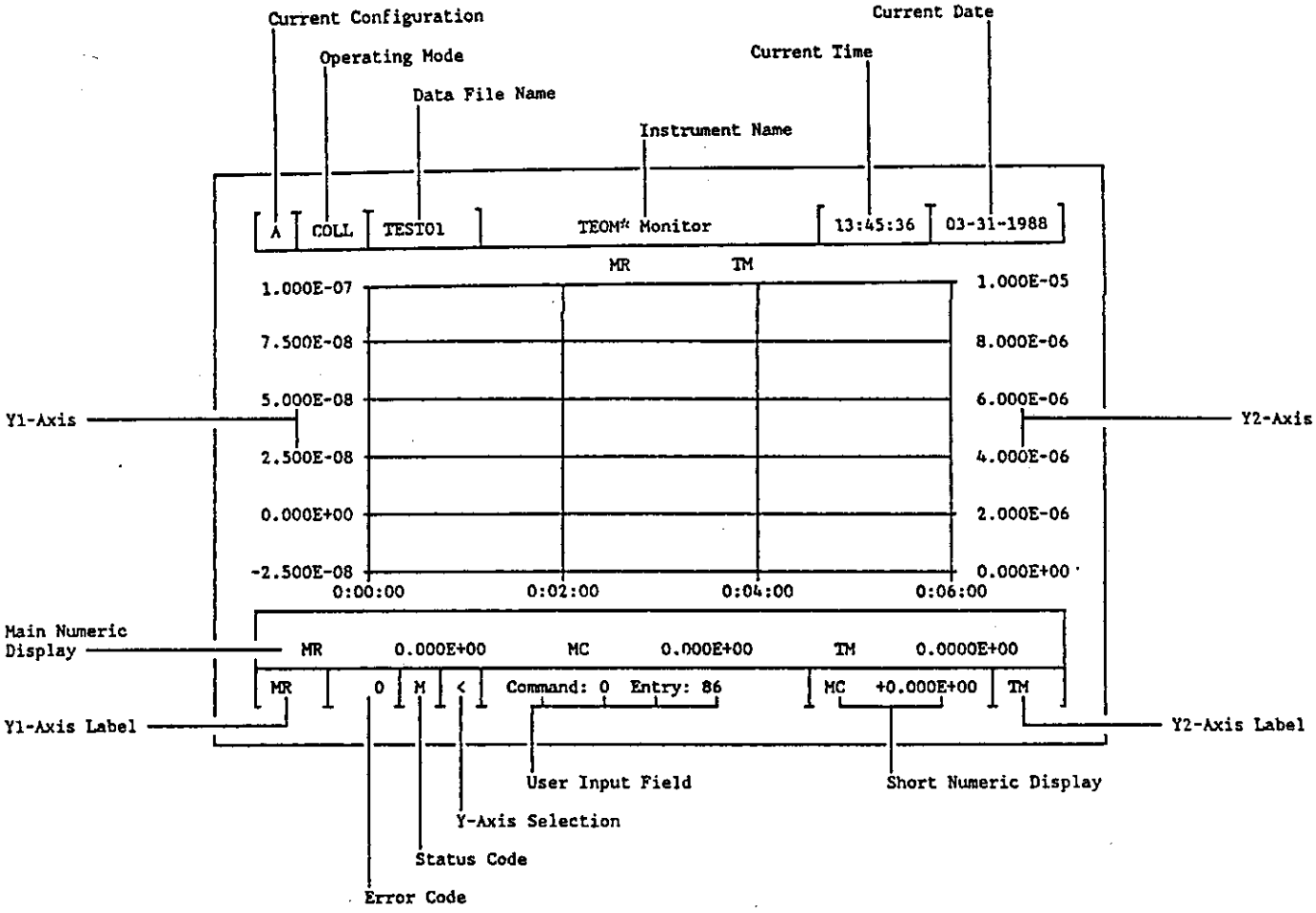

Figure 8. Components of the Main Display Screen of the TEOM® Particulate Monitor

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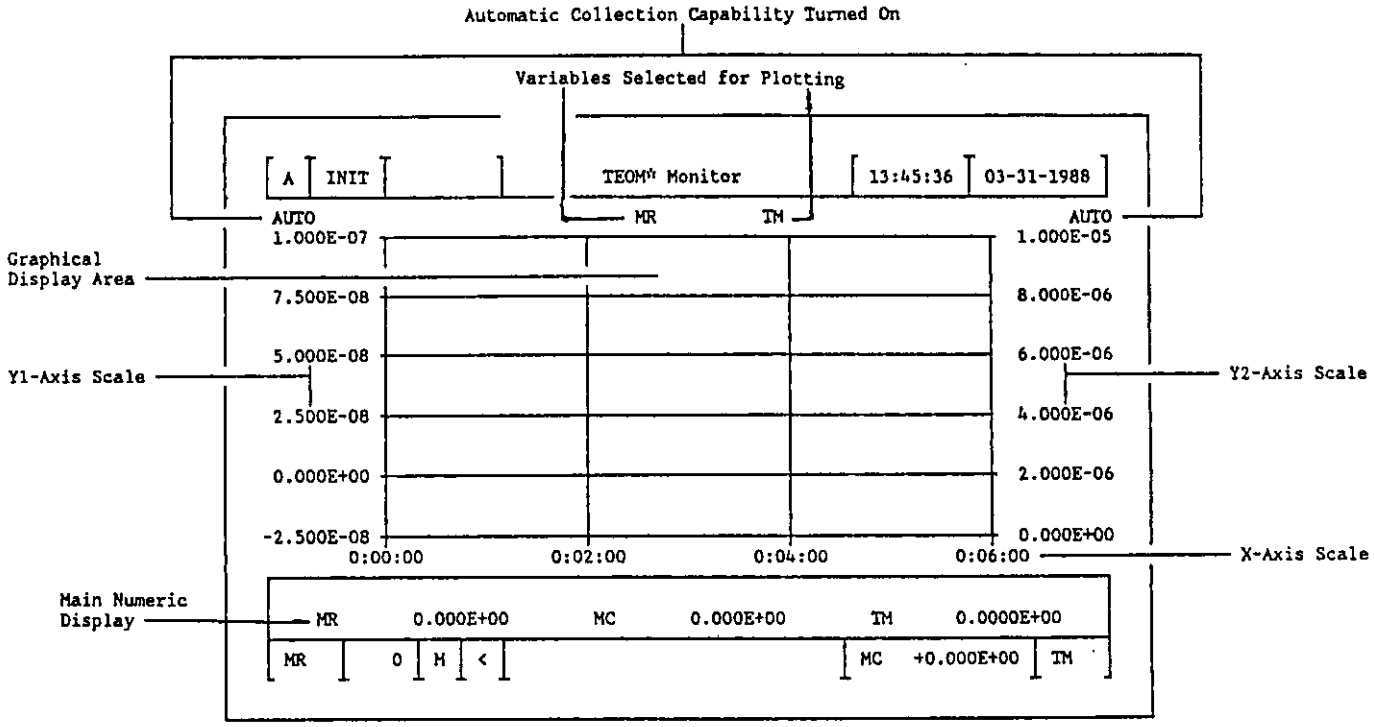

Figure 8. Components of the Main Display Screen of the TEOM® Particulate Monitor (cont'd)

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TP3 Programming

1. Description of the Instrument Cycle

This Section describes the steps involved in executing the instrument cycle of the TEOM® monitor. The instrument cycle is composed of the following modes:

Initialization Mode (INIT)	The instrument is in the INIT Mode when it is switched on, and after the main display screen has been cleared.
Collection Mode (COLL)	During the COLL Mode the TEOM® monitor collects mass rate, mass concentration and total mass data. The instrument plots and displays the information on the screen and saves data on disk.
Stop Mode (STOP)	The instrument enters the STOP Mode after the user has instructed the computer to stop data collection. In this mode, the user can print an image of the main display screen for future reference. After the screen is cleared in the STOP Mode, the monitor returns to the INIT Mode.

The function key commands used to switch the instrument from one operating mode to another are shown below:

F1	INIT Mode ------->	COLL Mode
F2	COLL Mode ------->	STOP Mode
F3	STOP Mode ------->	INIT Mode

Note: There is a Quick Reference Card which is supplied with the TEOM® monitor when purchased which provides a convenient summary of commands.

1.1 Executing the Instrument Cycle

Execution of these commands can only be done if the computer is equipped with the appropriate expansion boards.

1.1.1 Data collection (enter COLL Mode) - press F1 to start data collection. Entry into the COLL Mode is indicated by the mode indicator on the top line of the main display screen [see Figure 6(b)]. In this mode, the instrument collects, plots and displays mass rate, mass concentration and total mass data.
Note: If the instrument is configured to save data on disk, it requests a data file name in the User Input Field after F1 is pressed. In this case, enter one of these options followed by <Enter>.

- A data file name up to 8 characters long composed only of letters and numbers
- The number 0. In this case the computer automatically assigns a data file name according to the present date and time in the format:

mmddhhmm.PRN

where:
mm is the current month
dd is the current day
hh is the current hour
mm is the current minute

1.1.1.1 If the instrument is successful in creating the data file, the file name appears on the top line beside the COLL cell, of the main display screen [see Figure 6(b)]. All data files written by TP3 are given the extension .PRN for direct use with Lotus 1-2-3® spreadsheet software.

1.1.1.2 The Status Code on the bottom of the screen shows that data collection has begun. The status code M indicates that data collection has begun, but that the first total mass data point has not yet been calculated. A status code R means that total mass data are being computed, but that valid mass rate and mass concentration data have not yet been generated. the delays in computation are due to the averaging times selected for total mass, mass rate and mass concentration in the current configuration.

1.1.1.3 A blank status code indicates that valid data are being calculated for total mass, mass rate and mass concentration. If the status code D appears, the data disk has run out of capacity and data are no longer being saved. The program always closes data files in an orderly manner so that they are available for later evaluation

1.1.1.4 The variables plotted on the main display screen are indicated by the variable names shown just above the graphical display window of the main display screen. The definitions of the variable names may be found in Table 1.

1.1.2 Stop data collection (enter STOP Mode) - press F2 to stop data collection. Entry into the STOP Mode is indicated by the Mode indicator on the top line of the main display screen [see Figure 6(b) - STOP should replace COLL]. In this mode, the user may print an image on the main display screen by pressing F9. The user also has the option of returning to the INIT Mode or entering the Replot Mode.

1.1.3 Printing or restarting (enter INIT Mode) - press F3 to clear the screen and enter the INIT Mode. If data were stored on disk during the COLL Mode, the instrument asks the user in the User Input Field if he wants to enter the INIT Mode. Enter Y followed by <Enter> in response to this prompt to enter the INIT Mode. Entry into the INIT Mode is indicated by the Mode Indicator on the top line of the main display screen. From the INIT Mode, the user is able to execute commands to print data files, replot data, start another instrument cycle, or exit from the program. Refer to this Appendix, Section 1.1 to begin another instrument cycle.

1.2 Exiting the Program

The instrument must be in the INIT Mode to stop program execution and enter MS-DOS. Press F10 to exit from TP3 and enter MS-DOS. The instrument then asks in the user Input Field whether you want to exit from the program. Enter a Y followed by <Enter> to leave the program.
Note: Make sure that power has been turned off at the TEOM® Sensor Unit when the unit is not being controlled by the TP3 software.

2. Using Stored Data

2.1 Storage Format

All data files created by TP3 have the following attributes:

- The file name may be up to 8 characters long (letters and numbers), and is followed by the extension .PRN.
- Data files are stored in ASCII format, making them compatible with a wide range of commercially-available spreadsheet and word processing software. The files can also be read by programming languages such as BASIC, C and Pascal.
- The first 20 lines of each data file convey descriptive information about the instrument's hardware and software settings.
- The remaining part of the data file is made up of two or more columns containing real-time values for the variables stored on disk. The first column always contains the experimental time in seconds.

Table 2 lists the information contained in the 20 lines of the data files. the data file named BASELINE.PRN is provided in the C:/TEOMDATA subdirectory (hard disk systems) or on the TEOM® Data Diskette (floppy disk systems). The subdirectory C:\TP3 (or the provided floppy) also contains a LOTUS 1-2-3® template spreadsheet name AUTO3.WKS to aid in data analysis. The customer must own a copy of LOTUS 1-2-3® software to use the provided template file.

2.2 Replotting Stored Data in TP3

2.2.1 Data files may be replotted within the TP3 software by entering the Replot Mode. Data points may be replotted only if they have been saved on diskette or hard disk. The setting that causes the computer to store data on disk is part of the instrument's configuration. This parameter may be changed by entering the Configuration Definition Routine from either the INIT or Replot Mode.

2.2.2 The Replot Mode can be entered from either the STOP Mode or the INIT Mode. Press F7 when in the STOP Mode if the data file currently in the computer's memory is to be replotted. The TEOM® monitor enters the Replot Mode after this command is executed. Press F7 when in the INIT Mode to load a data file for replotting into the computer's memory. Then enter the name of the data file to be replotted (without the extension .PRN). The system then enters the Replot Mode. The same plotting, displaying and scaling commands are available in the Replot Mode as in the INIT and Collection

Modes. However, the F1, F2, and F2 ommand sequence used to guide the instrument cycle for data collection have different unctions in the Replot Mode.

2.2.2.1 The F1 command starts or re-starts the replotting of data. This command has no effect, however, if the replotting pointer has reached the end of the data file.
Note: Only those variables saved on disk may be replotted. All other variables are given a value of 0. The list of variables stored on disk during data collection is determined in the Configuration Definition Routine.

2.2.2.2 The F2 command stops the replotting of data. After replotting has stopped, the F9 command may be executed to print an image on the main display screen.
Note: Replotting may be resumed after F2 is entered by pressing F1 again.

2.2.2.3 The F3 command clears the screen and repositions the replotting pointer to the beginning of the data file. It also gives the user the option to re-enter the INIT Mode. Enter N to remain in the Replot Mode, or Y to re-enter the INIT Mode. Additional data files may be replotted by re-entering the INIT Mode and then executing the F7 command.

3. Configuration Definition Routine (CDR)

By entering the Configuration Definition Routine the user may define up to 26 different configurations. Each configuration has a single-letter name ranging from A to Z. When the computer is turned on, configuration A is automatically loaded into memory. To obtain a listing of the currently-defined configurations, enter ALT + C (hold down the ALT key and press C) when in the INIT Mode. The resulting display shows the full name of the files that store the operating parameters. These file names are made up of the instrument name, for example 1100 for the TEOM® Particulate Mass Monitor, and the configuration name ranging from A to Z. Press any key to return to the main display screen. Press F4 to load a different configuration into memory when in the INIT Mode or Replot Mode. AFter F4 has been pressed, the computer displays "Input New Config Name:" in the User Input Field. In response, enter the single-letter name of a different currently-defined configuration followed by <Enter>. The new configuration is then loaded into the computer's memory, and the settings of the new configuration are reflected on the main display screen. The name of the current configuration is changed in accordance with the user input.

3.1 Executing the CDR

The CDR can be executed when either in the INIT Mode or the Replot Mode. Press F8 when in the Initialization Mode. Press F8 when in the Replot Mode. This keystroke will only function if the replotting pointer is at the beginning of the data file, i.e., if you have just entered the Replot Mode or if you have just cleared the screen in the Replot Mode by pressing F3. The computer then lists the currently-defined configuration files in the TEOM® system. These file names are made up of the instrument model number, followed by single-letter configuration names. Press any key to continue.

3.2 Displaying the Configuration Screens (F1-F4)

The CDR allows the user to change the values of up to 80 program parameters displayed on four different screens. Screen 1 appears on the monitor when the routine is first executed. The number of the current screen is shown in the bottom right-hand corner of the display. The name of the current configuration appears in the lower left-hand corner of the screen. Keys F1 through F4 display screens 1 through 4. These commands may be entered in any order and as often as desired. Each screen contains 20 lines (slots) of information. Each of these Slots contains a description of a parameter, as well as the current value of the parameter.

3.3 Changing a Parameter's Value

Follow the steps below to change the value of a parameter, for example slot 0 (X-axis span): To change the value of parameter "X-axis span", slot 0 must appear on the computer monitor. If this is not the case, press F1 to choose screen 1. Press F6 to obtain the computer prompt "Slot:". Enter the number of the slot to be changed followed by <Enter>. In this case, type 0 followed by <Enter>. The computer responds by displaying ":". Type the new parameter value followed by <Enter>. To change the span of the X-axis to 3 minutes, enter 3 followed by <Enter>.

3.4 Saving the Present Configuration

Press F7 to save the current configuration on disk. (This keystroke saves changes made to the present configuration.)

3.5 Creating or Switching to Another Configuration

Press F8 to create or switch to another configuration. The computer displays the prompt "Enter File Name:". To create a new configuration, enter the single-letter name of a configuration that does not presently exist, followed by <Enter>. The new configuration initially takes the parameter values of the configuration presently loaded in the computer, or to load another configuration into the computer's memory, enter the single-letter name of an existing configuration, followed by <Enter>.
Note: The F8 command does not save changes made to the current configuration before loading a new configuration or loading a different existing one. Press F7 to save changes made to the current configuration before executing the F8 command.

3.6 Printing Configuration Information

Turn the printer on. Make sure that it is "on line", and that its print head is at the top of a new page. Press F9 to print the contents of the current configuration. When the F9 key is pressed in the INIT Mode, the user may choose to print the numeric contents of any data file stored on disk. The instrument is in the Print Mode during all of these printing operations.

Note: Because of the time required to print a screen image or the contents of a data file, the heating circuits in the TEOM Sensor Unit are turned off during printing. The user may have to allow for temperature to stabilize again before resuming data collection.

3.7 Exiting the CDR

Press F10 to exit to the main display screen and save the current configuration.

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COMPENDIUM APPENDICES

Appendix A	Abbreviations and Symbols
Appendix B	Definitions of Terms
Appendix C-1	Procedure for Placement of Stationary Active Samplers in Indoor Environment
Appendix C-2	Procedure for Placement of Stationary Passive Samplers in Indoor Environment

ACGIH	American Conference of Governmental Industrial Hygienists
AIHA	American Industrial Hygiene Association
ASHRAE	American Society of Heating, Refrigeration and Air Conditioning Engineers
ASTM	American Society for Testing Materials
B(a)P	benzo-a-pyrene
°C	degrees Celsius
cm	centimeter
cm^2	square centimeters
CO	carbon monoxide
COC	chain of custody
EPA	U.S. Environmental Protection Agency
°F	degrees Fahrenheit
ft	foot
ft^2	square feet
g	gram
HPLC	high performance liquid chromatography
in	inch
in^2	square inches
L	liter
L/min	liters per minute
m	meter
min	minute
mg	milligram
mm	millimeter
m^3	cubic meter
µm	micrometer
n	nano (10^{-9})
NBS	National Bureau of Standards
ng	nanogram
NIOSH	National Institute for Occupational Safety and Health
nm	nanometer
NO	nitric oxide
NO_2	nitrogen dioxide
NO_x	nitrogen oxides
PAH	polynuclear aromatic hydrocarbons
ppm	parts per million
ppm-hrs	parts per million-hours
QA	quality assurance
QC	quality control
RH	relative humidity

Accuracy	The difference between the measured value and the true value that has been established by an accepted reference method procedure. In most cases, a value is quoted by the manufacturer and no description is given to indicate how this value was obtained.
Active device	An instrument that employs a power source with a pump to pull the air across a sensing element or collector.
Air monitoring module	An assembly of air monitoring devices that are collected into one package to facilitate handling as a unit.
Analyzer	An analytical sampling device that determines the value of a pollutant concentration almost instantaneously.
Blank	A sample of the pollutant collection medium that is not exposed to air sampling but that is analyzed as part of the quality assurance program.
Calibration	The method for determining the instrument response to known-concentration gases (dynamic calibration) or artificial stimuli (static calibration).
Collection efficiency	The fraction of the incoming pollutant or parameter that is measured by the instrument.
Collector	A sampling device that collects a pollutant for subsequent laboratory analysis of pollutant concentration.
Fall time	The time interval between the initial response and a 90 percent response (unless otherwise specified) after a step decrease in the inlet concentration. This measurement is usually, but not necessarily, the same as the rise time.
Interferences	Any substance or species that causes a deviation of instrument output from the value that would result from the presence of only the pollutant of concern.
Lag time	The time interval from a step change in the input concentration at the instrument inlet to the first corresponding change in the instrument output.

Linearity	Expresses the degree to which a plot of instrument response versus known pollutant concentration falls on a straight line. A quantitative measure of linearity may be obtained by performing a regression analysis on several calibration points.
Long-term integrated	Techniques that produce an accumulated sample over an extended time period, such as a week.
Lower detectable limit	The smallest quantity of concentration of sample that causes a response equal to twice (sometimes 3 or 4 times) the noise level. (Not to be confused with sensitivity, which is response per unit of concentration.)
Microenvironment	A general location such as residence, office, car, bus, church, or supermarket that individuals move through during a 24-hour period of activity.
Monitor	The instrument or device used to measure air quality of meteorological parameters. Monitor also refers to the act of using the instrument or device.
Multi-parameter capability	Ability to measure other pollutants or parameters.
Passive	A sampling or analytical device that relies on diffusion to bring a pollutant in contact with a collector or an analyzer.
Personal monitors	Instruments for measuring pollutant concentration that can be worn conveniently on a person.
Portable monitors	Instruments that can be readily transported from one sampling location to another for personal or area sampling.
Protocol	Detailed scientific directions for performing a program.
Quality assurance	A system of activities that provides assurance that the quality control system is performing adequately.
Quality control	The activities performed that provide a quality product.

Range	The lower and upper detectable limits. (The lower limit is usually reported as 9.0 ppm. This is somewhat misleading and it would be better, however, to report it as the true lower detectable limit.)
Repeatability	The degree of variation between repeated measurements of the same concentration.
Reproducibility	The degree of variation obtained when the same measurement is made with similar instruments and many operators. In most cases, a value is quoted by the manufacturer and no description is given to indicate how this value was obtained.
Response time	The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent (unless otherwise specified) of the ultimate recorded output. This measurement is the same as the sum of lag time and rise time.
Retention time	The time interval from a step decrease in the input concentration at the instrument inlet to the first corresponding change in the instrument output.
Rise time	The time interval between the initial response and a 90 percent response (unless otherwise specified) after a step increase in the inlet concentration.
Sampling	The process of withdrawing or isolating a representative portion of an ambient atmosphere, with or without the simultaneous isolation of selected components for subsequent analysis.
Short-term integrated	Techniques for sampling frequencies that are generally on the order of hours to 1 day. Resulting data are capable of describing some aspects of short-term peaks.
Span drift	The change with time in instrument output over a stated time period of unadjusted continuous operation when the input concentration is a stated value other than zero. (Expressed as percent of full scale.)
Stationary monitor	An instrument that cannot be readily transported. This may be because of size, weight, the need to operate in a laboratory environment, fragility, or high maintenance requirements.

Warm-up time	The elapsed time necessary after startup for the instrument to meet stated performance specifications when the instrument has been shut down for at least 24 hours.
Zero air	Air which has been treated to ensure purity and lack of contaminants, that may be used to establish a zero reference point for an air quality analyzer.
Zero drift	The change with time in instrument output over a stated time period of unadjusted continuous operation when the input concentration is zero. (Expressed as percent of full scale.)

PROCEDURE FOR PLACEMENT OF STATIONARY ACTIVE SAMPLERS IN INDOOR ENVIRONMENTS

1. Scope

There are no standard practices available for selecting sampling areas for indoor environments. This procedure is intended to provide general guidelines in siting and locating stationary active samplers indoors. The purpose of this document is to ensure consistency of sampling site selection in indoor atmospheres.

2. Applicable Documents

2.1 ASTM Standards

D1356 Definition of Terms Relating to Atmospheric Sampling and Analysis

2.2 Other Documents

2.2.1 Wadden, R. A., and Scheff, P. A., *Indoor Air Pollution Characterization, Prediction, and Control*, ISBN: 0-471-8763-9, Wiley Interscience Publishing Co., New York, NY, 1983.

2.2.2 Nagada, et al., *Guidelines for Monitoring Indoor Air Quality*, Hemisphere Publishing Corp., New York, NY, 1987.

3. Summary of Method

3.1 Indoor air is collected by a stationary sampling system. The sampled air is either analyzed directly or stored in an appropriate container for later analysis.

3.2 Guidelines are given for determining sampling site location.

4. Procedure

4.1 The sampling inlet/probe of the stationary sampler should be located in an area that best represents peak pollutant concentrations experienced by the individuals occupying the area. The sampling locations may be in a general area such as a basement or warehouse. However, for more specific monitoring, samplers can be placed in a kitchen, living room, or office. A particular site within the area is selected to depict the air quality of the entire area.

4.2 Site selection in an occupied (i.e., lived in) area is primarily dependent upon occupancy patterns of the inhabitants as well as structural characteristics of the dwelling (i.e., age and building materials, type of appliances and furniture, and use of appliances). Additionally, emission source locations, available air volume to dilute source emissions, air circulation and exchange rate are important considerations when determining sampler location. In summary, the sampling area should be representative of the air quality in the indoor environment of concern, contents of the area, and occupant practices.

4.3 Once indoor sampling areas have been identified, inlet/probe locations may be determined. When selecting inlet/probe locations, the following areas should be avoided:

- areas of direct sunlight
- areas with noticeable drafts
- areas directly influenced by return or supply ducts
- areas that are directly impacted from outdoor sources
- exterior corners and walls
- probe heights below 1 m or above 2 m unless vertical gradients are being measured

4.4 Sampler instrumentation is also an important factor in selecting probe location. Samplers should be situated to minimize interference with indoor air. For unoccupied areas, major consideration should be given to sample flow rates (i.e., to avoid sampling system cleaning the air or contributing local exhaust) and heat sources. For occupied areas, especially residences, available space is an important issue.

4.5 New analyzers are compact for placement indoors and are configured to operate from battery power or to operate from household electric supply without interfering with normal occupancy. These systems generally require repackaging for use in the field. For systems with multiple analyzers and sophisticated data recording devices, a container is useful for transport and security. Before placing such an instrument indoors, the following questions should be answered:

- How many people will be needed to transport the monitoring package?
- What is the size of the smallest doorway through which the system is to be carried, including vehicles used to transport the package from place to place?
- Can a toddler pull or push it over?
- Will the size of the package interfere with normal use of the area by its occupants?
- Will the sampling system emit noise or odors that may be considered offensive to occupants?

4.6 If the system is to be operated from wall current, electric power is important for two reasons. The first is heat generated during operation of transformers, pumps, etc. If packaging confines natural ventilation around the instruments, the casing should provide for compensatory air movement with small fans or other devices. If sampling inlets are very close to the cabinetry, sampling results may be biased. The second aspect of electric power is the system amperage and grounding requirements. If monitoring is to take place in occupied structures, available circuits will be at a premium. A blown fuse or tripped breaker leads to lost data and guilt-ridden, if not infuriated, occupants. There are many structures that still have two-prong outlets; a "cheater plug" does not necessarily ensure a grounded connection. Inexpensive test devices are available to verify ground connections.

PROCEDURE FOR PLACEMENT OF STATIONARY PASSIVE SAMPLERS IN INDOOR ENVIRONMENTS

1. Scope

This document covers the placement and use of passive sampling monitors in the indoor atmosphere. The purpose of this document is to help ensure consistency of sampling within a variety of indoor environments and to facilitate comparison of monitoring data. This procedure may involve hazardous materials, operations, and equipment. This procedure does not purport to assess all of the safety problems associated with its use. It is the responsibility of whoever uses this procedure to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Applicable Documents

2.1 ASTM Standards

D1356 Definitions of Terms Relating to Atmospheric Sampling and Analysis Practice for Planning the Sampling of Indoor Air

3. Summary of Practice

3.1 Sample air is collected by adsorption onto a sorbent media or reacted with an appropriate chemical in order to subsequently undergo analysis for determination of concentration. The sampled air is circulated to the adsorption media or reaction chemical through diffusion.

3.2 Instructions are given for the handling and placement of passive monitors within an indoor environment.

4. Terminology

For definitions and terms used in this practice refer to D1356.

5. Significance and Use

5.1 Since analysis of the indoor environment is influenced by many factors except the method of sampling, an effort should be made to minimize interfering factors and maintain air at normal conditions in the area of the passive monitor.

5.2 Passive detection provides for time-integrated measurements. Passive monitors are usually placed in an indoor environment over a sampling period ranging from 3 days to 1 year. Due to the length of time involved with sampling, interfering factors should be anticipated and eliminated where possible.

5.3 Placement and recovery of the monitors can be performed by unskilled personnel with suitable instruction (even an occupant).

6. General Principles

6.1 Passive monitors rely on normal convection of air currents within an indoor environment for circulation of a representative sample atmosphere to the vicinity of the monitor. Subsequent collection of the sample component is performed through diffusion. Sampling adequacy is directly influenced by the ability of the monitor to be exposed to the representative sample atmosphere.

6.2 Variability of the results will decrease with consistency in sampling protocol as well as with increased sample component concentration.

7. Procedure

7.1 Predeployment Considerations

7.1.1 The occupants, if any, in the indoor environment to be sampled should not alter normal activities within the measurement period.

7.1.2 Deployment during remodeling or redecorating is not recommended. Changes in major furnishings such as stoves, HVAC systems, etc., should be avoided.

7.1.3 Deployment when seasonal alterations in insulation or building tightness are occurring or will occur during the measurement period should be avoided. (When long-term measurements on the order of months are being taken, this consideration is minimal.)

7.2 Measurement Conditions

7.2.1 Doors should be operated (opening/closing) in a manner consistent with normal occupancy. Windows should be kept closed when possible. Over an extended sampling period, the effect of a few days of open windows should be minimal on results.

7.2.2 The ventilation system should be operated in a manner consistent with normal occupancy.

7.2.3 The method of heating should not be altered during the sampling period. The normal occupancy heating method should be maintained.

7.2.4 The use of humidifiers/dehumidifiers is not recommended.

7.2.5 Normal occupancy activities should continue.

7.2.6 No effort should be made to additionally tighten the indoor environment or to provide additional ventilation.

7.2.7 The placement of the monitor should not prevent normal occupancy activity from occurring.

7.3 Deployment

The monitor should be deployed as soon as possible after receipt and within the limitations of the indicated storage life. A blank exposure should be retained for completeness utilizing an unexposed monitor of the same manufactured lot.

7.4 Placement

7.4.1 Indoor Atmosphere Considerations

7.4.1.1 The monitor should be situated in a location such that the monitor is exposed to representative sample air at normal conditions.

7.4.1.2 Humidity - Locations near sinks, tubs, showers, stoves, washers, driers, or humidifiers/dehumidifiers should be avoided.

7.4.1.3 Temperature - Locations near furnaces, vents, sinks, tubs, showers, electric lights, or electrically operated devices which may produce heat should be avoided.

7.4.1.4 Meteorologic - Locations of direct sunlight and seasonal or short-term meteorologic variations should be avoided (e.g., drafty windows or doors).

7.4.1.5 Airflow - Location in direct airflow such as near furnace vents, appliance fan vents, computer cooling fans, and HVAC intake/exhaust should be avoided. Areas where a known draft or pressure differential between areas of a building should also be avoided.

7.4.2 Spacial Considerations

7.4.2.1 The monitor should be placed in an open and unobstructed area where normal air convection will be encountered. The monitor should be placed at least 20 cm (8 in) below the ceiling, 50 cm (20 in) above the floor and 15 cm (6 in) from a wall. Outside walls should not be used if possible. Suspending monitors from the ceiling may be suitable.

7.4.2.2 The monitor should be placed in a position where disturbance will not occur during the measurement period.

7.4.3 Occupant Considerations

7.4.3.1 The monitor should be placed out of the reach of small children and pets.

7.4.3.2 The placement of the monitor, if not deployed by the occupant, should be agreeable and approved by the occupant.

7.5 Sampling

7.5.1 The sampling period begins when the lid or container of the monitor is removed at which time the time and date should be transcribed into a log book. A means of either resealing the monitor in the container or replacing the lid should be assured prior to the end of the sampling period.

7.5.2 Since damage could occur during shipping and handling of the monitor, inspect the monitor and package carefully.

7.5.3 The monitor should have a permanently attached identification code or serial number which should be transcribed into a log book. The log book should include information describing the location of the monitor and pertinent information regarding the building such as construction ty[illegible]eating system, insulation, occupancy number and patterns, and major appliance location. Include a diagram of the sampling location and building depicting the information listed in this subsection. If the occupant deploys the

monitor, sufficient instructions should be included regarding proper location and sampling conditions. A form should be included for easy collection of information necessary for log book entries.

7.5.4 If the monitor is deployed for other than a screening measurement, the monitor should be placed by a reliable professional familiar with the monitor used. For specific measurements, a deviation from the guidelines in Sections 7.2.1 through 7.4.2.2 is permissible.

7.6 Passive Monitor Recovery

7.6.1 The sampling period is terminated when the monitor is removed and sealed from the sample environment.

7.6.2 Record the time and date for measurement termination. Any damage or variance in the monitor since deployment should be noted in the log book.

7.6.3 Adequate information should be entered in the log book to permit interpretation of results and comparison to similar measurements. Any variation in the sampling location or building structure should be noted.

7.6.4 The monitor should be analyzed as soon as possible.

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