IMPROVE
STANDARD OPERATING PROCEDURES

SOP 251
Sample Handling

Date Last Modified   **Modified by:**
       09/12/96   **EAR**

SOP 251 Sample Handling

TABLE OF CONTENTS

LIST OF FIGURES

Technical References
TI 101A   Filter Procurement and Acceptance Testing
TI 101C   Filter Cassette Construction

1.0   PURPOSE AND APPLICABILITY

This standard operating procedure (SOP) describes the laboratory procedures for initializing and processing IMPROVE aerosol filter samples in preparation for analysis. Preparation of filters for sampling or analysis is the responsibility of the lab manager and lab technicians working for the Air Quality Group. Processing includes removing samples that do not meet the acceptance requirements for elapsed time or flow rate, and contacting site operators, as necessary, to repair damaged equipment or correct faulty collection techniques.

The filter preparation and sample handling is done in a building constructed in 1992 by the University to support the aerosol research of Crocker Nuclear Laboratory. It consists of an administrative room, a research and testing room, and a sample handling and gravimetric laboratory. The Air Quality Group Annex building (the Annex), houses the IMPROVE network staff and researchers, in addition to sample handling and gravimetric analysis equipment. Crocker Nuclear Laboratory, a controlled access facility, houses the hybrid integrating plate analysis system, the X-ray Fluorescence analysis system, the Particle Induced X-ray Emission analysis system, and the Proton Elastic Scattering Analysis system.

The sample handling and gravimetric laboratory is climate controlled and entry is situated such that there is no pass through foot traffic. Entrance requires that passage be made over a sticky floor mat material to capture dust and foreign particles. The laboratory area floor and work surfaces are vacuumed weekly with a high efficiency HEPA cleaner. All counter surfaces are cleaned once per week with reagent grade alcohol, though the areas in front of the electrobalances are cleaned daily. The entire Air Quality Group Annex building (the Annex), is on the same central air supply system. However, the air is separately filtered prior to entering the sample handling room, and the room is maintained at positive pressure to reduce fugitive dust levels.

The loading and unloading of filters and gravimetric analysis are performed in the sample handling laboratory, while receiving and shipping, sample tracking, leak checking, data entry, and communication with site operators are done in the research and testing room. Staff members have desks in the research and testing room, while researchers are housed in the administration section.

2.0 RESPONSIBILITIES

2.1 Laboratory Manager

The lab manager shall:
- oversee and maintain records on site and sampler operation
- supervise and train lab technicians
- review all log sheets for completeness, and to check the validity of the samples prior to downloading of the samples by lab technicians.
- resolve any inconsistencies on the log sheet or in the samples
- contact site operators regarding equipment or procedural problems
- oversee filter handling procedures
- order supplies, as necessary, for lab use
- clean and maintain the analysis laboratory

2.2 Laboratory Technician

The lab technician shall:
- pre weigh and individually identify filters for use at IMPROVE aerosol sampling sites
- load filters into sampling cassettes
- leak check sampling cassettes
- mail cassettes in shipping containers to sites
- receive exposed cassettes
- enter log sheets into the logs database
- download filters for Ion Chromatography or Thermal Optical Reflectance analysis into labeled petri dishes
- post weigh exposed filters

3.0   REQUIRED EQUIPMENT AND MATERIALS

The equipment and materials required for filter handling are listed below.

3.1 Filter and Cassette Requirements

- Quality tested and approved stretched Teflon membrane filters
- Quality tested and approved Nylasorb™ filter medium
- Acceptance tested Quartz filters prepared by the Thermal Optical Reflectance Analysis Laboratory
- Acceptance tested Quartz filters impregnated with K_2CO_3 and glycerol prepared by the SO_2 analysis laboratory
- Filter cassettes parts, and completed cassettes constructed and assembled as described in TI 101C, and in Figure 1 below.

Figure 1 IMPROVE Filter Cassette Parts
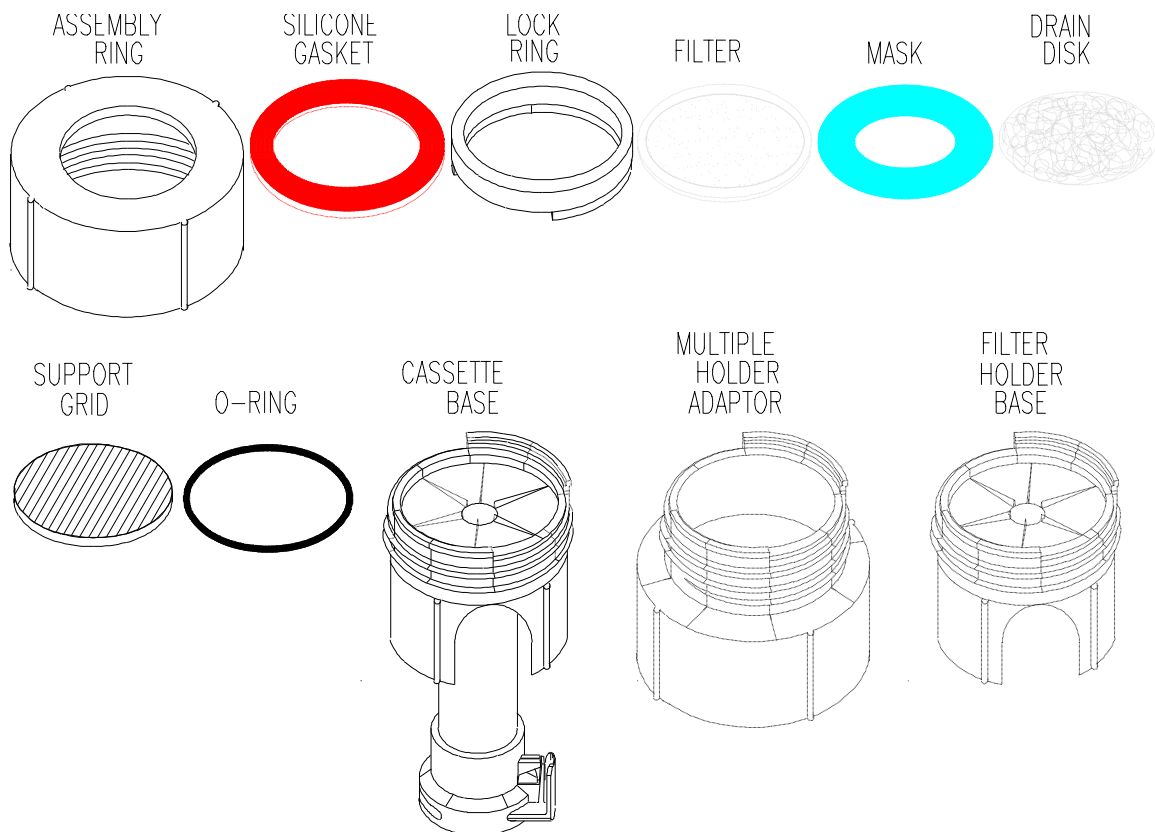
- Assembly ring
- silicone gasket
- lock ring
- 22mm support grid
- ethylene propylene and Viton™ o-rings
- cassette base

- multiple holder adapter (for C/C, D/S, A/S filters)
- 25mm diameter, 2.2cm^2 area mask
- Gelman 22mm drain disk
- Forceps, stainless steel, blunt tipped
- Red protective caps for filter cassettes
- yellow protective hose covers for field blank cassettes
- site and sampling date stickers for sampling cassette labeling
- 47mm petri dishes
- Ziplock™ two quart plastic bags
- Cassette shipping boxes
- reagent grade alcohol
- Kimwipes™

3.2 Filter Weighing Equipment

- Cahn electrobalance, sensitive to $\pm$ 1μg
- Computer capable of running MicroSoft FoxPro®
- Polonium strip ionization units
- Calibration weights, 200.000 mg class 1.1 (Class M) mass, 20.000 mg class 1.1 (Class M) mass
- tare weight, 50.000 mg class 1.1 (Class M) mass
- zero area bail for electrobalance
- nylon forceps
- reagent grade alcohol
- Kimwipes™

3.3 Leak Check Equipment

- leak check device, see schematic C76-NPS-2664
- air pump for leak check device
- 30 mm diameter silicone pipette bulb
- forceps
- clean petri dishes
- leak check card (to verify filter identification, and cassette integrity prior to mailing cassettes to the site)
- reagent grade alcohol
- Kimwipes™

3.4 Exposed Filter Processing Equipment

- cassette and box tracking chart
- Computer capable of running MicroSoft FoxPro®
- 47mm petri dishes for ion and carbon analysis filters
- stainless steel blunt tipped forceps
- reagent grade alcohol
- Kimwipes™
- brush for cleaning quartz filter cassettes

- Three numbered, 50 position, Petri dish trays, one for each non-Teflon™ filter substrate
- Petri dish shipping trays
- shipping boxes
- Blue Ice™ cooler packs

4.0   METHODS

Sample handling refers to the preparation of filters for use in the field, and the initial processing and gravimetric analysis of the returned filters in preparation for compositional analysis by Ion Chromatography, Thermal Optical Reflectance, or Hybrid Integrating Plate/Sphere Method followed by X-ray Fluorescence, Particle Induced X-ray Emission and Proton Elastic Scattering Analysis. Sample handling entails only the work done in the filter processing room of the Air Quality Group annex. Field procedures used by the site operators are covered in SOP 201.

There are eight steps involved in the sample handling procedure used for the IMPROVE network.

4.1 Purchase and Preparation of the Filters, Cassettes, and Module A masks at UC Davis and at the Carbon and SO2 laboratories. (see SOP101, TI 101A, and TI 101C)
4.2 Programs and Databases for Filter Handling
4.3 Electrobalance Controls and Calibrations.
4.4 Pre-Measurement of the Gravimetric Mass and Uploading of Filters into Cassettes
4.5 Leak Checking of Cassettes and Preparation for Shipping to the Field
4.6 Receipt of Boxes from the Site and Entry of Data into Computer
4.7 Downloading of the Exposed Filters for Ion Chromatography or Thermal Optical Reflectance Analysis
4.8 Downloading of Exposed Filters for Post-Measurement of the Gravimetric Mass and Transfer of Exposed Filters to Slide Frames for Elemental Analysis

4.1   Purchase and Preparation of Filters and Cassettes

The procedures for purchasing, preparing, and assembling filters and cassettes for the IMPROVE sampling network are covered in SOP101, Procurement and Acceptance Testing Procedures for IMPROVE Aerosol Sampling Equipment and Supplies. Details of the filter acceptance testing and preparation procedures are listed in TI 101A. Details of the cassette preparation and assembly are covered in TI 101C. Therefore, this material will not be repeated in this section; interested persons may refer to the documents listed above for information.

4.2 Programs and Databases for Filter Handling

Filters are initiated and tracked using a PC based data handling system composed of a controlling database which accesses and organizes data from several smaller databases. Programs to input and access data from the databases have been created. The basic database structure, and the programs used to access and connect the databases, are shown below in chart 1.

Chart 1 Programs and Databases

OUTPUT DATA FILES

AUXDATA

DATA OUTPUT
PROGRAM

| WEIGHTS DATABASE | IONS DATABASE | CARBON DATABASE | SO2 DATABASE | LASER DATABASE | ELEMENTAL DATABASE |

DATABASES

PRE   POST   CHANBCS   LASER   TRAY

PROGRAMS

LOGS DATABASE

PRIMARY
DATABASE

LOGSIN

DATA INPUT
PROGRAM

FIELD LOG
SHEETS

FIELD DATA
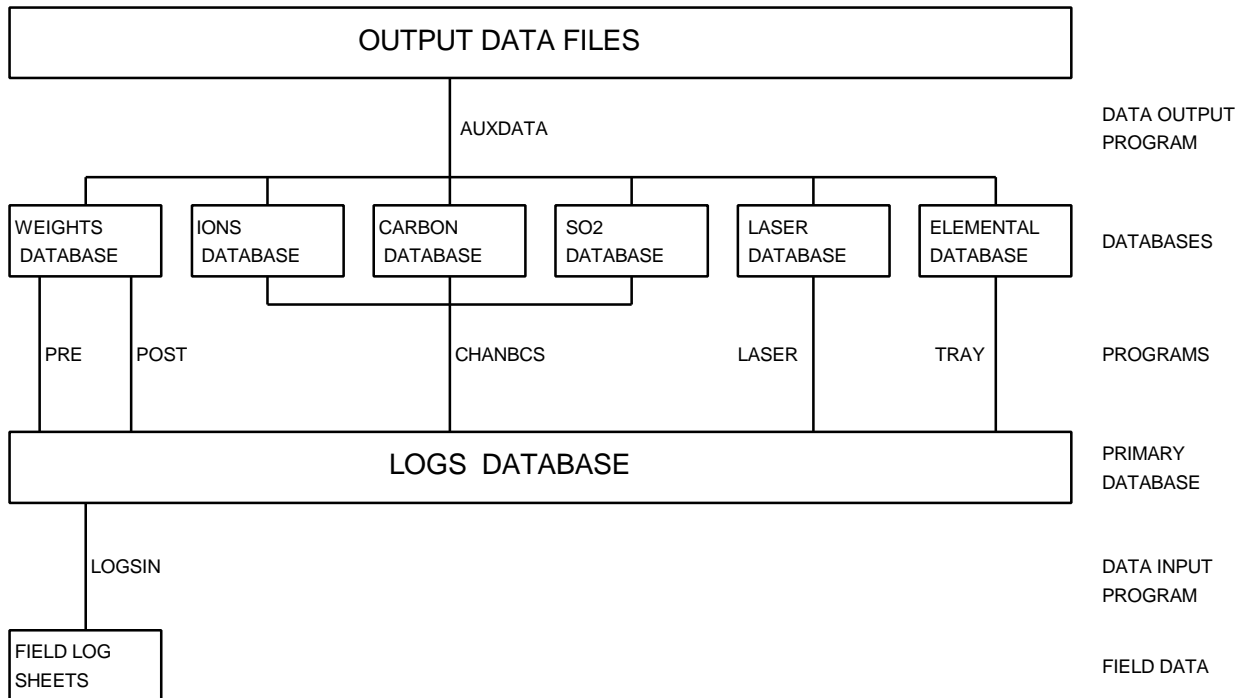
4.2.1 Databases
LOGS

The controlling database is the LOGS database. This database is organized to include all data for a single site/sample date in one record. Each record includes all the information from each module at a site for one sample date. Site, sample date, pump vacuum for each module, initial and final small gauge vacuum readings for each module, initial and final magnehelic gauge readings for each module, start time, current temperature, elapsed time for each module, status of each sample, person entering the data, date the samples returned from the field, quarter, comments on status, and area of the sample are fields included in the LOGS database. Since the LOGS database is the primary database, it is protected and input, other than through the programs listed in Chart 1, is restricted.

WEIGHTS

The WEIGHTS database, tied to the LOGS database through the PRE and POST programs, is organized to include mass and identification data for each filter in a single record. Each record contains a filter identification number, site, sample date, channel, status, pre weight, post weight, sample weight, pre weight time and date, post weight time and date, electrobalance used for pre weight, electrobalance used for post weight, person entering the data, expected start time, expected sample duration, quarter, and analysis tray position.

PRETARE

The PRETARE database, tied to the WEIGHTS database through the PRE program, is organized to include the tare weight for conversion of Cahn 25 weights to Cahn 31 equivalent weights, the correlation between the Cahn 31 weights and the Cahn 31 equivalent weights for the 20 controls filters weighed to derive the equation,

and the starting date for the new equation in a single record. New equations are derived and entered by the lab manager.

C25CALIB, C31CALIB

The C25CALIB and C31CALIB databases are accessed through the PRE or POST programs, and are organized to include the name of the balance operator, the date and time of calibration of the balance, the zero weight value, the calibration weight value, the test weight value (tare weight for the Cahn 25), and the magnetic field reading. These are recorded whenever the balance is calibrated. C25CALIB contains Cahn 25 calibration data, and . C31CALIB contains Cahn 31 calibration data.

CONTROLS

The CONTROLS database is accessed through the PRE or POST programs, and is organized to include the following information for each balance (Cahn 25 and Cahn 31): filter identification name; Pre weight date; Post weight date; # of days between Pre and Post weighing of the filter; Pre, Post, re-Pre and re-Post weights; Post minus Pre weight; re-Post minus re-Pre weight; Post minus re-Post weight; Pre minus re-Pre weight; differences between the Pre, Post, re-Pre and re-Post weights measured on the Cahn 31 and Cahn 25; name of the balance operator responsible for each measurement; relative humidity during each measurement; and temperature of the room during each measurement. These are calculated and recorded when the control filters are weighed.

IONS

The IONS database, tied to the LOGS database through the CHANBCS program, is organized to include ionic species and filter identification for each Nylasorb™ filter sample. All data for each filter is recorded in a single record. Each record contains the site, sample date, start time, status, Cl- concentration, NO_2^- concentration, NO_3^- concentration, and $SO_4^=$ concentration. All concentrations are in ng/m^3 .

CARBON

The CARBON database, tied to the LOGS database through the CHANBCS program, is organized to include the eight measured carbon channels, five composite carbon variables, and filter identification for each quartz filter sample. All data for each filter is recorded in a single record. Each record contains the site, sample date, start time, status, replicate markers, five composite carbon variables, four fields of organic carbon concentrations, one field for carbon concentrations derived during pyrolysis of the filter, and three fields of elemental carbon concentrations. All concentrations are in ng/m^3 .

SO2

The SO2 database, tied to the LOGS database through the CHANBCS program, is organized to include ionic species and filter identification for each impregnated quartz filter sample. All data for each filter is recorded in a single record. Each record contains the site, sample date, start time, status, and $SO_4^=$ concentration. All concentrations are in ng/m^3 .

LASER

The LASER database, tied to the LOGS database through the LASER program, is organized to include transmission and reflectance measurements, calculated uncorrected absorption values, and filter identifications for each channel 'A' filter. This data, for each filter, is recorded in a single record. Each record contains status, site, sample date, start time, channel, elapsed time, flow rate, transmission, reflectance, absorption values uncorrected for sample density, measurement errors, measurement minimum detectable limit, initials of person collecting data, and date and time of measurement.

PIXE

The PIXE database, tied to the LOGS database through the TRAY program, contains the elemental concentrations derived from XRF and PIXE, and filter identification for each channel 'A' filter. For each filter, this data is arrayed in a single record. XRF elements recorded include species from Sulfur through Zirconium. PIXE elements recorded include species from Sodium through Lead. For each species, the following are recorded in ng/m^3: elemental concentration, error in concentration, and minimum detectable limits.

OUTPUT DATA FILES

The OUTPUT DATA FILES are site and quarter specific. The files are stored on the FTP site maintained by Fort Collins. Not all collected species are reported in the OUTPUT DATA FILES. Only forty-two elemental, ionic, and carbon channel species are reported for each sample.

4.2.2 Programs

PRE

The PRE program accesses the LOGS database to develop a list of filters to be uploaded into cassettes. Next, it queues the filters for weighing, and records pre weight values from the electrobalance to the WEIGHTS database. Finally, it records the site code, sample date, and start time to the LOGS database, thereby completing the initialization of the records for each filter being sent to the field. If the pre weight/post weight method is replaced by beta-gauge measurements of mass, PRE will be replaced by a program called UPLOAD which will be identical to PRE except it will not access of the WEIGHTS database. A simplified schematic of the functioning of PRE follows in Chart 2.

Chart 2 PRE program schematic
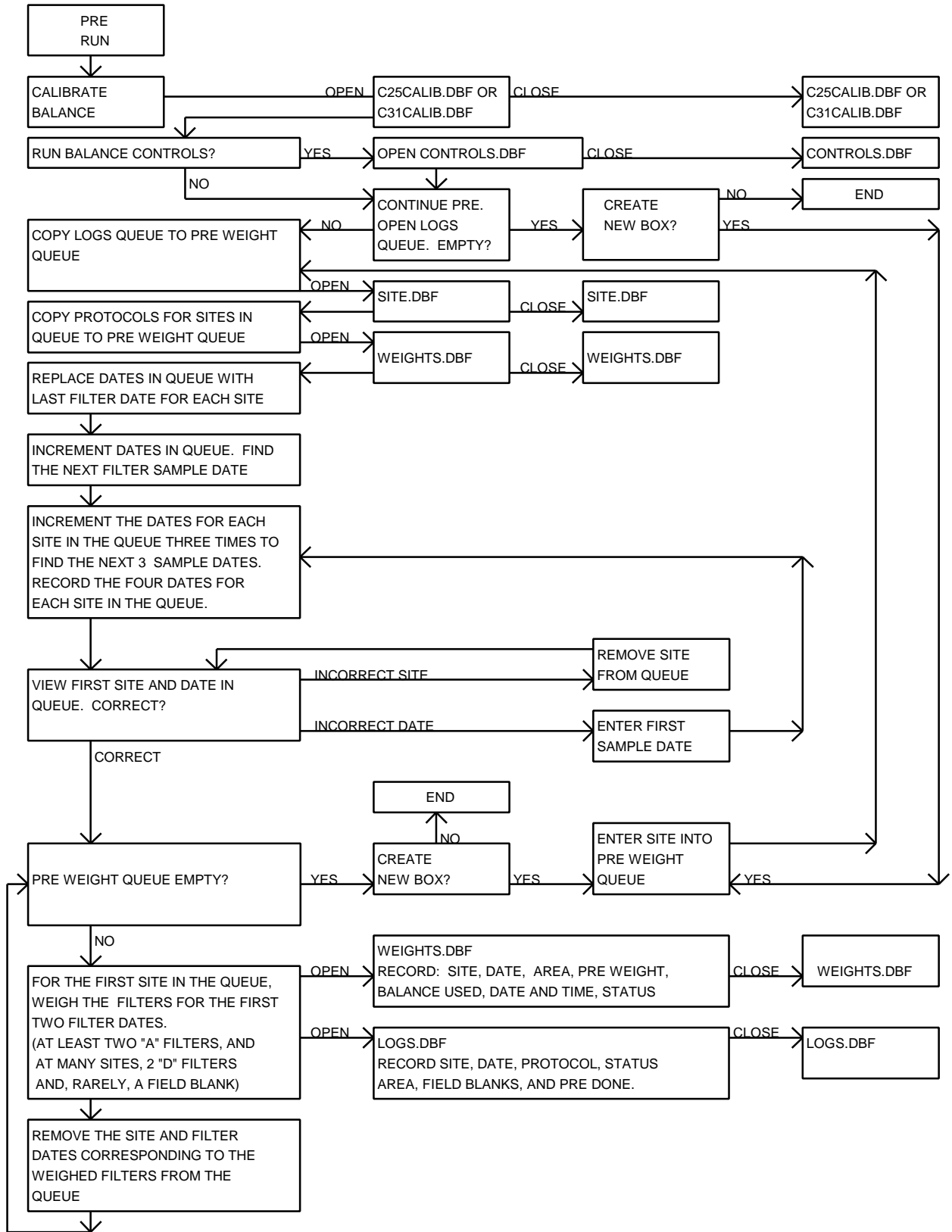
LEAK CHECK

The LEAK CHECK program is a quality assurance and data accounting program. It accesses the LOGS database to check for agreement between the sampling protocol displayed in the box, and the protocol described in the LOGS database. Once the contents of each shipping box have been verified and the cassettes checked for leaks, the LEAK CHECK program adds the current date and the box identification to a temporary file of boxes not yet returned from the field. This provides a quick reference of which boxes were shipped and when they were shipped.

LOGSIN

The LOGSIN program records data from the field log sheets for exposed filters in the LOGS database. Through this program, temperatures, pump vacuum readings, vacuum gauge and magnehelic gauge readings, elapsed sampling time, and filter status are recorded in the LOGS database for each filter.

POST

The POST program accesses the LOGS database to develop a list of exposed filters to be downloaded from cassettes. Next, it queues the exposed filters for weighing, and records post weight values from the electrobalance into the WEIGHTS database. Finally, it displays the tray and position in which the filter should be mounted, and notes the record is complete in the LOGS database. If the pre weight/post weight method is replaced by beta-gauge measurements of mass, POST will be replaced by a program called DOWNLOAD which will be identical to POST except it will not access of the WEIGHTS database. A simplified schematic of the functioning of POST follows in Chart 3. A new program, BETAWTS, will be used to collect beta-gauge measurements of mass for transfer to the WEIGHTS database, should a Beta-gauge system be installed.

CHANBCS

The CHANBCS program accesses the LOGS database to organizes the B channel (ions), C channel (carbon), and S channel (SO2) filters for shipment to the analysis contractors. Organization is by date and time the log sheet was entered into the LOGS database. The display shows the site name, sample date, and status of each filter. Filters are organized for shipment to the analysis contractor according to the CHANBCS queue. When four complete boxes of ions, carbon, or SO_2 filters are available, CHANBCS creates a file containing the organization information to send to the contractor with the filters. This file is used for quality assurance to compare the LOGS database with the actual filters, and is used by the contractors as a template for reporting their data to the Air Quality Group.

LASER

The LASER program accesses the LOGS database to organize a quarterly analysis queue. Organization is by site, sample date within the requested quarter, and start time. The LASER program collects data from the hybrid integrating plate system (HIPS), associates it with the filter identification information from the LOGS database, and stores the resulting record in the LASER database. The database, when complete, contains status, site name, sample date, start

time, channel, elapsed time, flow rate, transmission, reflectance, name of operator collecting data, and date and time collected. The LASER database also includes the calculated values of filter absorption uncorrected for loading effects, with error and minimum detectable limits for all values.

TRAY

The TRAY program accesses the LOGS database to organize an analysis queue by quarter for XRF and PIXE. Organization is by site, sample date within the quarter requested, and start time. The files created by the TRAY program are used to queue the XRF and PIXE/PESA analyses.

Chart 3 POST program schematic
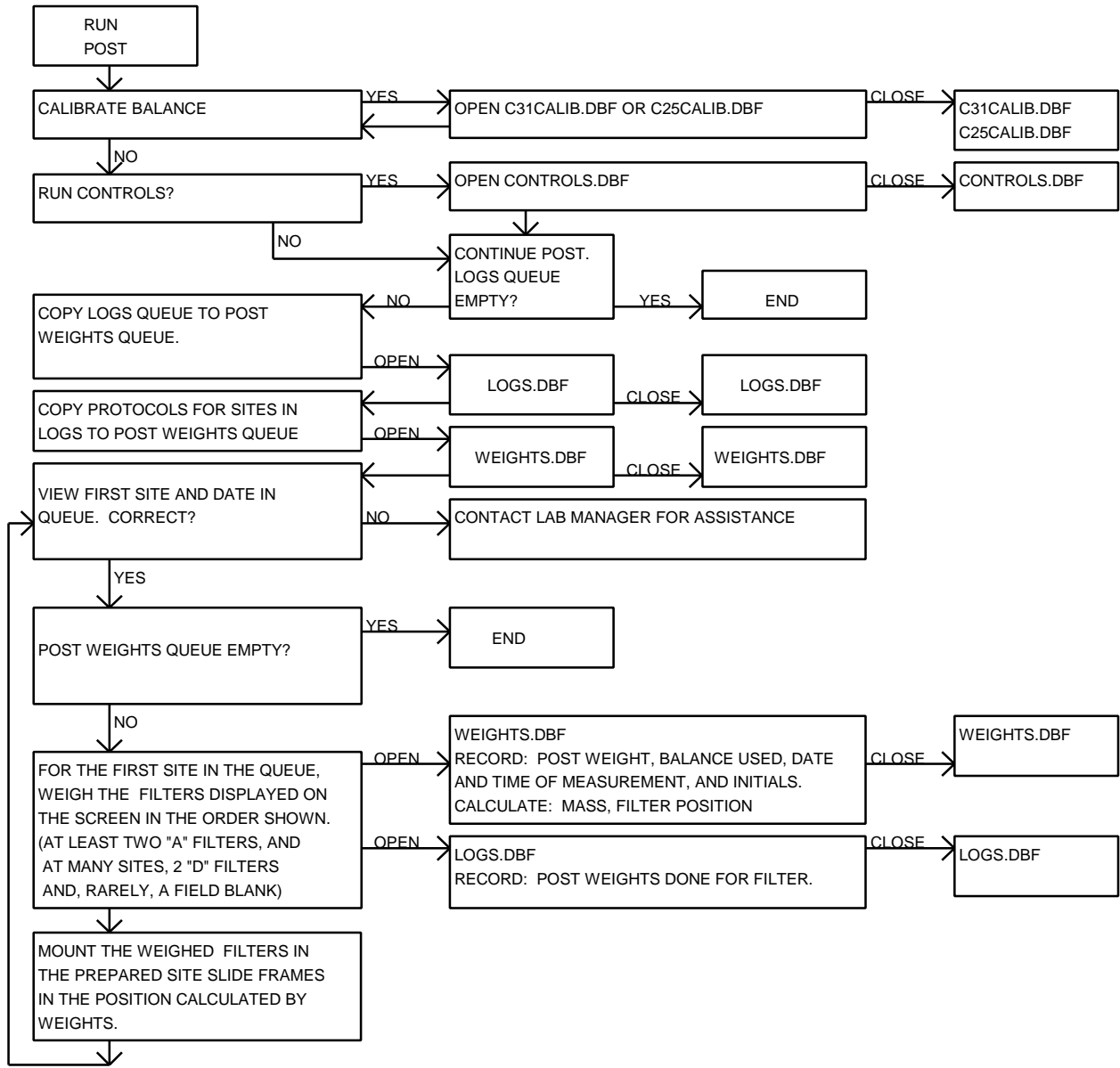
AUXDATA

The AUXDATA program accesses the secondary databases for two purposes: to allow quality assurance of the data, and to create final ASCII data files, as shown in Chart 1. AUXDATA organizes by site, sample date, and start time. Using AUXDATA, the quality assurance specialist can edit the status, site name, and sample date in the secondary data files, as necessary, to correct inaccurate data entries. AUXDATA also calculates error values for the ions, carbon, and SO2 data, and selects data from the other databases to include in the final data files. The final ASCII data files contain the following: site, sample date, start time, sample duration, flow rate, B_{abs}, and concentrations, errors and minimum detectable limits of $PM_{2.5}$ mass, PM_{10} mass, H, Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Pb, Se, Br, Rb, Sr, Zr, Mo, $SO_4^=$, Cl^-, NO_2^-, NO_3^-, SO_2, and organic and elemental carbon channels.

4.3 Electrobalance Controls and Calibrations.

Gravimetric mass analysis is performed using Cahn 31 and Cahn 25 Electrobalances modified with a zero area bail and vertical counterweight. Polonium strip ionization units are used to reduce electrostatic effects in the weighing cavity and on individual filters. A segregated laboratory area is used to control human traffic and to stabilize the temperature and relative humidity of the weighing environment. The area is cleaned with a high efficiency HEPA vacuum cleaner, and a tacky floor covering is installed at the entrance to the sample handling room to minimize dust artifact.

Gravimetric analysis of IMPROVE filters currently uses the difference method to determine the mass of the collected aerosol. The pre weight of each filter is measured prior to being loaded into a cassette and sent into the field for sampling. Once exposed and returned to the sample handling room, the filter is removed from the cassette, and the post weight of the filter is measured. The mass of the aerosol is determined by calculating the difference between the pre and post weights.

The weighing procedures are directed by sample handling computer codes, the PRE and POST programs described in Section 4.2. Both the PRE and the POST program run on either balance (Cahn 25 or Cahn 31 electrobalances), but the Cahn 31 balance is typically used for post weights, and the Cahn 25 is typically used for pre weights. The output of the balances are entered directly into the computer database, with the technician directing, via keyboard, the data collection and storage.

Correlating the weights measured on the two balances is done through an equation based upon the daily controls weights. Any significant change in the equation is an indication of damage to one or both of the balances. Repairs are effected and a new correlation equations is developed to describe the relationship between the two balances prior to resumption of weighing on the damaged balance. The balances are left on at all times to reduce wear on the equipment, and to ensure consistent, stable operation.

4.3.1 Cleaning and Maintenance of the Sample Handling Room

The requirements for a sample handling room include a reduced dust environment, and, over the twenty-four hour period prior to analysis of exposed filters, temperature in the range of 15º C to 30º C with variation less than ± 3º C, and relative humidity in the range of 20% to 40% with variation less than ±5%.

To reduce dust levels in the sample handling room, the room is over-pressured, with the inlet air passing through a high efficiency filter to reduce daily dust build up. Every Friday, the sample handling room is thoroughly cleaned, after insuring that all filters have been protected against contamination. To reduce fugitive dust levels, all surfaces are cleaned with a high efficiency HEPA vacuum. The floors are cleaned with a mild cleaning solution, if necessary. Finally, all work surfaces are cleaned with reagent grade alcohol (or another reagent grade solvent, if necessary) and Kimwipes™. This procedure reduces the possibility of contamination should a filter fall to the work surface. Following the Friday cleaning, no analysis shall occur for at least twenty-four hours to reduce the potential for contamination of filters by compounds used in the cleaning process.

Temperature control is through a central heating/air conditioning unit used for the entire Air Quality Group Annex building. The set temperature is 22.2º C, and the sample handling room rarely varies more than ± 3º C from that value over a twenty-four hour period. When the temperature range is exceeded, analysis is discontinued until the temperature fluctuation is corrected.

Relative humidity is set at 35% with an allowed twenty-four hour variation of ±5%. When the relative humidity is too low, a humidifier is utilized to increase the humidity to acceptable levels. When relative humidity exceeds 40%, a dehumidifier system is triggered to reduce humidity levels. As the entire Air Quality Group Annex building runs off a central air processing system, humidity control involves large volumes of air.

4.3.2 Calibration and Maintenance

The balance is cleaned and calibrated every day at 8 am and 1 pm. It is also recalibrated if the balance fails a "zero" test that is performed periodically. The procedures described here are outlined in the Cahn 31 Electrobalance Instruction Manual in Section 4, Operations.

A calibration log database is maintained for each balance; C25CALIB for the Cahn 25, and C31CALIB for the Cahn 31.   All calibrations are recorded in these databases. Significant events concerning the balance and any balance maintenance other than routine procedures are recorded in the log of the lab manager.

Procedures performed by the lab manager are outlined in section 4.3.2.1.

The steps for cleaning and calibration are as follows:

1.   Clean the metal and plastic forceps with ethanol and a Kimwipe™.

2.   Clean the work surface around the balance with ethanol and a Kimwipe™

3.   Clean the top surface and the strips of the anti static ionizing units by gently rubbing with a Kimwipe™ wetted with ethanol. Do not neglect to clean the ionizing unit in the electrobalance.

4.   Replace the clean ionizing unit in the center back of the balance cavity, and close the door on the weighing chamber.

5.   Start the PRE or POST program (generally, run PRE on the Cahn 25, and POST on the Cahn 31).

6.   Follow the order dictated by the program. Instructions will appear next to the flashing cursor, or in the message box at the top of the screen. Enter your initials, then select the 'CALIBRATE BALANCE' option in the message box at the top right of the menu screen.

7.   For the Cahn 25 only, remove the test weight from the tare bail. Note, the test weight is a small weight that hooks over the zero area tare bail, (not over the weighing bail). Do not remove the tare bail. Place the test weight in the small black container inside the weighing chamber for safe storage during the calibration procedure.

8.   Allow the balance to stabilize with no weights on the pan. The computer will automatically record the mass to the screen when the balance has stabilized; this is the "zero" mass. It should be within 0.010 mg of 0.000 . If not, contact the lab manager (see step 3, section 4.3.2.1 for lab manager procedures)

9.   If the zero value is unstable, select the "DISCARD AND RETRY" option in the message box, then wait for the balance to stabilize and for the computer to record a new weight. Once the zero value is stable, select the "KEEP AND CONTINUE" option in the message box at the top of the screen. This will place the value recorded to the screen in step 9 in permanent memory, as a record of the drift of the 'zero' for the balance.

10.   Set the zero on the balance:
      a)   For the Cahn 31 balance, depress the tare button on the balance. This forces the "zero" mass to be exactly 0.000.
      b)   For the Cahn 25 balance, unlock the 'zero' knob on the front of the balance by moving the black lever on the knob one quarter turn clockwise. Use the fine and coarse adjustment dials to set the zero to exactly 0.000. Lock the

zero by moving the black lever counterclockwise into its vertical, 'locked' position.

11. Calibrate the balance. Momentarily ground yourself by touching the balance casing. Use nylon forceps to remove the certified calibration weight from its container. Gently place it in the center of the zero area bail (balance pan) and allow the mass reading to stabilize and stop decreasing. The computer will record a reading to the screen when the balance has stabilized.
    a) Use a 200.000 mg class 1.1 (Class M) mass to calibrate the Cahn 31.
    b) Use a 20.000 mg class 1.1 (Class M) mass to calibrate the Cahn 25.

12. If the calibration value is unstable, select the "DISCARD AND RETRY" option in the message box, then wait for the balance to stabilize, and the computer to record a reading to the screen.

13. Once the calibration value is stable, select the "KEEP AND CONTINUE" option in the message box at the top of the screen. This will record the initial value for the calibration weight, from step 12, to a permanent file as a record of the drift of the "calibration" weight for the balance. Once the computer has recorded a value, set the calibration point.
    a) For the Cahn 31 balance, depress the calibration button on the balance. This forces the "calibration" mass weight to be exactly 200.000.
    b) For the Cahn 25 balance, unlock the 'calib' knob on the front of the balance by moving the black lever on the knob one quarter turn clockwise. Use the fine and coarse adjustment dials to set the calibration to exactly 20.000. Lock the calibration value by moving the black lever counterclockwise into its vertical, 'locked' position.

14. Remove the calibration weight from the bail, using the nylon forceps, and replace it in its storage container.

15. Check the calibration of the balance using the test weight. Momentarily ground yourself by touching the balance casing. Use the nylon forceps to remove the test weight from its container.
    a) For the Cahn 31, place the test weight in the center of the zero area bail (balance pan) and allow the mass reading to stabilize and stop decreasing. The computer will record a reading to the screen when the balance has stabilized. The test weight is an old 50.000 gm calibration weight.
    b) For the Cahn 25, hang the test weight on the tare bail (right side of the balance), and allow the mass reading to stabilize and stop decreasing. The computer will record a reading to the screen when the balance has stabilized.

16. If the test weight value is incorrect or unstable, select the "DISCARD AND RETRY" option in the message box, then repeat step 16. Once the test weight

value is correct and stable, select the "KEEP AND CONTINUE" option in the message box.

17.   For the Cahn 31 only, remove the test weight, using the nylon forceps, and replace it in its container. The Cahn 25 test weight is actually the filter tare, and must be on the tare bail whenever a filter is being weighed.

18.   Enter the current temperature from the thermometer above the balance. Measure and enter the ambient humidity from the gauge at the Cahn 31 balance. Finally, enter the magnetic field strength from the gauge on the wall on the west side of the weight room.

19.   Allow the balance to return to "zero." Compare the zero value and the value determined for the 50.000 mg mass to the expected values posted on the balance. If they exceed ±2 micrograms, repeat the procedure. If variations greater than ±10 micrograms are observed, report to the laboratory manager so that he/she can take appropriate action (section 4.3.2.1 step 3).

20.   On a random basis, but at least semiannually, the laboratory supervisor shall request a comparison of the normal calibration standards with a master set of reference standard masses maintained by the laboratory supervisor. After calibration, measure these 200.000, 50.000, and 20.000 mg standards and report their masses to the supervisor. The results are used to verify the integrity of the electrobalance and the standard masses used in daily calibrations.

21.   The electrobalance is available to run controls or for routine determination of mass.

   4.3.2.1 Calibration and Maintenance Procedures Performed by the Lab Manager
The Lab Manager is required to supervise and train technicians, and to maintain delicate equipment. The Lab Manager must also perform quality control procedures including cleaning, repairs, and data trend reviews. The following are performed by the lab manager at the intervals listed, and as necessary.

1.   Weekly cleaning of the sample handling room. The lab manager shall perform or supervise the following cleaning procedures on Friday afternoons. Following the Friday cleaning, no analysis shall occur for at least twenty-four hours to reduce the potential for contamination.
   a)   Verify that all filter samples are in sealed containers to avoid contamination by compounds created or used in the cleaning process.
   b)   Vacuum all surfaces in the sample handling room with a HEPA vacuum cleaner to reduce fugitive dust levels.
   c)   Clean the floor, if necessary, using an absorbent towel and a mild cleaning solution.

d) Clean the work counter with Kimwipes™ and reagent grade alcohol (or another reagent grade solvent, if necessary). This procedure reduces the possibility of contamination should a filter fall to the work surface.

2. Once per week, or if there is visible material in the balance, the lab manager shall perform the following:

a) Carefully remove each balance pan by inserting closed forceps below the wire yoke of the pan bail and lifting the hook from the eye. To protect the hangdown and hangdown loop, <u>do not</u> grasp the bail or hook assembly with the forceps. Gently rest the pans on a fresh clean Kimwipe™.

b) Remove the anti static ionizing strip from the weighing cavity.

c) Clean the inside of the balance cavity using a cotton-tipped applicator wetted with reagent grade ethanol. Extra caution and care is required. Gently brush the exposed surfaces of the cavity, taking care not to disturb the hangdowns extending from the top surface. Gently close the glass slide door and gently brush the outer surface with the ethanol.

d) Clean the top surfaces of the bails (balance pans) with a cotton-tipped applicator wetted with ethanol. The entire surface must be clean and dry. <u>Do not</u> use any solvent other than ethanol on the bails.

e) Gently return the bails to their hangdown loops. Use the bail-lifting procedure previously described. Do not put any stress on the bail or hangdown loop

3. If the 'zero' reading on the balance has changed by 0.01 milligrams or more, the following are the possible causes and cures for the problem.

a) The bail or the tare weight is dirty. Visually inspect each, as a 10 microgram particle shall be obvious. Clean them, if necessary, following the procedures of step 2 above.

b) The bail or the tare weight have been damaged, and have thereby lost mass. Question the previous user of the balance to determine whether either the bail or the tare were dropped, bent, or scarred during the last use period.

c) There may have been an electrical 'brown out' during the period following the last use of the balance. Generally, if the power outage is complete, the balance shall display a blinking 888.888. However, large power fluctuations can cause the balance to behave abnormally. The tare button should be pressed and the calibration proceed normally.

d) The 'hangdowns' in the balance cavity may have been bumped or pulled, affecting the balance mechanism. Question the last persons to use the balance, and then continue the calibration, resetting the zero and calibration values, and verifying that the test weight is within 0.002 micrograms of the expected value. If the test weight is not within the expected range, check the linearity of the balance (see part e below) to determine whether the balance was seriously affected.

e) To run a linearity check on the balance (if the balance is suspected to be damaged), utilize the series of four standard weights stored in the lab managers desk. The four weights, 200 mg, 100 mg, 50 mg, and 20 mg, must be weighed on the suspect balance, and a regression line developed.

i) Use the nylon forceps to remove a weight from its container and place it on the weighing bail in the balance.
ii) Wait until the balance has stabilized (approximately one minute), then record the displayed weight as the 'y' value and the certified mass as the 'x' value.
iii) Remove the weight from the bail, using the plastic forceps, and replace it in its protective container.
iv) Repeat steps i. through iii. for the other three weights.
v) Linearly regress the 'y' values versus the 'x' values. Calculate the r^2 value.
vi) If the r^2 is not better than 0.995, the balance requires maintenance.
f) To check the stability of the balance, reweigh the last 20 archived control filters, and develop a regression line comparing the reweight values to the original values.
i) Reweigh the series of 20 old controls filters on the suspect balance
ii) Plot the reweights versus the original weights.
iii) Derive the best line fit equation correlating the original weights to the reweights.
iv) Calculate the standard deviation and the r^2 of the line fit.
v) If the standard deviation is greater than ±3 micrograms, and the r^2 is not better than .995, the balance should be carefully inspected and submitted for maintenance.

4.3.3 Balance Controls Filter Analysis

The control filters check the performance of the gravimetric analysis systems over the typical month-long period between pre and post weighing of filter samples. Control filters monitor artifact collection by filters in cassettes. They also provide a twice daily check of the correlation between the two electrobalances used in filter processing. (Note: the two balances shall not give the same value because the Cahn 25 uses a substantial tare to maintain the filters within its most sensitive weighing range. A calibration equation, derived from a series of 20 controls filters weighed on each balance, is applied to the Cahn 25 data to yields Cahn 31 equivalent weights. If these do not correlate well with the measured Cahn 31 weights, the lab manager shall create a new correlation equation.)

A fresh control filter is weighed on both balances every day at the calibration times of 8 a.m. and 1 p.m., and then is placed in a cassette and stored in the laboratory for six weeks. A second control filter, one that had been weighed six weeks earlier and stored in a cassette, is removed from the cassette and also weighed on both balances. A basic flow chart of the Controls procedure can be found in Figure 2 below.

Figure 2 Flowchart for Controls Filter Processing

```
                    ┌─────────────────────────────────────┐
                    │ 8:00 a.m Daily Controls Procedures   │
                    └─────────────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Get new filter from stock  │   │ Get oldest archived filter │
        │                            │   │ cassette                   │
        └────────────────────────────┘   └────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Label filter               │   │ Remove filter and label    │
        │                            │   │ from cassette.             │
        └────────────────────────────┘   └────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Weigh filter = Pre weight  │   │ Weigh filter = Post weight │
        └────────────────────────────┘   └────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Place filter in labeled    │   │ Place filter in labeled    │
        │ Petri dish                 │   │ Petri dish                 │
        └────────────────────────────┘   └────────────────────────────┘
                         │                        │
                    ┌─────────────────────────────────────┐
                    │ 1:00 p.m Daily Controls Procedures   │
                    └─────────────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Re-weigh Pre filter =      │   │ Re-weigh Post filter =     │
        │ RePre weight               │   │ RePost weight              │
        └────────────────────────────┘   └────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Mount in cassette          │   │ Replace in Petri dish      │
        └────────────────────────────┘   └────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Label cassette             │   │ Label Petri dish           │
        └────────────────────────────┘   └────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Place cassette in order on │   │ Archive Petri dish for     │
        │ archive shelf              │   │ four weeks                 │
        └────────────────────────────┘   └────────────────────────────┘
                         │                        │
        ┌────────────────────────────┐   ┌────────────────────────────┐
        │ Archive cassette for six   │   │ Replace filter in Teflon   │
        │ weeks                      │   │ filter stock               │
        └────────────────────────────┘   └────────────────────────────┘
```

The control filters facilitate determination of the following:

- Any change in the equivalency of the two balances. The Cahn 31 reconstructed weights (from applying the calibration equation to the Cahn 25 weights) should match the Cahn 31 measured weights within the quoted precision of the balance ($\pm$ 2.5 micrograms). If the weights do not correlate well, the problem is noted and rectified before the measurements of real filters is performed.

- Any change in either electrobalance between morning and afternoon. Controls are run in the afternoon instead of evening since the pre-weighed filters are mailed out at 2:00p.m. daily.   If controls indicate a potential problem with the electrobalance, the filters can be removed from the shipping queue and reweighed once the problem is resolved.

- Any shift in readings between the pre-weights and post-weights for an ambient sample. As pre and post weights are performed six weeks apart, a drift or shift in either balance could lead to erroneous gravimetric measurements. Control filters provide a daily record of balance consistency.

- The uncertainty of the analysis. The difference between the morning weights and the afternoon re-weights provides an estimate of the precision of each electrobalance.

4.3.3.1 Morning Filter Controls (8 a.m.)

1. Select the 'RUN CONTROLS' option on the menu screen of the PRE or POST program. Follow the order dictated by the message box in the upper right side of the screen.

2. Note the indicated Pre and Post control numbers displayed on the screen (e.g. I-nnn).

3. Remove an unused Teflo™ filter from the filter stock and place it on one of the Polonium strip ionization units. To remove a clean filter from stock, gently slip the flat forceps under the outer polyolefin support ring. IMPORTANT: The forceps should contact only the outer support ring of the filter. Do not touch the filter membrane as it punctures and tears easily. Place a slide Petri dish on the work surface next to the filter, labeling the cover with a white sticker carrying the Pre control filter number.

4. Remove the cassette labeled with the Post control filter number from the controls storage shelf. This should have lowest number in the queue. Remove the filter from the cassette, placing it on a Polonium strip ionization unit. When removing exposed filter from cassettes, the forceps should contact only the outer support ring of the filter. Do not touch the filter membrane area. Place a slide Petri dish on the work surface next to the filter, labeling the cover with the white sticker carrying the Post control filter number from the cassette.

5. Place the Pre control filter on the bail (zero area balance pan) of the Cahn 31 electrobalance. Close the door on the balance. When the computer determines that the balance is stabilized, it will automatically record the weight. Select "KEEP AND CONTINUE", or, if you are not satisfied with the mass measurement, select "DISCARD AND RETRY" and wait for the balance to re-stabilize and record a new weight.

6. Once you have selected "KEEP AND CONTINUE", remove the filter, as directed by the message box, and place it in the Petri dish labeled with the Pre identification sticker. The computer will write the data to the CONTROLS database.

7. Place the Post control filter on the bail of the Cahn 31 electrobalance. Close the door of the balance. When the computer determines that the balance is stabilized, it will automatically record the weight. Select "KEEP AND CONTINUE", or, if you are not satisfied with the mass measurement, select "DISCARD AND RETRY" and wait for the balance to re-stabilize and record a new weight.

8.   Once you have selected "KEEP AND CONTINUE", remove the filter, as directed by the message box, and place it in the Petri dish labeled with the Post identification sticker. The computer will write the data to the CONTROLS database.

9.   Note the differences displayed on the screen between the Pre measurements on the Cahn 31 and the Cahn 25, the Post measurements on the Cahn 31 and the Cahn 25, and between the Post weight measurement and the Pre weight measurement for that same filter thirty days earlier.
   a)   If the difference between the balances is less than ±3 micrograms, and the thirty day difference is less than 0.005 milligrams, press "KEEP AND CONTINUE" to end the Controls subroutine of the program.
   b)   If the difference between the balances is larger than that listed in step a, contact the Lab Manager for assistance before pressing "KEEP AND CONTINUE". One balance may be damaged, malfunctioning, or may have simply drifted. In any case, a new balance correlation equation must be derived, following the procedures of section 4.2.2.3.
   c)   If the thirty day difference is larger than that listed in step a, check that the controls filter has not been damaged. Notify the Lab Manager, then press "KEEP AND CONTINUE". Two consecutive days of large thirty day differences is an indication that the balance has changed significantly within the past month, and may require maintenance or repair.

10.   Move the Petri dishes containing the controls filters to the Cahn 25 electrobalance. Start the PRE or POST program on the Cahn 25 computer. Select 'RUN CONTROLS' on the menu screen of the PRE or POST program. Follow the order dictated by the program.

11.   Remove the filters from the Petri dishes, placing each on a Polonium anti-static strip next to the labeled Petri dish cover. Repeat steps 5 through 7 for the Cahn 25 balance, then store the filters in Petri dishes on the shelf next to the controls filter cassette queue.

12.   If a new balance correlation equation is required, the lab manager shall perform the following:
   a)   Reweigh the series of 20 old controls filters on each balance,
   b)   Plot the Cahn 25 weights versus the Cahn 31 weights.
   c)   Derive the best line fit equation correlating the Cahn 25 weights to the Cahn 31 weights.
   d)   Calculate the r2 of the line fit. If it is not better than .995, return step a and repeat the procedure.
   e)   Enter the equation into the PRETARE database.

4.3.3.2   Afternoon Filter Controls (1 p.m.)

1. Move the Petri dishes containing the controls filters to the Cahn 25 electrobalance. Start the PRE or POST program on the Cahn 25 computer. Select 'RUN CONTROLS' on the menu screen of the PRE or POST program. Follow the order dictated by the program.

2. Remove the filters from the Petri dishes, placing each on a Polonium anti-static strip next to the labeled Petri dish cover. When removing exposed filter from the slide Petri dishes, the forceps should contact only the outer support ring of the filter. Do not touch the filter membrane area.

3. Note the indicated Pre and Post control names displayed on the menu screen (e.g. I-nnn). Verify these match the names on the Petri dishes.

4. Place the Pre control filter on the bail (zero area balance pan) of the Cahn 31 electrobalance. Close the door on the balance. When the computer determines that the balance is stabilized, it will automatically record the re-pre weight. Select "KEEP AND CONTINUE" or select "REWEIGH FILTER" if you are not satisfied with the mass measurement. Remove the filter and place it in the labeled Pre Petri dish.

5. Place the Post control filter on the bail of the Cahn 31 electrobalance. Close the door of the balance. When the computer determines that the balance is stabilized, it will automatically record the re-post weight. Select "KEEP AND CONTINUE", or select "REWEIGH FILTER" if you are not satisfied with the mass measurement. Remove the filter and place it in the labeled Post Petri dish.

6. Note the differences displayed on the screen between the Pre and re-Pre, the Post and re-Post weights, the thirty day re-Pre and re-Post measurements, the re-Pre measurements from the Cahn 31 and the Cahn 25, and the re-Post measurements from the Cahn 31 and the Cahn 25. If the differences between balances are less than ±0.002 milligrams, and the re-weight differences are less than or equal to ±0.003 milligrams, press "KEEP AND CONTINUE" to end the Controls subroutine of the program. If the differences are larger, contact the lab manager before pressing "KEEP AND CONTINUE" to exit the controls subroutine.

7. Move the Petri dishes containing the controls filters to the Cahn 25 electrobalance. Start the PRE or POST program on the Cahn 25 computer. Select 'RUN CONTROLS' on the menu screen of the PRE or POST program. Follow the order dictated by the program.

8. Repeat steps 2 through 6 for the Cahn 25 electrobalance

9. Remove the Pre filter from the petri dish and install it in the empty controls cassette. Remove the label carrying the filter name (i.e. "I-nnn") from the Petri dish and place it on the side of the control cassette containing the Pre filter. Place the Pre control cassette on the shelf in the Controls queue.

10. Open the Controls filter archive box on the shelf and remove the oldest control filter in its Petri slide dish (the oldest one has the lowest number in its filter name). Remove and discard the label from the Petri dish. Remove the filter from the Petri slide dish and place it back in the clean filter stock.

11. Place the Post filter, in its Petri slide dish, in the Controls filter archive box.

12. Proceed with normal analysis.

4.4 Pre-Measurement of the Gravimetric Mass and Preparation of Field Log Sheets

The procedures in this section are triggered by running the PRE program. A flow chart for the PRE program can be found in Chart 2. When PRE is started, it checks the LOGS database and creates a list, LOGS QUEUE, of sites requiring boxes of filters. It downloads the site configuration information from the SITE database, then checks the WEIGHTS database to determine the sample date of the last filter weighed for each site. PRE next determines the next four sample dates required at each site. The site name, configuration, and sample date information is placed in a queue and is used to drive the pre-weights program for gravimetric analysis.
The 'blue box' shipping container was specifically designed to transport a two week supply of filter cassettes between UC Davis and the sampling sites. On the front there is a mailer with a reversible prepaid mailing label. The UC Davis address is on one side of the mailer, while the site address is on the other. Each shipping container is assigned to a given site. For each site there are three shipping containers in the system.

The flow diagram for sample handling of clean filters is shown in Figure 3.

Figure 3 Flow Diagram for Sample Handling of Clean Filters

A	B	C	D	D
fine Teflon	fine nylon	fine quartz	PM10 Teflon	impregnated

```
                    ┌──────────┐   ┌──────────┐                   ┌────────────┐
                    │  punch   │   │ prefire *│                   │impregnate *│
                    │ filters  │   │ send to  │                   │  send to   │
                    │          │   │  Davis   │                   │   Davis    │
                    └────┬─────┘   └────┬─────┘                   └─────┬──────┘
   ┌──────────┐          │              │        ┌──────────┐           │
   │premeasure│          │              │        │premeasure│           │
   │   mass   │          │              │        │   mass   │           │
   └────┬─────┘          │              │        └────┬─────┘           │
   ┌────┴──────────────────────────────────────────────────────────────┴───┐
   │        load clean filters into cassettes, attach ID labels            │
   └──────────────────────────────────┬────────────────────────────────────┘
   ┌──────────────────────────────────┴────────────────────────────────────┐
   │       leak-test all cassettes, ship box of cassettes to site          │
   └────────────────────────────────────────────────────────────────────────┘
```
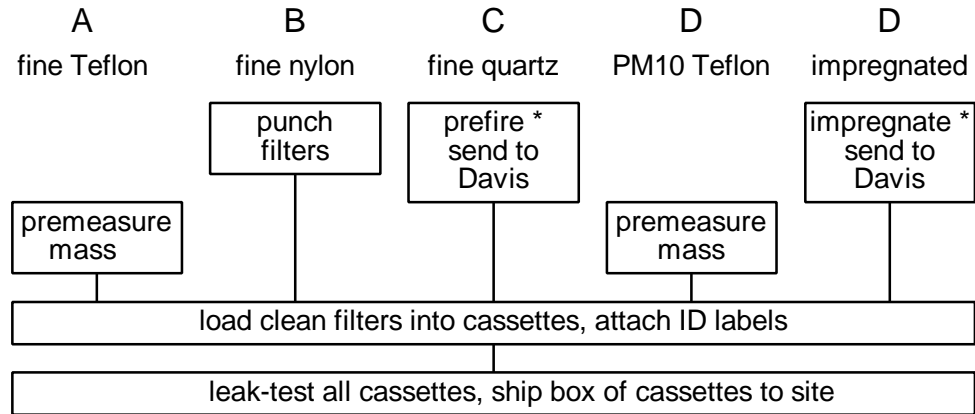
Operations by ions, carbon, and SO_2 contractors are indicated by asterisk.

---

1. Remove four clean Teflon filters from the clean filter stock and place on the de-ionizing units. To remove a clean filter from stock, gently slip the flat forceps under the outer polyolefin support ring. IMPORTANT: The forceps should contact only the outer support ring of the filter. Do not touch the membrane area as it punctures and tears easily.

2. Type PRE at the C:\ prompt on the balance computer to start the PRE program. Follow the order dictated by the program. Enter your initials at the prompt. The program will reach a screen shown below in Figure 4.

Figure 4 Menu Screen at Start of PRE Program

```
┌──────────────────────────────────────────────────────────────────────────────┐
│ <F5> to Skip Box and Remove from Queue   <F1>=calib   <F2>=Controls   <CR> To Continue │
├──────────────────────────────────────────────────────────────────────────────┤
│                          PREWEIGHTS:  CAHN25                                    │
│  ┌──────────────────────────────────────────────────────────────────────────┐ │
│  │ Site Code   ┌────────┐                                                    │ │
│  │             │ CRLA1  │                                                    │ │
│  │             └────────┘                                                    │ │
│  │                                                                           │ │
│  │                                                                           │ │
│  │                                                                           │ │
│  │                                                                           │ │
│  └──────────────────────────────────────────────────────────────────────────┘ │
│                          LAST CALIBRATION                                       │
├──────────────────────────────────────────────────────────────────────────────┤
│ The Last Calibration Was Done    08/08/96  08:45:50        Tare Wght=-28.569    │
└──────────────────────────────────────────────────────────────────────────────┘
```

3. Retrieve the box from the uploading shelf having the same site code as that shown on the menu screen in Figure 4.

    a) If this box doesn't exist, contact the lab manager for assistance. The lab manager shall have the record indicating whether the box was removed because the site was discontinued, or because there were too many filters for the site in the field at the same time.   Do not press the <F5> key to remove the box from the queue unless advised to do so by the lab manager. Once <F5> is pressed, the next box in the queue will appear on the screen. Retrieve this box from the uploading shelf.

4. Press <CR> to initiate the next sample box for the site listed on the screen.   A new screen will be produced, an example of which is shown in Figure 5 below. Instructions will be given by the Pre program in the box in the upper right corner of the screen. The shaded box on the screen will indicate the location in which data is currently being collected.

Figure 5 Pre-Weight Entry Screen

active box   message box

Place a filter on the balance

PREWEIGHTS: CAHN25

SiteCode BLIS1

field blank

SamDat/StrTim		MediaId	A_Chan		Position	MediaId	D_Chan	
09/04/96	00:00	TFEM11689	0.000	0.000	1	TFEM11690	0.000	0.000
09/07/96	00:00	TFEM11691	0.000	0.000	2	TFEM11692	0.000	0.000
09/04/96	00:00	TFEM11691	0.000	0.000	1			
09/11/96	00:00	TFEM11693	0.000	0.000	3	TFEM11694	0.000	0.000
09/14/96	00:00	TFEM11695	0.000	0.000	4	TFEM11696	0.000	0.000

1. Open the bottom drawer of the file cabinet in the sample handling room and locate the folder for the site shown on the screen. The folder contains pre-printed labels for the filter cassettes. If there are stickers showing earlier dates than those requested by the computer, contact the lab manager for instructions.

2. Label each cassette in the 'Week 1' bag for the site with the appropriate pre-printed label. Place the label on the threaded top cap of the cassette, centering it between the exposed ridges. Each label contains the site name, sample date, and the module and channel code. The cassettes are color coded to indicate module and channel, as shown in Figure 6 below, but must be labeled for site and sample

date. Note also that for double stage cassettes, there is a label for each filter in the cassette. The primary filter has the suffix "P," while the secondary filter has the suffix "S." If there appear to be too many or too few labels for the number of cassettes in the box, call the lab manager for instruction.

Figure 6 Cassette Color Coding

Cassette color code	Module and channel code
red/black	A1 = Module A, channel 1 (Wednesday sample)
red/white	A2 = Module A, channel 2 (Saturday sample)
yellow/black	B1 = Module B, channel 1 (Wednesday sample)
yellow/white	B2 = Module B, channel 2 (Saturday sample)
green/black	C1 = Module C, channel 1 (Saturday sample)
green/white	C2 = Module C, channel 2 (Saturday sample)
blue/black	D1 = Module D, channel 1 (Saturday sample)
blue/white	D2 = Module D, channel 2 (Saturday sample)

3.   Place the first filter gently on the bail or pan inside the electrobalance. Avoid causing the bail to swing or bounce. Once the filter is placed, close the door on the balance. A message will appear in the message box on the computer screen "*Waiting for balance to stabilize. Press <spacebar> to stop*".

4.   When the balance has stabilized, the computer will automatically record the weight. The message box will display the following message: "*Press <CR> to record weight or <TAB> to fields you want to change*".
   a)   If <CR> is pressed, the weight in the shaded box will be stored in temporary memory, but will not be recorded permanently into the WEIGHTS database until all the filters for the box have been weighed. If the program crashes, or the power goes out, the weights on the screen will not have been saved. The program will move the active box to the next input position once <CR> is pressed.
   b)   If <TAB> is pressed, the active box will move to the next input field. Using <TAB> to increment through the screen, data such as mask size, may be changed by keyboard input.   Press <TAB> until the cursor is in the desired location, then enter the corrected information. <TAB> allows movement only within the screen. When the active box reaches the last input field on the screen, it will jump back to the first input field.

5.   Once the weight has been recorded, remove the protective red cap from the appropriate cassette from the 'Week 1' bag. Unscrew the threaded cassette top cap and remove the lock ring. Carefully check the cassette and red cap, cleaning them with Kimwipes™ or a clean brush if necessary.
   a)   Check the threaded cassette top gasket; if it is worn or dirty, replace or clean it.

b) For module A filter cassettes, check the condition, type, and the cleanliness of the collection mask. If the mask is the incorrect size, dirty, wrinkled, misshapen, or worn, replace it with a new mask. (If no mask is used, the media identification number will contain M0. If a 2.2 cm^2 area mask is used, the media identification number will contain M1.).

c) Check the drain disk, for module A, C, and D filter cassettes; module B cassettes do not require drain disks. The drain disk should be flat, smooth, and clean. Replace it if necessary.

d) Remove the O-ring (ethylene propylene for module A,B, and D cassettes, Viton™ for module C cassettes) and the filter grate, checking them for damage. If the o-ring is deformed, worn, or damaged, replace it. If necessary, clean the filter grate by gently brushing with the brush reserved for non-quartz cassettes. Be careful to seat the filter grate with the finer grids oriented up and the large bar grids down.

e) Center the clean drain disk on the filter grate. For a module A cassette, center the collection mask over the drain disk.

f) Check the lock ring for cleanliness and to verify it has no nicks or slices that could allow leaks. Replace it if necessary.

6. Install the weighed filter in the cassette.
   a) Center the filter in the cassette with the flat side of the filter facing up. The rough and extended edges of the filter should mate with the o-ring, except in the A module where they will mate with the mask. This orientation will present an open, smooth, and flat surface to the incoming air flow when sampling occurs.
   b) Gently place the lock ring on the cassette over the filter, taking care to keep the filter centered and to avoid damage the filter support ring.
   c) Place the threaded cap and gasket over the lock ring and tighten it. The lock ring will tend to shift position and must be watched carefully to avoid damage to the filter. Keep all elements centered thread the cassette finger tight.
   d) Reinstall the protective red cap over the cassette, and place the cassette in the 'Week 1' bag.

7. Repeat steps 7 through 10 for the other Teflon filters to be sent in the box. Simultaneously, or after the Teflon filters have been weighed, the ions, carbon, and SO2 filters must be installed in cassettes. The procedure is described below.
   a) The B module cassettes, color coded yellow/black and yellow/white, must be loaded with Nylasorb™ filters for ion analysis.
      i) Check the cleanliness of the first cassette (yellow/black) following the procedures for Module A cassettes from step 9.
      ii) Obtain a clean nylon filter from the supply stock, stored in the refrigerator, and load it into the cassette directly onto the diffusion and support grid, similar to the process described in step 10.

        iii)   If a new lot of filters is started, record the new lot number, and the site code and sample date for the first cassette in which it is used. Give this information to the lab manager for entry into a database.

        iv)   Put the cassette in the resealable 'Week 1'bag.

        v)   Repeat for the second module B cassette (yellow/white).

b)   The C module cassettes, color coded green/black and green/white, must be loaded with quartz filters for Thermal Optical Reflectance analysis for organic and inorganic carbon. Most Module C cassettes contain one stage, although a few specified sites have cassettes with two filter stages, each loaded with a quartz filter. The first or primary filter collects all the particles and absorbs organic cases, while the secondary filter only absorbs gases.

        i)   Check the cleanliness of the first cassette (green/black) following the procedures for Module A cassettes from step 9. If this site has double cassettes, disassemble both sections and check the cleanliness. Assure the <u>two</u> flat silicone rubber gaskets are used in the cassette cap of the first section. Two gaskets are needed to provide spacing so that the hold down retainer of the cyclone manifold will fit.

        ii)   Load a clean quartz filter from the supply into each section of the cassette, following the procedures in step 10 for module A filters.

        iii)   If a new lot of filters is started, record the new lot number, and the site code and sample date for the first cassette in which it is used. Give this information to the lab manager for entry into a database.

        iv)   Put the cassette in the resealable bag.

        v)   Repeat for the second (green/white) quartz cassette.

c)   If the computer indicates that a dynamic field blank (FB) cassette is to be included in the bag, repeat the procedures above.

d)   Some D module cassette have a second stage, meant for collecting SO2 gas. These cassettes are color coded blue/black and blue/white. The second stage will undergo ion chromatography analysis.

        i)   Check the cleanliness of the bottom section of the first cassette, following the procedures from step 9.

        ii)   Insert the clean impregnated filter in the bottom section with the drain disc centered underneath, as described in section 10. Clean the forceps before handling other filters.

        iii)   If a new lot of filters is started, record the new lot number, and the site code and sample date for the first cassette in which it is used. Give this information to the lab manager for entry into a database.

        iv)   Put the cassette in the appropriate re-sealable bag.

        v)   Repeat for the second cassette. Cover the container of impregnated filters and keep it inside the designated box.

e)   If the computer indicates that a dynamic field blank (FB) cassette is to be sent, prepare and install the filter into the field blank cassette following the same procedures used to install filters into normal cassettes.

8. Once all the input fields on the screen contain weights, and the ions and carbon analysis filters are loaded into cassettes, the box is complete. The message box will read "<CR> to Record Weights or <TAB> to fields you want to change". Once <CR> is pressed, the following events will occur.

    a) PRE will create a log sheet and send it to the printer queue. This will include the dates for the sample change and sampling, the sample identification numbers for all cassettes, the filter identification numbers for Teflon, and the lot numbers for the quartz and impregnated, the pre-weights for the Teflon filter, along with the name of the technician and date weighed. The log sheet should be folded and placed in the 'Week 1' bag with the associated cassettes.

    b) The message box will read "Writing to Weights File" as the PRE program saves the filter weights and identification to the WEIGHTS database, and saves the identification and site configuration information to the LOGS database.

    c) The screen for the 'Week 2' bag of filters for the site will automatically appear once the data for the 'Week 1' bag is saved. To complete a weighing session, both weeks of filters for a site must be weighed and installed into cassettes.

9. Repeat steps 5 through 11 for the 'Week 2' bag of filters for the site. Once step 11 is completed, the menu screen for the PRE program will reappear (as seen in Figure 4). To process the next box, begin again at step 1.

4.5 Leak Checking of Cassettes and Preparation for Shipping to the Field

At this point, all the cassettes have been loaded with clean filters and have been labeled with the site, sample date, and module. Each shipping box contains two weeks of cassettes, sorted in two single week sealable bags with their log sheets. The last step prior to sending the cassettes into the field for sampling is to check that the right number and type of filters are being sent, and that the cassettes do not have any leaks that could affect the flow rate measurement. The leak checking and cassette verification procedures are described below.

1. Start the LEAKCHECK program at the DOS prompt. The program will display, in the order preweighed, the type and configuration of filters and cassettes expected in each shipping box. It will also display the site and sample date for each filter, and the allowable leak under full vacuum for each type of cassette.

2. Check each cassette against the list, verifying filter type, site and sample date.

3. Mount each cassette on the leak test stand using the quick connect fastener. Remove the protective red cap.

4. Check each cassette for excessive leaks.

    a) Place the leak-cap gently over the open cassette face. Do not touch the filter.

    b) Open the vacuum valve and observe the flow gauge.

i)   If the leak rate under full vacuum (28" Hg) is less than the values indicated below in Figure 7, the cassette is considered acceptable.

ii)   If, for any cassette, the leak rate exceeds this value, find and reduce the leak by tightening the threaded fittings, re-centering components, or replacing O-rings or gaskets.

Figure 7 Acceptable Leak Check Parameters

configuration	flow
A, B, or D	1 L/min
C	3 L/min
C+C	4 L/min
D+S or A+S	2 L/min

5.   Remove the leak-cap from the cassette, and install it on the next cassette on the leak check device. Repeat step 4 for each cassette, except the field blank which is not checked.

6.   For the B module cassettes only (yellow/black, yellow/white), check the flow through the filter. Without the leak-cap, open the vacuum valve and observe the flow gauge.

     a)   If the flow rate is off the scale (more than 5 liters per minute), the cassettes is fine. Close the vacuum valve.

     b)   If the flow rate is at or near 0 liters per minute, the cassette contains a filter spacer as well as a filter. Close the vacuum valve. Open the cassette, remove the spacer (a round, yellow sheet of paper), close the cassette, and try the flow check again.

7.   When all the cassettes listed on the LEAKCHECK screen have been checked and tested, seal them in their single week bags, install them in their blue mailing boxes, and place the shipping boxes in the outbound mail tub. Then, press enter to verify the box was leak tested, and to increment to the next box in the queue for testing.

8.   Repeat steps 1 through 7 until all boxes have been prepared for mailing, and the leak testing queue is empty.
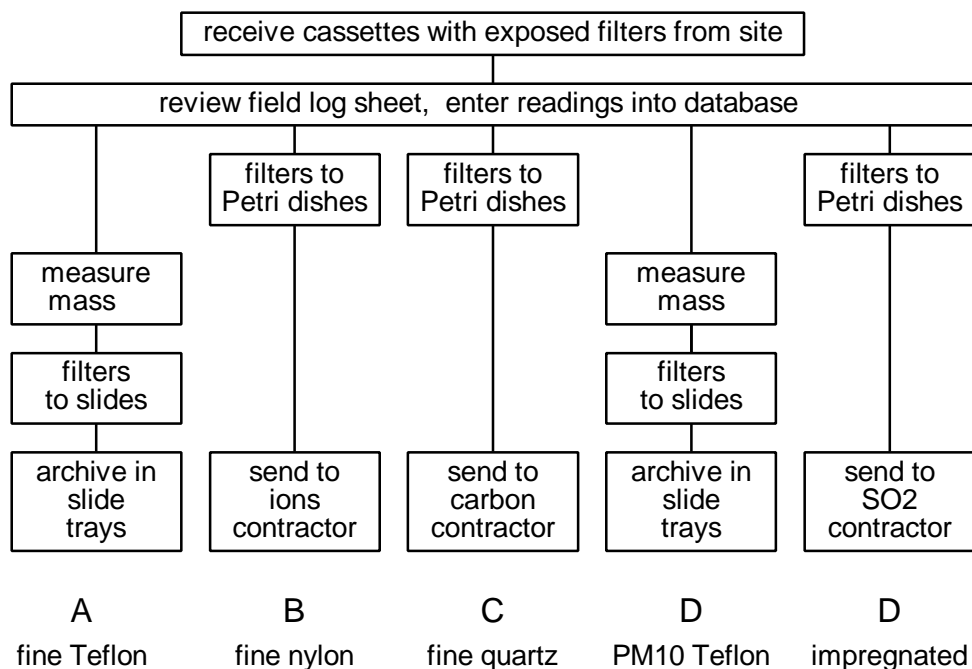
4.6 Receipt of Boxes and Entry of Log Sheet Information

The receipt of boxes containing exposed filter cassettes initiates the second phase of the sample handling procedure. The first phase involved preparing clean filters and cassettes, and mailing them to each site for sampling. The second phase, sections 4.6, 4.7 and 4.8 will involve processing the exposed filters in preparation for ionic, carbon, or elemental analysis.

Normally each shipping container holds cassettes for two weeks, with the cassettes and Field Log Sheets for each week in a re-sealable bag.   When both weeks of cassettes have been used, the box will be closed and mailed back to the Air Quality Group using the

reversible first class mail label on the front of the box. Once the box arrives at the Air Quality Group, it is processed following the procedures described in Figure 8.

Figure 8 Flow Chart of Sample Handling of Exposed Filters

```
                    ┌───────────────────────────────────────────┐
                    │  receive cassettes with exposed filters from site  │
                    └───────────────────────────────────────────┘
        ┌──────────────────────────────────────────────────────────────────────┐
        │        review field log sheet,  enter readings into database          │
        └──────────────────────────────────────────────────────────────────────┘
            │         ┌──────────┐   ┌──────────┐         │         ┌──────────┐
            │         │filters to│   │filters to│         │         │filters to│
            │         │Petri     │   │Petri     │         │         │Petri     │
            │         │dishes    │   │dishes    │         │         │dishes    │
            │         └──────────┘   └──────────┘         │         └──────────┘
       ┌─────────┐                                   ┌─────────┐
       │ measure │                                   │ measure │
       │  mass   │                                   │  mass   │
       └─────────┘                                   └─────────┘
       ┌─────────┐                                   ┌─────────┐
       │ filters │                                   │ filters │
       │to slides│                                   │to slides│
       └─────────┘                                   └─────────┘
       ┌─────────┐  ┌─────────┐  ┌─────────┐  ┌─────────┐  ┌─────────┐
       │archive  │  │send to  │  │send to  │  │archive  │  │send to  │
       │in slide │  │ions     │  │carbon   │  │in slide │  │SO2      │
       │trays    │  │contractor│ │contractor│ │trays    │  │contractor│
       └─────────┘  └─────────┘  └─────────┘  └─────────┘  └─────────┘

           A            B            C            D            D
       fine Teflon  fine nylon   fine quartz  PM10 Teflon  impregnated
```

4.6.1 Receipt of Shipping Container

1.   Immediately upon receipt of the shipping container of exposed cassettes from the site, update the IMPROVE Dot Chart by placing a blue dot in the site/week box, on top of the yellow coloring. This is a visual reminder of how many boxes are currently in the field for each site.

2.   Arrange the boxes in alphabetical order on the countertop.

3.   Open each box and remove the log sheets, checking that the dates on the log sheets match the dates on the filter cassettes. Keep the log sheets in alphabetical and date order, and place them in the logs entry box. If the log sheet and/or filters for either week is missing, do not process the filters. Return the log sheet(s) to the shipping box with the filters, post a note containing a description of the problem on the front, notify the lab manager, and place the shipping box on the problem samples shelf in the weight room.

4. Clean the outside of the shipping container. Verify that a 1-1/2" X 3" white tag is mounted on the right side of the box to allow postal authorities to affix required postage.

5. Reverse the prepaid mailer label.
6. Queue the boxes on the shelf to the left of the ions filter downloading area. They should remain in the same order as the log sheets, arranged from right to left.

4.6.2 Reviewing the Field Log Sheets

The Field Log Sheets are reviewed and entered into the database twice; first by a technician, and second by the lab manager. The initial review and entry is conducted prior to the removal of the filters. The second review and entry, by the lab manager, is conducted following the downloading of the ions filters and inspection of the A and D channel filters.

If correction or explanation of information on the log sheet is required, the lab manager shall be consulted. Corrections shall be made by drawing a single line through the existing information using a fine red pen, and entering the correct information. Comments should also be made using the fine red pen. All comments or corrections on the log sheet shall be entered into the LOGS database. To review the field log sheets, use the following procedure.

1. Before entering data, check the dates the filters were installed and removed from the sampler. These are filled in by the site operator on the log sheet. The sample dates on the filter identification tags and the log sheet should fall within the time period between installation and removal. If not, the cassettes were installed incorrectly, requiring the following actions.
   a) Notify the lab manager so that he/she may contact the site operator and determine the cause of the problem
   b) Do not enter data for the 'problem' log sheet into the LOGS database. If LOGSIN is running and is currently on the site and week for the 'problem' log sheet, use the <PageUp> key to move to the previous log sheet entered for the site. Press <F3> to exit from the log sheet.
   c) Remove both log sheets for the 'problem' site from the logs entry box, place them in the shipping box with their associated filters, note the problem on the front of the box, and place the box on the problem filters shelf
   d) Enter the site name for the next log sheet in the queue and continue.

2. Check for omissions. If data were not entered, inform the laboratory manager and call the field operator. (If the correct data cannot be absolutely determined, but the filter appears normal, the sample shall be labeled with the code 'QD' for questionable data. It shall be processed normally, but shall be subject to more

intense scrutiny during the quality assurance procedure. If the filter does not appear normal, the sample shall receive the status code 'XX' for an unusable sample, and shall not be processed further.)

3. Review the comments by the field operator and take action accordingly. If an equipment or hardware malfunction is reported, inform the field specialist and, if time allows, take action to correct the problem.

4. Verify that all closed-solenoid gauge readings (system vacuum without sampling air flow) are greater than 16 inches mercury. If any reading is lower, inform the field specialist as a replacement pump or leak check set must be sent to the site. Also, depending on the severity of the problem, the filters may be invalidated for the module with the low vacuum gauge reading. See step 7 for more information.

5. Verify that magnehelic values fall within the range 0.20" H_2O to 0.60" H_2O. Final readings below this range may indicate the filter has clogged and is therefore suspect. See step 8 for more information.

6. Verify that the sampling durations are approximately 24.00 hours.
    a) For sites running with small generators known to have variable frequency (CANY), if the times range from 23.6 to 24.4 hours, correct the times to 24.00 hours. (The controllers use quartz clocks and are not affected by the line frequency.) This variation reflects a frequency variation of ± 1 hertz.
    b) Filters with elapsed times less than 18 hours are considered invalid. If the elapsed time is less than 18 hours, the samples are not valid. See step 8 to determine the problem code to enter into the computer.
    c) Filters with elapsed times greater than 24 hours may have sampled on successive weeks. This is rare as lock out devices have been installed at most sites to prevent subsequent week sampling on the same cassettes. If the elapsed time is greater than 24 hours, the samples are not valid. See step 7 to determine the problem code to enter into the computer.

7. If the samples are not valid, they are generally removed from the normal system at this point and put in the Problem Filter Archive.
    a) Record a short description of the problem, if the field operator has not done so, and indicate the problem code to be used in the comments section of the field log sheet. A short explanation, such as "Cassette loose and moved from sample port" or "Magnehelic tubing to cyclone detached " is adequate.
    b) Add the appropriate problem code, listed below, to the Field Log Sheet at the end of each invalid sample record.
    c) Fill out a problem code sticker with the site and sample week date, circling the appropriate problem code. Post this tag on the bag containing the invalid samples to indicate their status to the technicians downloading the filters.

d)   If there are questions concerning the validity of the samples, check with the laboratory supervisor. If necessary, call the field operator.

e)   The five codes indicating invalid samples, and the accompanying reasons for invalidating a sample and archiving it are:

   i)   NS: not serviced. The operator did not change the sample during the allotted 3-day period, and the site did not have a lock-out device installed. The sample will have an elapsed time over 24 hours and shall receive the NS code. As a result of the time overrun, the next week's sample will have an elapsed time under 24 hours. If the next week's sample has an elapsed time greater than 18 hours (and less than 24 hours), it will be valid. If its elapsed time is less than 18 hours, it shall receive the NS code and will be invalid.

   ii)   PO: power outage. Power interruptions caused the sample to run less than 18 hours.

   iii)   SE: site error. The filters were incorrectly installed in the sampler by the operator, resulting in an unusable sample.

   iv)   EP: equipment problem. Some portion of the sampler malfunctioned, resulting in an invalid sample due to flow rate, elapsed time, pump vacuum, etc.

   v)   XX   invalid for other reasons. The filter has a hole, the filter has obvious problems (not centered properly, grill upside down), the filter was dropped or contaminated by water, the cassette is broken and suspected to have been leaking during sampling, or no data were recorded by the operator and the filter does not appear normal.

8.   In some cases, when the filter clogs, when data is not collected by the site operator, or when the filter is not installed in the sampler, the invalid samples are given a problem code, but are not removed from the normal processing system. The samples shall be analyzed, but the data shall be subject to intense scrutiny during the quality assurance procedures. If the data correlate well with the other samples, they shall be allowed into the final data set. If the data do not correlate well, or there are no other filters with which to run a correlation, the data shall be discarded during the data validation procedures. To begin this process;

a)   Record a short description of the problem, if the field operator has not done so, and indicate the problem code to be used in the comments section of the field log sheet.

b)   Add the appropriate problem code, listed below, to the Field Log Sheet at the end of each invalid sample record.

c)   Fill out a problem code tag with the site and sample week date, circling the appropriate problem code. Post this tag on the bag containing the invalid samples to indicate their status to the technicians downloading the filters.

d)   If there are questions concerning the validity of the samples, check with the laboratory supervisor. If necessary, call the field operator.

e) The two codes indicating suspect samples that may not be invalid are:
    i) <u>CG</u>: clogged filter. The final magnehelic reading is less than 1/2 of the initial reading, resulting in unreliable flow rate measurements. (This sample is kept to document the factors involved in clogging.)
    ii) <u>QD</u>: questionable data. The data are suspect due to lack of initial or final readings, unusual looking deposits, strange elapsed times, or unusual pump vacuum readings.
    iii) <u>FB</u>: field blank. Designated field blank, or the filter was returned without having been installed in the sampler.

4.6.3 Entry of Field Log Sheet Data into Database

To enter the log sheets, once they have been reviewed, start the LOGSIN program to access the LOGS database.

1. Follow the directions on the menu screen, entering your initials, then the site name for the first log sheet. A screen identical to the log sheet will appear, with blank spaces to enter the data collected by the site operator.
2. Enter, one field at a time, the data collected by the site operator. If an error is made, you may use backspace to return to the incorrect field and overwrite it, or you may continue to the end of the sheet, then press tab to increment through the fields in the sheet.
3. Enter all comments from the sheet by pressing the key <F12> then typing the comments. The first line refers to the Wednesday sample. The second line refers to the Saturday sample. Press the <Enter> key to switch from the Wednesday to the Saturday comments section, or to return to the log sheet screen.
4. To save the data on a sheet, press the key <F3>.
5. To see the previous sheet, press the key <PageUp>
6. Once the data are saved, the values shall be recorded to the LOGS database, and the samples will appear in the ions filter downloading queue, and in the post weighing queue. The order of the downloading follows the order that the field data was entered.

After the data have been entered by a lab technician, the filters for ions, carbon, or SO_2 analysis are downloaded into Petri dishes for shipment to the analysis contractors. At the same time, the A and D channel filters (for elemental and PM_{10} mass analysis) are checked, while remaining in their cassettes, for visible damage or unusual deposits. Any unusual condition is recorded on the log sheets and, if necessary, a problem code tag is filled out and attached to the bag containing the suspect filter. Following the downloading of the ions filters and checking of the Teflon filters, the log sheets are re-entered by the lab manager to ensure no entry errors have been made, and that no conditions that could result in invalid samples were missed by the lab technicians. To perform the second entry of the log sheets, the lab manager follows the procedure listed below.

1. Start DOUBLELOGS to access the LOGS database. The previously entered log sheets will come up on the screen, in the order entered.
2. Re-enter each log sheet, following the procedures listed for the initial entry.

3. Press the key <F12> to view the comments entered. Correct or add comments at this time.
4. Press the key <F3> when done entering the sheet. The computer will compare the newly entered sheet with the original and will highlight any differences.
5. Press the key <F5> to move to and accept or correct any differences.
6. Press the key <F3> once the changes are made to save the sheet and advance to the next sheet in the queue.

4.7 Downloading of Exposed Filters for Ion or Carbon Analysis

This step can begin only after the field log sheet data has been entered into the LOGS database. The order of the unloading, or downloading, follows the order that the field log sheet data was entered. The procedures for downloading are as follows:

1. Start the CONTRACTOR code on the downloading terminal.
2. Check the numbered petri dish trays for each contractor against the display on the downloading terminal. The filter identification, on tags on the outside of each petri dish, and position in the trays should match those in the CONTRACTOR code. If not, contact the lab manager for assistance.
   a) If the mismatch is within the current box, determine the extra or missing filter and remove or return it to the petri tray. Missing filters occur when a filter is mistakenly put in the problem filter file. Extra filters occur when invalid samples are not recognized and are downloaded as normal.
   b) If the mismatch is not within the current or previous box, the LOGS database may have been damaged. Contact the database manager to recover the missing information, or a backup copy of the LOGS database.
3. Open the box containing the site and sample dates indicated on the downloading terminal as being next in the queue. The re-sealable plastic bag shall be opened to allow the filters to come to equilibrium.
4. Unload the nylon filters from the module B cassettes into clean petri dishes.
   a) Remove the protective red cap from the first cassette (yellow/black) in the first bag and verify its cleanliness. If required, wipe the inside of the cap with a Kimwipe dampened with ethanol.
   b) Remove the cassette top and the lock ring.
   c) Transfer the nylon filter to a Petri dish and transfer the accompanying Sample Identification Tag from the cassette to the Petri dish.
   d) Sort the petri dish into the numbered petri tray in the order required by the CONTRACTOR code queue. When the numbered tray is full, carefully transfer the Petri dishes, in order, into a white Petri shipping tray. Label the tray with the filter type ("B"), and with the site and sample date of the first and last filters in the box.   Verify that the filters in the tray are in the order dictated by the CONTRACTOR queue. Place the Petri tray in the refrigerator for storage.
   e) If a filter was not loaded into the cassette, do not simply include an empty petri dish in the queue. Instead, make a comment on the log sheet and in the LOGS database, and change the status of the filter to XX by editing the log sheet through the LOGSIN program. Once the status is changed, press

the <F11> key in the CONTRACTOR program to recreate the queue, and the filter will no longer appear.

f) Check the condition and the cleanliness of the EP O-ring. Discard the O-ring if it is deformed. Clean the filter grate and O-ring by gently brushing with the camel-hair brush reserved for non-quartz cassettes.

g) Reassemble the cassette, and replace it in the re-sealable bag in the shipping box.

h) Repeat for the second cassette (yellow/white) and any field blank in the bag.

5. Unload the quartz filters from the module C cassettes into clean petri dishes. The Module C cassettes contain either one or two stages, each with a quartz filter. The first or primary filter collects all the particles and adsorbs organic gases, while the secondary filter only adsorbs gases

a) Remove the protective red cap from the first cassette (green/black) in the first bag and verify its cleanliness. If required, wipe the inside of the cap with a Kimwipe dampened with ethanol.

b) Remove the cassette top and the lock ring.

c) Transfer the fine quartz filter from the first cassette (green/black) to a Petri dish and transfer the accompanying Sample Identification Tag from the cassette to the Petri dish. If the cassette has two stages, and therefore has a secondary filter, transfer the secondary filter and its label to another Petri dish. Verify that the P, for Primary, and S, for secondary, designations are clearly marked on the filter identification labels.

d) Sort the petri dishes into the numbered petri trays in the order required by the CONTRACTOR code queue. When the numbered tray is full, carefully transfer the Petri dishes, in order, into a white Petri shipping tray. Label the tray with the filter type ("C"), and with the site and sample date of the first and last filters in the box. Verify that the filters in the tray are in the order dictated by the CONTRACTOR queue. Place the Petri tray in the freezer for storage.

e) If a filter was not loaded into the cassette for either the primary or the secondary stage, do not simply include an empty petri dish in the queue. Instead, make a comment on the log sheet and in the LOGS database, and change the status of the filter to XX by editing the log sheet through the LOGSIN program. Once the status is changed, press the <F11> key in the CONTRACTOR program to recreate the queue, and the filter will no longer appear.

f) Disassemble and clean the cassette. Gently brush away quartz filter debris with the brush reserved for quartz cassettes. Make sure no debris is retained under the lip of the anti-twist lock ring. Brush the debris into a waste receptacle. Be careful not to raise or breathe any filter debris dust during this operation. The quartz debris is highly electrostatic and can contaminate other samples. Discard and replace the drain disk if it is dirty and the O-ring if it is deformed.

g) For a double cassette, make certain that <u>two</u> flat silicone rubber gaskets or a single EP O-ring are used in the cassette cap of the first section to ensure adequate spacing for mounting the cassette on the cyclone in the sampling module. Disassemble both sections of the cassette and clean as described in part f.

h) Reassemble the cassette, and replace it in the re-sealable bag in the shipping box.

i) Repeat for the second cassette (green/white) and any field blank in the bag.

6. For sites having SO_2 filters as a second stage behind a Teflon filter, download only the SO_2 filter into a clean petri dish.

a) Remove the protective red cap from the first cassette (red/black or blue/black) in the first bag and verify its cleanliness. If required, wipe the inside of the cap with a Kimwipe dampened with ethanol.

b) Check the status of the teflon filter in the primary stage of the cassette. Do not open this stage, merely inspect the filter visually with the cassette still intact.

   i) If the filter is missing or the deposit area is visibly damaged, annotate the field log sheet, and change the status code to XX. Add the comment on the log sheet to the LOGS database, and change the status of both the Teflon filter and the SO_2 filter to XX by editing the log sheet through the LOGSIN program.

   ii) If the deposit appears non-uniform, contact the lab manager, and make a comment on the log sheet. The lab manager shall decide whether to declare the Teflon filter status XX, or to declare it QD. Add the comment on the log sheet to the LOGS database, and change the status of the filter by editing the log sheet through the LOGSIN program.

c) Leaving the primary stage intact, unscrew the secondary stage adapter and remove the lock ring.

d) Transfer each impregnated SO_2 filter to a Petri dish and attach the accompanying filter identification label to each Petri dish.

e) Sort the petri dishes into the numbered petri trays in the order required by the CONTRACTOR code queue. When the numbered tray is full, carefully transfer the Petri dishes, in order, into a white Petri shipping tray. Label the tray with the filter type ("S"), and with the site and sample date of the first and last filters in the box.   Verify that the filters in the tray are in the order dictated by the CONTRACTOR queue. Place the Petri tray in the refrigerator for storage.

f) If an SO_2 filter was not loaded into the cassette for the secondary stage, do not simply include an empty petri dish in the queue. Instead, make a comment on the log sheet and in the LOGS database, and change the status of the filter to XX by editing the log sheet through the LOGSIN program. Once the status is changed, press the <F11> key in the CONTRACTOR program to recreate the queue, and the filter will no longer appear.

g) Disassemble and clean the cassette. Gently brush away any filter debris with the brush reserved for SO_2 filter cassettes. Make sure no debris is retained under the lip of the anti-twist lock ring. Brush the debris into a waste receptacle. Discard and replace the drain disk if it is dirty and the O-ring if it is deformed. Clean the support grid with water, if it is dirty, rinsing it with reagent grade alcohol as a final step prior to replacing it in the cassette.

h) Reassemble the cassette, and replace it in the re-sealable bag in the shipping box.

i) Repeat for the second cassette (red/white or blue/white) and any field blank in the bag.

7. Repeat the steps 2 through 6 for the second week's bag in the shipping box.

8. Find the site corresponding to the box just downloaded on the dot chart. Determine the next two week dates, for that site, for which no box has yet been initiated. Make a new box label from a sticky 1"x3" label, on which the site code and sample week dates have been written. Place the label on the top, left side of the shipping container. Annotate the Dot Chart with a small line tic in the upper right hand portion of squares representing the two sample weeks, newly initiated for the site.

9. Use the pre printed date labels in the ions downloading area to label the week 1 bag with the first new week date, and the week 2 bag with the second new week date. Do not yet remove the old week dates, or the problem tags.

10. Place the shipping box on the shelf above the downloading area to the left of the Cahn 31 electrobalance. This is the location of the post weighing queue. The boxes should be kept in the order in which the log sheets were entered (alphabetical and by date) and arranged from right to left.

11. Check the number of trays in the refrigerator and freezer for each type of filter. Four full trays of any single type of filter comprise an analysis lot. Once four full trays of a substrate exist, they must be checked, labeled with a lot identification number, boxed, and sent to the contractor for analysis.

a) The filters should not be mailed on a Friday as they are shipped overnight and cannot be received on Saturdays. If it Friday, or late Thursday afternoon, do not continue this process until Monday.

b) Remove all four trays from the refrigerator or freezer.

c) Start the CONTRACTOR code on the downloading computer. Select the appropriate database to view:
i) press the key <F1> to access the 'B', or ions, filter database
ii) press the key <F2> to access the 'C', or carbon, filter database
iii) press the key <F3> to access the 'S', or SO_2, filter database

d) Verify the identification and position of each filter in each of the four trays. Start with the oldest tray. Use the <PageUp> key to move to the previous tray in the database. Use the <PageDown> key to move to the next tray in the database.

e) Once all the labels and positions have been verified in the first tray, select the "print sheet" option by simultaneously pressing the keys <Ctrl> and

<P> in the CONTRACTOR program.   This will create a database file containing only the contents of the tray that has just been checked. The file will be located in the directory U:\IMP\LOGS under the sub-directory IONS, CARBON, or SO2. The file name will be the site name and sample date of the first filter in the tray.

f)   Repeat steps c, d, and e for all four trays.

g)   View the lots of filters currently being analyzed by the contractor by pressing the <F12> key in the CONTRACTOR program (press the <Esc> key to exit).

h)   Quit the CONTRACTOR program by pressing the key <F4>.

i)   Open each of the database files created in step e and print them.

j)   Open a new e-mail letter, using the e-mail address of the primary contact at the contractor's laboratory.

k)   Copy the appropriate form letter for analysis from the directory V:\LAB\LETTERS, and paste the letter into the e-mail document.

l)   Check the archive file to determine the last filter lot sent to this contractor. Increment the lot number by one. This will be the current lot number.

m)   Add the current lot number and current date to the e-mail letter in the spaces provided.

n)   Count the number of IMPROVE network samples. Include this information in the space provided in the form letter.

o)   Count the number of non-IMPROVE network samples (Tahoe basin sites), and include this number in the space provided in the form letter.

p)   Print a copy of the e-mail letter.

q)   Attach the four database files created in step e to the e-mail document, then send the document.

r)   Collect the printed documents; the e-mail letter and the four database files, staple them together, and file them in the appropriate 'Archive of Filters Sent for External Analysis' file.

s)   For ions analysis of 'B' module or 'S' filters, pack the four checked petri trays in a box, attach a shipping label to send them FedEx™ overnight to the address listed on the form letter, and call Federal Express to request a pick-up.

t)   For carbon analysis of 'C' module filters, pack the four checked petri trays in a box with four frozen packets of blue ice. Attach a shipping label to send them FedEx™ overnight to the address listed on the form letter, and call Federal Express to request a pick-up.

4.7 Post-Measurement of Gravimetric Mass

The Teflon filters from Modules A and D are generally unloaded and post weighed at the Cahn 31 balance. The post weighing order is driven by a queue reflecting the order in which the logs were entered. Post weighing shall not occur until the balance is calibrated and controls have been run.

The procedures in this section are triggered by running the POST program. A flow chart for the POST program can be found in Chart 3. When POST is started, it check the LOGS database and creates a list, POSTQUEUE, of boxes to be down loaded. It acquires the configuration, and the site, sample date, and pre weight data and places this information in the POSTQUEUE to drive the POST program for gravimetric analysis. To post weigh filters for the IMPROVE network, the following procedure is used.

1.   Start the POST program at the Cahn 31 electrobalance computer. The program will generate a post weighing queue, which may be viewed in its entirety, as opposed to line by line, by pressing the key <F12>. The single line view will resemble Figure 9 below.

Figure 9 POST Program Menu Screen

<F5> to Skip Box and Remove from Queue   <F1>=calib   <F2>=Controls   <CR> To Continue

POSTWEIGHTS: CAHN31

Site Code CRLA1

LAST CALIBRATION

The Last Calibration Was Done   08/08/96 08:45:50   Tare Wght=-28.569

2.   Retrieve the first box in the queue from the post weight queue shelf to the left of the Cahn 31 balance.

  a)   If this box is not on the post weighing queue shelf, check the contractor queue shelf. If the box is missing, contact the lab manager for assistance. Do not press the <F5> key to remove the box from the queue unless advised to do so by the lab manager. Once <F5> is pressed, the next box in the queue will appear on the screen. Retrieve this box from the downloading shelf.

3.   Press the <Enter> key on the POST program menu screen to post weigh the first box in the queue. A post weight screen, identical to the pre weight screen, except with the pre weights filled in, will be produced. Instructions will be given by the POST program in the message box in the upper right corner of the screen. A highlighted box, the active box, will indicate the location in which data is currently being collected. A sample post weight screen, with the first 'A' post weight filled in, appears in Figure 10 below. Note that once the post weight is entered, the Pre field changes to show the difference between the pre and post weights rather than the pre weight value. In the case of the first filter, MediaId TFEM11689, the pre

weight is no longer displayed. Instead, the difference between the pre and post weights, 0.645 milligrams, is displayed in the pre weight field.

Figure 10 Post Weight Entry Screen

message box   active box

	Place a filter on the balance	

POSTWEIGHTS: CAHN31

SiteCode BLIS1

SamDat/StrTim		MediaId	A_Post	A_Pre	Status	Position	MediaId	D_Post	D_Pre
09/04/96	00:00	TFEM11689	29.132	0.645	NM	1	TFEM11690	0.000	32.685
09/07/96	00:00	TFEM11691	0.000	31.526	NM	2	TFEM11692	0.000	29.746
09/04/96	00:00	TFEM11691	0.000	29.996	NM	1	*field blank*		
09/11/96	00:00	TFEM11693	0.000	28.967	NM	3	TFEM11694	0.000	33.677
09/14/96	00:00	TFEM11695	0.000	29.563	NM	4	TFEM11696	0.000	28.435

4. Remove the 'Week 1' bag of cassettes from the shipping box.
5. Remove the protective red cap from the first cassette (red/black) and verify its cleanliness. If required, wipe the inside of the cap with a Kimwipe dampened with ethanol.
6. Remove the cassette label, containing the site and sample data information and place it on the counter directly in front of a Polonium strip ionizing unit.
7. Remove the cassette top and the lock ring. Transfer the fine Teflon filter to the polonium strip ionizing unit, labeled in step 6, for at least thirty seconds.
8. Check the Week 1 bag for problem tags. Problem filters should be placed in Petri dishes, labeled with both the cassette label removed in step 6 and the problem code, and placed in the Problem Filter Archive.
9. Inspect the Teflon filter.
    a) If an incorrect collection mask was used, change the mask size code on the post weight screen to the correct mask code. Use the <tab> key to increment through the screen to the MediaId. The fifth and sixth characters of the MediaId describe the filter mask. M0, or unmasked, has collection area of 3.51 cm^2, M1 has collection area of 2.2 cm^2, and M2 has collection area of 1.1 cm^2. Only M0 and M1 should be used in the IMPROVE network. If the mask size code listed in the MediaId does not describe the mask used on the filter, type in the correct code. Use the <tab> key to return to the appropriate point on the screen for the next post weight entry.

b)   If the filter is missing or the deposit area is visibly damaged before or during sampling,

     i)   Annotate the field log sheet with a description of the problem, and change the status code to XX.

     ii)   Access the LOGS database through LOGSIN and access the site having the damaged filter. Use the <PageUp> key to move to the correct record.

     iii)   Add the comment on the log sheet to the LOGS database, using the <F12> key.

     iv)   Change the status of the Teflon filter, and the SO_2 filter if it comprises the second stage of the Teflon filter cassette. The new status, XX, should be typed into the status field.

     v)   If there is an SO_2 filter from the same cassette as the problem Teflon filter, remove it from the SO_2 petri tray and transfer it to the Problem Filter box.

     vi)   If the Teflon filter is damaged, remove it from the Polonium strip, insert it into a clean Petri dish, label the Petri dish with the filter identification label from the cassette, and place the Petri dish in the Problem Filter box.

c)   If the deposit appears non-uniform, contact the lab manager, and make a comment on the log sheet. The lab manager shall decide whether to declare the Teflon filter status XX, or to declare it QD. Add the comment on the log sheet to the LOGS database, and change the status of the filter by editing the log sheet through the LOGSIN program as described in part b above.

10.   Check the condition, type, and the cleanliness of the collection mask, the drain disk, and the EP O-ring. Discard the drain disk if dirty and the O-ring if deformed. Clean the filter grate and O-ring by gently brushing with the camel-hair brush reserved for non-quartz cassettes. Discard and replace the collection mask if it is the wrong size, dirty, wrinkled, or if the visible aerosol deposit is not uniformly circular.

11.   Reassemble the cassette.

12.   Remove the filter from the ionizing unit, and place it on the zero area bail (balance pan). Avoid causing the bail to swing or bounce. Once the filter is placed, close the door. At this point the computer will begin checking for stability, and will display the message '*Waiting for Balance to Stabilize*' in the message box.

13.   When the computer determines the balance has stabilized, it will record a weight into temporary memory. The message box will display the following message: "*Press <CR> to record weight or <TAB> to fields you want to change*". Check that the weight appears stable and reasonable, and that the differential weight is reasonable, then press the entry key to record the weight in the computer.

a)   If <CR> is pressed, the weight in the shaded box will be stored in temporary memory, but will not be recorded permanently into the WEIGHTS database until all the filters for the box have been weighed. If the program crashes, or the power goes out, the weights on the screen will

not have been saved. The program will move the active box to the next input position once <CR> is pressed.

b) If <TAB> is pressed, the active box will move to the next input field. Using <TAB> to increment through the screen, data such as mask size, may be changed by keyboard input.   Press <TAB> until the cursor is in the desired location, then enter the corrected information. <TAB> allows movement only within the screen. When the active box reaches the last input field on the screen, it will jump back to the first input field.

14. Once the filter weight has been accepted into the temporary database, the message box will read "*Remove filter from the balance*". Remove the weighed filter from the balance and place it on the Polonium ionizing strip.

15. The filters are stored in labeled slide frames inside 30 position slide trays. Each slide tray is designed to contain the filters for a single site, quarter, and channel (A module or D module).

a) Locate the appropriate slide tray from the shelves around the Cahn 31 electrobalance.

b) Open the slide tray and locate the first empty slide frame (the slide frame in the lowest numbered position).   The label on this frame should correspond to the filter site and sampling date label. If not, determine what happened to the missing filters. The possibilities include:

i) The sample was invalid, so the status was changed and the filter was placed in the problem file. Action: remove the empty slide frame(s) corresponding to the invalid sample(s) from the slide tray and place them in the recycle pile to the right of the Cahn 31 electrobalance.

ii) The box containing the missing filter(s) has not yet returned from the field. Action: verify that the lab manager is aware of the situation, then skip to the correct slide frame and proceed normally.

c) Turn the slide frame upside-down and insert it in the filter mounting apparatus.

d) Center the filter on the slide frame, with the deposit side up, then install the black rear side of the frame over the filter.   Seal the two sides of the slide frame together by pressing firmly, then verifying no gap exists between the black and white slide frame halves. The black frame halves are stored in a box to the right of the Cahn 31 electrobalance.

e) Return the slide to the tray, replacing it in the position from which it was removed. This position must match the position listed on the POST program screen for the filter.

f) The black side of the slide (and the deposit side of the filter) should face the front of the tray (toward tray position 1). This ensures the filter identification label will be visible to the camera during elemental analysis; allowing verification by the analysis technician of correct positioning of the filters during an analysis run.

g) Throw away the label on the counter in front of the Polonium strip ionizing unit that refers to the filter just mounted in the slide frame. It is no longer needed.

16. Repeat steps 5 through 15 for the other Teflon filter cassettes in the Week 1 bag, following the order dictated by the screen; red/black, blue/black, red/white, blue/white (A1, D1, A2, D2).
17. Weigh the field blank, if a teflon filter field blank was included in the box.
18. Repeat steps 5 through 15 for the Week 2 bag of cassettes.
19. Remove the problem tags and the old week date labels from each sample week bag.
20. Transfer the shipping container to the queue for clean filter processing.