IMPROVE Standard Operating Procedure
for the Sample Handling Laboratory
SOP 251

IMPROVE Program
Crocker Nuclear Laboratory
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Table of Contents

1. **PURPOSE AND APPLICABILITY**

This standard operating procedure (SOP) broadly outlines the laboratory procedures for preparing, dispatching, and processing IMPROVE aerosol filter samples to make them available for exposure and, ultimately, for analysis. The preparation of filters for deployment to sites and for analysis is the responsibility of the laboratory manager, staff members (staff research associates and junior specialists), and the student laboratory assistants who work for the Air Quality Group. This SOP covers the period from 1999 when the Version II samplers were introduced until 2011 when the Mettler balances were introduced and the sample handling operations were moved from the Annex to the main Crocker Lab building.

This document is intended to give only the outlines of how samples are handled. Each of the stations involved in sample handling has a specific function and a set of procedures associated with that function. A detailed explanation of each of these procedures is required. Thus, descriptions of the procedures used at each station are given in the Technical Information (TI) documents that are referenced within this SOP.

The goal of processing is to ensure that the samples are handled uniformly, carefully, and systematically, in order to provide the highest degrees of comparability and accuracy possible. Such a goal requires that the processing include procedures for evaluating filters, assessing samples, and removing any samples that do not meet acceptability requirements for elapsed time, proper handling, or flow rate. It also may entail 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 of California, Davis 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 houses the IMPROVE network staff and researchers, as well as the sample handling and gravimetric analysis equipment. Crocker Nuclear Laboratory, a controlled access facility, houses the Hybrid Integrating Plate Analysis System (HIPS), the X-Ray Fluorescence Analysis System (XRF), and the Proton Elastic Scattering Analysis (PESA) system.

The loading and unloading of filters for analysis occurs in the sample handling laboratory, while the receiving and shipping of samples, sample tracking, leak checking, data entry, and communication with site operators are done in the research and testing room.

2. **SUMMARY OF THE METHOD**

"Sample handling" refers to the process in which three types of filters (Teflon®, quartz, and nylon) are prepared for use in the field as well as the initial processing and gravimetric analysis of the returned filters. The initial analysis of returned filters prepares them for compositional analysis by ion chromatography, thermal optical reflectance, or the hybrid integrating plate/sphere (HIPS) method. Filters analyzed using the HIPS method then undergo XRF analysis and PESA.

Sample handling is a cyclical process. The cassettes needed for three weeks of data collection are provided with weighed and approved filters loaded in them; the cassettes are then are packed (with record-keeping log sheets and flashcards) into boxes to be sent to the data collection sites. The filters are then exposed for a standardized length of time. When all cassettes have been sampled, site operators pack the cassettes that contain the exposed filters into the boxes and return the boxes to the IMPROVE laboratory. The cassettes with the exposed filters are opened in the laboratory; the box contents are evaluated, weighed, and organized for analysis. Nylon filters are shipped to the ion contractor and the quartz filters are shipped to the carbon contractor for off-site analyses. Teflon® filters that have been sampled in modules that collect $PM_{2.5}$ undergo HIPS, XRF, and PESA analysis at Crocker Nuclear Lab and are stored in-house. Teflon® filters that have been sampled in modules that collect PM_{10} are stored after gravimetric analysis. After the filters have been removed, the cassettes are loaded with new filters, packed in designated boxes, and sent by common carrier to the data collection sites.

3. **DEFINITIONS**
   * Filter lot: filters manufactured under the same conditions and time, which are grouped by an identifying lot number.
   * Cassette: a plastic holder that contains a filter substrate or "dummy."
   * "Dummy": a 25mm or 37mm piece of material used in cassettes that are not sampled.
   * Cartridge: consists of a cartridge plate and 3-4 cassettes inserted in the cartridge plate.
   * C-clips: plastic clips used to hold the cassette in place in the cartridge.
   * Blue box: boxes that contain filter cartridges.
   * $PM_{2.5}$: Particulate matter, aerodynamic diameter of 2.5 micrometers or less.
   * PM_{10}: Particulate matter, aerodynamic diameter of 10 micrometers or less.
   * "A" filters: 25mm Teflon® filters (3 µm pore size) that are sampled in modules that collect $PM_{2.5.}$
   * "B" filters: 37mm Nylon filters that are sampled in modules that collect $PM_{2.5.}$
   * "C" filters: 25mm quartz filters that are sampled in modules that collect $PM_{2.5.}$
   * "D" filters: 25mm Teflon® filters (3 µm pore size) that are sampled in modules that collect $PM_{10.}$
   * "PRE" station: Station at which "A" and "D" Teflon® filters are pre-weighed on a microbalance, and nylon, quartz, and Teflon˚ are loaded into cassettes. Cassette, bag, and box labels are applied at this station as well.
   * "Leak Check" station: Station at which loaded cartridges and log sheets are double-checked for accuracy before being shipped and where shipping labels are created.
   * "B/C" Download station: Station at which "B" and "C" filters, as well as any terminal status "A" and "D" filters, are downloaded into Petri dishes.
   * "A/D" Download station: Station at which "A" and "D" filters are post-weighed and placed into containers for further analysis or archiving.
   * Slides: used to store the "A" and "D" filters.
   * Field blank (FB): a filter of any of the three substrates (quartz, nylon, Teflon®) that is sent out into the field but is not sampled.

- Neck-ties: thin stickers that have the module letter (A, B, C, D, X) typed on it, used to wrap around cassette tops to indicate which modules they are to be loaded in.
- Control filters: filters that monitor artifact collection of filters in cassettes and that check the performances of the gravimetric analysis systems.
- "Problem" filters: filters that have any of the terminal statuses.
- Terminal status: indicates that a filter will either not be analyzed further or that any previous analysis performed has been declared invalid.
- "PO": terminal status that stands for "Power Outage."
- "BI": terminal status that stands for "Bad Install."
- "EP": terminal status that stands for "Equipment Problem."
- "NS": terminal status that stands for "No Sample/Not Serviced."
- "OL": terminal status that indicates that the site was offline.
- "XX": terminal status that means the filter is invalid for a reason not covered by any other terminal status.
- "NM": status that indicates the filter is normal.
- "QD": status that stands for "Questionable data," analyzed as normal.
- "SO": status that stands for "Sent Out," meaning the filter is out in the field.
- "UN": status that implies that an analysis is missing for a filter.
- "SA": status that stands for "Sample Anomaly," meaning that an unusual occurrence happened during sampling but the sample is considered valid.

4. HEALTH AND SAFETY WARNINGS

Standard laboratory safety and health rules are followed in the sample handling laboratory.

Filter cartridges and used Petri dishes are cleaned using small amounts of ethanol. Ethanol is a colorless liquid that can be irritating to the skin and eyes. Nitrile gloves are available for use if desired in order to prevent direct contact with skin. Ethanol is toxic and not to be ingested. For more information on the use and handling of ethanol, please visit the EH&S website (http://safetyservices.ucdavis.edu).

Polonium strips (radioactive polonium sources) are used as antistatic devices. Their inventory, which includes location, size and appropriate disposal, needs to be kept current at all times according to the EH&S, state, and local regulations. The polonium strips are replaced yearly, and the previous strips are given to EH&S.

5. CAUTIONS

IMPROVE filters are delicate and must be handled with care. If a filter is dropped or torn before being sent out into the field, it must be taken out of circulation and replaced with a clean filter. If a sampled filter is torn or dropped after returning from the field, it must be reported and noted in the database.

Because three different filter types are employed, care must be taken to avoid cross-contamination between filters. Quartz filters are the most prone to flaking and thus special care is to be used when downloading and uploading those filters. Special forceps designated for quartz filters are used to load clean quartz filters and download sampled ones. Kimwipes™ that

are used to clean quartz cassette bottoms are not to be used to clean cassettes from any other filter type.

Special care must be used when downloading and uploading Teflon® filters. Because the Teflon® filters are sampled in either a $PM_{2.5}$ module ("A" filters) or a PM_{10} module ("D" filters), it is important to ensure that the correct weight is recorded for each Teflon® filter and that the filters are uploaded in the appropriate cassette and downloaded in the corresponding slide.

6. INTERFERENCES

There are several interferences that may generate weighing artifacts (gain or loss) of the samples.

Environmental conditions, especially excessively varying temperature or relative humidity, may influence the gravimetric measurements.

Neutralization of the electrostatic charge buildup on the filter (ex. resting filters on the polonium strips prior to weighing) is critical to prevent bias in the weighing process.

Cross-contamination due to the use of red caps to cover the cassettes may take place if they are not regularly cleaned and are not specific to a site or particular cassette. Laboratory personnel are instructed to clean any red caps that appear to be dirty in order to prevent this.

There is a slight risk of losing some sample from Teflon® filters that are placed into slides if there is a large amount of sample on the filter, which may affect any future re-weights. Careful handling is applied in the process to minimize these effects.

Filter contamination may occur during the handling and shipping of the blue boxes. To account for and monitor this issue, field blanks are loaded and analyzed. Field blanks are clean filters that are placed in the same cartridge as regular filters but do not undergo sampling.

7. PERSONNEL QUALIFICATIONS, DUTIES, AND TRAINING

7.1 Supervising Staff Research Associate (Laboratory Manager)

The staff research associate who holds the title of laboratory manager will:

- Oversee and maintain records on site and sampler operation.
- Supervise and train staff specialists and associates.
- Oversee the resolution of any inconsistencies on the log sheets or in the samples.
- Oversee filter-handling procedures.
- Ensure the cleanliness and orderly procedure of the laboratory and its functions.

7.2 Staff Members

Staff members will:

- Oversee and train new student laboratory assistants.
- Download flashcard data.
- Review all log sheets for completeness.
- Resolve any inconsistencies on the log sheet or in the samples.

- Enter log sheets into the LOGS database.
- Contact operators regarding procedural problems.
- Oversee filter-handling procedures.
- Order supplies, as necessary, for laboratory use.
- Clean and maintain the sample handling laboratory.
- Assist with sample handling if necessary.

7.3 Student Laboratory Assistants

The student laboratory assistants will:

- Pre-weigh and individually identify filters for use at IMPROVE aerosol sampling sites.
- Load filters into sampling cassettes.
- "Leak Check" sampling cassettes (check integrity of filters and ensure correct loading of screens, filters, and dummy positions).
- Mail cassettes in shipping containers to sites.
- Receive exposed cassettes.
- Download filters for Ion Chromatography or Thermal Optical Reflectance analysis into labeled Petri dishes.
- Post-weigh exposed filters.
- Download "A" and "D" Teflon® filters into labeled slides.

Training in the IMPROVE sample handling laboratory follows a general pattern. New staff members and laboratory assistants are trained on the nylon/quartz download ("B/C download") and "Leak Check" stations first in order to get familiar with sample handling and to learn how to identify properly loaded filters. The "A/D download" station is taught next, where general balance training occurs, followed by the "PRE" station. Detailed instructions on these stations can be found in the TI documents for SOP 251.

8. EQUIPMENT AND SUPPLIES

The equipment and materials required for sample handling vary from station to station and are detailed in the individual technical documents.

9. PROCEDURAL STEPS

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. Sample handling entails only the work done in the IMPROVE sample handling laboratory and the research and testing room, which are housed in the Annex building of Crocker Nuclear Lab on the UCD campus.

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

A. Balance Calibration and Controls

B. Box Processing
C. "B/C" Download
D. "A/D" Download
E. "PRE"
F. The "Leak Check" Station
G. "B/C" Shipping

A. *Balance Calibration and Controls*

Each Teflon® filter is weighed both before it is placed in the cassette (before being sent to the site) and after being sampled at the site. The weighing procedure is an important element in the quality control of the analysis of the filters. In order to provide the highest degree of comparability in the reports from day to day, the balances in the weight lab are calibrated twice every day, once in the morning and once in the afternoon. Control filters monitor both artifact collection on the filters as well as balance performance. Control filters are weighed after calibrations. Morning calibrations and controls are completed before any of the weighing for the day can begin. The procedures involved in calibrating the balances and performing controls are described in detail in the technical document TI 251B.

B. *Box Processing*

Boxes containing sampled filters are delivered every day to the main building of Crocker Nuclear Laboratory. They are then transferred to the Box Processing station in the Annex building. At this station, the boxes are inspected and the flashcard and log sheets are removed. If any problems are noted at this stage, they are resolved or flagged for further inquiry. The data on the flashcards are checked and downloaded. Log sheets are checked for validity and entered into a computer database. A detailed account of procedures used at this station is described in the technical document TI 251C.

C. *"B/C" Download*

After the initial box processing station, the box then goes to the first of the download stations, "B/C Download." At this station, student lab assistants retrieve boxes according to a computer-generated download list and remove the sampled nylon ("B") and quartz ("C") filters from their cassettes. The filters are inspected for flaws or injury and then are placed in labeled Petri dishes. Any terminal status "A" or "D" filters are removed at this station as well. These Petri dishes are placed in labeled Petri trays, which are then stored in the refrigerator (for nylons) or freezer (for quartz) until they are shipped for off-site evaluation and analysis.

A detailed description of the procedures used at this station is provided in the associated technical document TI 251D.

D. *"A/D" Download*

After a box has gone through the "B/C Download" station, it is ready for "A/D Download." At this station, student laboratory assistants retrieve boxes according to a computer-generated queue and weigh the sampled "A" and "D" Teflon® filters on a microbalance. Weights are recorded in a computer program. The filters are then placed in labeled slides and are stored in designated slide trays. Any problems are noted and brought to the attention of a staff member.

The procedures used at this station are explained further in the technical document TI 251E.

E. "PRE"

After being processed through the download stations, boxes are now full of empty cassettes and need to be provided with fresh filters. This process is accomplished at the "PRE" station, which is assigned only to staff members or experienced student laboratory assistants. At this station, cassettes are loaded with clean filters, field blanks are added, and flashcards are assigned. Labels for the blue boxes, week-bags, and cassettes are applied at this station as well.

Further details are provided in the technical document TI 251F.

F. The "Leak Check" Station

The "Leak Check" station serves as a final quality control for outgoing boxes. The contents of each of the three reclosable bags in a box are examined. All three week-bags should contain a log sheet and cartridges loaded with fresh filters. The Week 1 bag should also contain a flashcard. A shipping label is then printed and applied to the box.

A detailed description of the procedures used at this station is described in two technical documents, TI 251G ("Leak Check" Station) and TI 251H (Blue Box Shipping).

G. "B/C" Shipping

While the "A" and "D" filters are analyzed on-site at the Crocker Nuclear Laboratory, the "B" and "C" filters are organized and then sent offsite for analysis. "B" filters are shipped to the Research Triangle Institute

Directions for organizing and shipping these filters are located in TI 251D.

10. DATA AND RECORDS MANAGEMENT

The main programs used in the sample handling laboratory are FoxPro® programs called "PRE" and "POST." These programs prompt the user to weigh specific boxes through a queue and record the weights and other information for each sample in two databases, WEIGHTS.dbf and LOGS.dbf, respectively.

11. QUALITY ASSURANCE AND QUALITY CONTROL

The sample handling lab focuses on several areas to limit sources of possible contamination and to ensure accurate filter weighing and loading. These areas include the cleaning and maintenance of the

room, the calibration and maintenance of the balances, and procedures that occur during sample loading and downloading to prevent loading mistakes and to document possible contamination during the process.

11.1 Cleaning and Maintenance of the Sample Handling Laboratory

The entry to the sample handling laboratory is situated so that there is no pass-through foot traffic. The laboratory area floor and work surfaces are vacuumed weekly with a high efficiency HEPA cleaner. All counter surfaces are cleaned weekly with reagent grade alcohol, though the areas in front of the microbalances are cleaned daily. The entire Air Quality Group Annex building is on the same central air supply system. However, the air is separately filtered before it enters the sample handling room, and the room is maintained at positive pressure to reduce fugitive dust levels.

11.2 Balances

Several methods are employed to ensure that the balances are weighing accurately. Laboratory quality control checks include replicate weighing of control filters and twice daily weighing of "test" weights. These checks are detailed below:

11.2.1 "Test" Weights

Weight tests are performed to track the validity of balance equations throughout the day and to check the consistency between the balances. 50mg "test" weights are weighed twice a day. At these times, a tare weight, a calibration weight, a "test" weight, and a gauss meter reading are taken for each Cahn balance through the FoxPro® POST program. These "test" weights have shown that the balances maintain reliable balance equations throughout the day.

11.2.2 Control Filters

The control filters check the performances of the gravimetric analysis systems over the typical period between pre- and post-weighing of filter samples. Control filters monitor the artifact collection of filters in cassettes. They also provide a twice-daily check of the correlation between the master balance (Cahn 31) and the other balances used in filter processing.
A fresh control filter is weighed on both balances in the morning and afternoon every day after "test" weights are measured. It is then placed in a cassette and stored for 32 days. A second control filter, weighed 32 days previously and stored in a cassette, is removed from the cassette and also weighed on both balances. More detailed instructions can be found in TI 251B.
The control filters facilitate determination of the following:
- Any change in the equivalency of the master balance and the other balances. The balances should produce filter weights that are within ±0.003mg of each other. If the weights do not correlate well, the problem is noted and the metallic test weight is checked.
- Any change in any balance between morning and afternoon.

- Any shift in readings between the pre-weights and post-weights for an ambient sample. As pre- and post-weights are performed about five 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 microbalance.

11.3 Quality Assurance Procedures in the Laboratory

Multiple steps are taken to make sure that samples are loaded and weighed properly and that any contamination is noted. These steps include "PRE" and "Leak Check," which are detailed in the Procedures section of this SOP and in technical documents TI 251F and TI 251G. New Teflon® filters received from the manufacturer are inspected for defects at the "PRE" station and only defect-free filters are utilized and loaded into cartridges. The "Leak Check" station is in place to ensure that cartridges are configured and labeled properly as well as to check that the clean filters being sent out are free of any contamination and have been correctly loaded. When samples return from the field and are downloaded, any potential contamination or filter damage is reported to a staff member and/or the laboratory manager and noted in the LOGS database for review after further analysis.

12. REFERENCES

EH&S Website: http://safetyservices.ucdavis.edu
TI 251A: Box Cycles and Cartridge Orientation
TI 251B: Calibration and Control Filters
TI 251C: Box Processing
TI 251D: B/C Download
TI 251E: A/D Download
TI 251F: PRE
TI 251G: Leak Check Station
TI 251H: Blue Box Shipping

Related SOPs:
SOP 276: Optical Absorbance
SOP 301: XRF Analysis of Aerosol Deposits on Teflon Filters
SOP 351: Data Processing and Validation