

IMPROVE Standard Operating Procedure for the Sample Handling Laboratory SOP 251

*IMPROVE Program
Crocker Nuclear Laboratory
University of California, Davis*

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DOCUMENT HISTORY

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3.1	11/1/2015	TK/KT	Section 1-12	The SOP reflects the integration of the new Lab Application and the use of Reporting Services to review and manage data. Minor updates in procedures and terminology reflect these adaptations.

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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 PM collection and, ultimately, for analysis. The preparation of filters for deployment to sites and for analysis is the responsibility of laboratory technicians and the student laboratory assistants under the general supervision of the Laboratory Manager.

This document is intended to give only the outlines of how samples are handled. Each of the processes involved in sample handling has quite 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 filter 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 includes 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 may also entail contacting site operators, as necessary, to correct faulty collection techniques.

Filter preparation, sample handling and gravimetric analysis currently take place in the sampling handling laboratory in Crocker Nuclear Laboratory (Jungerman Hall) at the University of California, Davis.

2. SUMMARY OF THE METHOD

Clean Teflon[®], nylon, and quartz filters are loaded into cartridges to be sent out to approximately 170 IMPROVE sites. The following are the major steps in the process of handling the filters:

- The Teflon[®] and nylon filters are obtained from the manufacturer and are characterized for the specific lot/shipment (see TI 251C for more details). The quartz filters are obtained from the Desert Research Institute, where they are pre-fired prior to shipment.
- Clean filters are visually inspected for contamination or tears.
- The Teflon[®] filters are pre-weighed on a Mettler XP6 microbalance and the values are recorded in a database.
- Filters are packed with corresponding log sheets and a flashcard and are then shipped out to IMPROVE sites.
- After filters have been sampled and are shipped back, they are processed and analyzed by the sample handling lab. This includes reviewing the data on the flashcards and accompanying log sheets, processing and preparing the nylon and quartz filters to be shipped for further analysis, weighing the sampled Teflon[®] filters on a Mettler XP6 balance, and recording the post-weights in a database.
- Teflon[®] filters that have been sampled in a PM_{2.5} module are prepared for additional analysis (XRF analysis). Teflon[®] filters sampled in a PM₁₀ module are archived.

3. DEFINITIONS

- Gravimetric analysis – determination of particulate concentration based on the difference between post and pre weight of each sample
- Mettler XP6 microbalance: microbalance with readability of 1 µg and a maximum capacity of 6.1g.
- 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.
- Loose screen: a stainless steel 25 mm screen that is placed on top (downstream) of the Teflon® and quartz filters after they have been loaded into cassettes.
- PM_{2.5}: Particulate matter, aerodynamic diameter of 2.5 micrometers or less.
- PM₁₀: 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 PM₁₀.
- Cartridge Preparation station: Station at which “A” and “D” cartridges are cleaned, all cartridges are labeled, and “B” and “C” filters are loaded.
- Pre-Sample Weigh In: Station at which “A” and “D” Teflon® filters are pre-weighed on a microbalance and loaded into cassettes.
- Quality Control (Q/C) station: Station at which loaded cartridges and log sheets are double- checked for accuracy before being shipped and where shipping labels are created.
- Shipping/receiving station: Station at which filters are packed into designated blue boxes and prepared for shipping, as well as where filters are unloaded from blue boxes and prepared for analysis upon their return.
- Post-Sample Processing station: Station at which “B” and “C” filters transferred into Petri dishes.
- Post-Sample Weigh In: Station at which “A” and “D” filters are post-weighed and placed into containers for further analysis or archiving.
- Field blank (FB): a filter of any of the four substrates (quartz, nylon, Teflon®) that is sent out into the field but is not sampled.
- Neckties: thin stickers that have the module letter (A,B,C,D) typed on it, used to wrap around cassette tops to indicate which modules they are to be loaded in.
- Lab Blanks: 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 screens as well as 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.

5. CAUTIONS

Laboratory coats and gloves are available for all personnel and help minimize the potential for laboratory contamination. The clean room floor mat at the entrance to the lab also helps to avoid major contamination from outside the lab.

The temperature in the laboratory can be set and controlled from within, but the control of RH (relative humidity) is much more difficult to obtain. Both parameters are monitored and registered regularly.

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 discarded 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 processing and loading those filters. Special forceps designated for quartz filters are used to load clean quartz filters and processing sampled ones. Kimwipes™ that are used to clean quartz cassette bottoms are not to be used to clean cassettes from any other filter type. Loose screens used in quartz cartridges are also cleaned and stored separately from those used in Teflon® cartridges.

Special care must be used when processing and loading Teflon® filters. Because the Teflon® filters are sampled in either a PM_{2.5} module (“A” filters) or a PM₁₀ module (“D” filters), it is important to ensure that the correct weight is recorded for each Teflon® filter and that the filters are loaded in the appropriate cassette and processed in the corresponding container.

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 (passing them through Haug anti-static units, keeping polonium strips inside the balance weighing chambers, etc.) 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.

Minimal sample may be lost when filters are removed from their cassettes, particularly when the filter sticks to the screen. There is also 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.

7. PERSONNEL QUALIFICATIONS, DUTIES, AND TRAINING

The sample handling laboratory’s personnel consists of laboratory technicians and laboratory student assistants. All laboratory personnel perform under the general supervision of the laboratory manager.

All personnel employed in the sample handling laboratory obtain extensive training in sample handling and must have familiarity with the SOPs, with the procedures for each station (detailed descriptions of these procedures can be found in the technical documents associated with this SOP) and with gravimetric analysis before being allowed to process any of the actual IMPROVE samples.

In general, the technical laboratory personnel should meet the minimum qualifications listed below:

- Undergraduate-level course work in chemistry, physics, and mathematics including laboratory classes and/or equivalent experience.
- Experience working in a sample handling laboratory.
- Interpersonal, verbal and written communication skills to clearly and effectively interact with a diverse group of individuals to secure and/or provide information to clarify situations, resolve problems.
- Knowledge and technical experience with analytical instrumentation.

- Experience calibrating analytical equipment.
- Experience using diagnostics tools to determine sources of errors and how to correct them.
- Experience initiating, establishing, interpreting and implementing laboratory procedures.
- Experience working with computer data analysis and graphing software and MS Office software.
- Bachelor's degree and experience in a physical sciences discipline or equivalent combination of education and experience.

The laboratory technician shall:

- Oversee and train new lab technicians and student assistants
- Review flashcard data
- Review all log sheets for completeness, and check the validity of the samples prior to processing of the samples by lab assistants
- Resolve any inconsistencies on the log sheet or in the samples
- Enter log sheets into the logs database
- Contact site 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
- Clean loose screens for quartz and Teflon® filters with reagent grade alcohol

The student laboratory assistants shall:

- Pre weigh and individually identify filters for use at IMPROVE aerosol sampling sites
- Load filters into sampling cassettes
- "Q/C" 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
- Process filters for Ion Chromatography or Thermal Optical Reflectance analysis into labeled petri dishes
- Post weigh exposed filters
- Process "A" Teflon® filters for XRF analysis into labeled petri dishes
- Process "D" Teflon® filters into slides for storage
- Clean cassette bottoms, cartridge plates, and 37mm screens thoroughly with Kimwipes™ and reagent grade alcohol

Training in the IMPROVE sample handling laboratory follows a general pattern. New laboratory technicians and laboratory assistants are trained on the nylon/quartz processing (Post-Sample Processing") and "Quality Check" stations first in order to get familiar with sample handling and to learn how to identify properly loaded filters. After these stations

have been successfully learned, new employees are taught the Cartridge Preparation station to familiarize them with the two different site schedules (2-3-2' and 3-2'-2). The Post-Sample Weigh In station is taught next, where general balance training occurs, followed by the Pre-Sample Weigh In station. Detailed instructions on these stations can be found in the TI documents for SOP 251.

8. EQUIPMENT AND SUPPLIES

The sample handling laboratory employs the use of two Mettler XP6 microbalances to do the pre- and post-weighing of the Teflon® filters. Documentation and technical specs are located in the Mettler XP6 User Manual, which is kept in the sample handling laboratory at all times.

Equipment and materials required for filter handling are listed below:

Filter and Cassette Requirements

- Quality tested and approved stretched Teflon® membrane filters
- Quality tested and approved nylon filters
- Acceptance tested quartz filters prepared by the Desert Research Institute
- Filter cartridge and cassette parts, and completed cartridge and cassettes constructed and assembled:
 - Cartridge plates
 - O-rings—hydrogenated nitrile butadiene rubber and Teflon® encapsulated silicone (FEP)
 - 25mm and 37mm cassette bottoms
 - Cassette tops for 25mm filters
 - Cassette tops with fixed screens for 37mm filters
 - Clean 25mm loose screens for Teflon® and quartz cassettes
 - C-clips
- Red, yellow, green and blue dot stickers for cartridge plate labeling
- U-Line 8x10" 6 Mil re-closable bags
- Red, yellow, green and blue "neckties" for cassette tops
- Forceps, stainless steel
- Red protective caps for filter cassettes
- Assorted stickers for labeling boxes, bags, etc.
- Filter-slide mounts, 18x24, 2mm
- Labeling supplies (Sharpies and highlighters, assorted colors)
- Blue site-specific shipping boxes, 3 sizes
- Reagent grade alcohol
- Kimwipes™
- Compact flashcards
- Small re-closable bags for flashcards
- Log sheet paper

- Printer

Filter Weighing Equipment

- 2 computers with Lab Application and network connections
- 2 Mettler XP6 microbalances, sensitive to $\pm 1 \mu\text{g}$
- 2 Mettler weighing tables
- 2 computers capable of running Microsoft FoxPro®
- Polonium strip ionization units, small and large
- Stainless steel test weights, 100mg and 50mg (Troemner UltraClass™)
- Forceps, stainless steel with ceramic tips
- 2 HAUG U-electrode antistatic units (1 per balance)

Exposed Filter Processing Equipment

- Computer for running macros associated with nylon/quartz/problem filter processing and Lab Application
- Stainless steel blunt tipped forceps
- Stainless steel forceps with ceramic tips
- Petri dishes for ion and carbon analysis filters
- Reagent grade alcohol
- Kimwipes™
- Container for used loose quartz screens
- 4 numbered, 50-position petri dish trays (1 for quartz filters, 1 for nylon filters, 1 for “problem” filters, 1 for inactive quartz field blanks)
- Petri dish shipping trays for quartz and nylon filters
- Shipping boxes (12” cubed)
- Blue Ice cooler packs (for quartz shipments)
- Nitrile gloves
- Arbor presses
- Slides for Teflon® filters
- Slide stickers
- Numbered, 50-position Petri dish trays for “A” and “D” Teflon® filters
- Petri dishes
- Container for used Teflon® screens
- Cotton swabs
- Temperature probe

Blue Box Shipping Equipment

- Computer with Internet access and Lab Application
- Thermal printer
- UPS label rolls
- UPS pouches
- Blue boxes

- Boxtop stickers
- Box labels
- USPS mail pouches

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 in the designated shipping/receiving area for the sample handling lab. Both of these areas are housed in Crocker Nuclear Laboratory (Jungerman Hall) on the UCD campus. Standard Operating Procedure #201 covers field operations used by the site operators.

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

- A. Purchase and Preparation of the Filters and Cassettes at UC Davis and at the Carbon Laboratory at the Desert Research Institute
- B. Balance Calibration and Controls
- C. Preparation of Cassettes and Loading of Nylon and Quartz Filters
- D. Pre-Measurement of the Gravimetric Mass and Loading of Teflon® Filters into Cassettes
- E. "Quality Check" of Cassettes and Preparation for Shipping to the Field
- F. Receipt of Boxes from the Site and Entry of Data into Computer
- G. Processing of the Exposed Nylon and Quartz Filters for Ion Chromatography or Thermal Optical Reflectance Analysis and Terminal Status Filters
- H. Nylon and Quartz Filter Shipping
- I. Processing of the Exposed Teflon® Filters for Post-Measurement of the Gravimetric Mass

A. Purchase and Preparation of the Filters

Teflon® and nylon filters are purchased by UC Davis and undergo preliminary testing before full shipments are accepted for delivery. The acceptance testing on quartz filters is done by the quartz contractor before filters are sent to the sample handling laboratory at UC Davis and deployed to the field. Detailed descriptions of the filter acceptance testing and preparation procedures performed at UC Davis can be found in TI 251C.

B. Balance Calibration and Reference Weights

Two Mettler Toledo XP6 microbalances are employed for gravimetric analysis of IMPROVE samples. The microbalances were certified upon initial installation by a Mettler technician and serviced/recertified at least annually and on an as needed basis by an authorized Mettler technician. Records of all tests performed and the certifications are kept in the laboratory.

In order to provide the highest degree of comparability in the reports from day to day,

the balances in the sample handling laboratory are calibrated in the morning and checked three times a day with reference weights (Troemner UltraClass™ stainless steel weights, 100mg and 50mg). Morning calibration, test weights, and reference filters are completed by a lab technician before any of the weighing for the day can begin. The balances must meet certain criteria during calibrations and test weights in order to be cleared for weighing IMPROVE samples. These criteria and the procedures involved are described in detail in the Quality Assurance section of this SOP and in TI ff 251A and TI 251B.

C. Preparation of Cassettes and Loading of Nylon and Quartz Filters

The first station involved in the preparation of filters to be sent out to the field is the Cartridge Preparation station. At this station, the cartridges from all three weeks are inspected to ensure that they are oriented correctly and that they have been placed in the proper bags after processing. Detailed explanations of proper cartridge orientation and box schedules can be found in TI 251Q, "Box Cycles and Cartridge Orientation."

"A" and "D" cartridges and cassette bottoms are cleaned at this station. Flashcards are also assigned and entered into the database here. All cartridges and week bags are then labeled with new stickers for the next cycle. The instructions for printing labels can be found in TI 251P, "General Laboratory Procedures." Nylon and quartz filters are loaded at this station, and field blanks are assigned as necessary. For specific, step-by-step instructions for the procedures at this station please refer to TI 251H.

D. Pre-Measurement of the Gravimetric Mass and Loading of Teflon® Filters into Cassettes

The pre-weighing of Teflon® is done at this station using the Lab Application. "A" and "D" filters are weighed and the values are recorded along with the site names and sample dates for the filters. Filters are loaded into cassettes, and complete cartridges are placed into the proper bags. Finally, log sheets that were generated for each week by the Lab Application are collected from the printer and put into the corresponding week bag. Detailed instructions for this station can be found in TI 251I.

E. Quality Check of Cassettes and Preparation for Shipping to the Field

The "Quality Check," or "Q/C," station is where cartridges are inspected to ensure that they have been loaded properly. Once the cartridges and log sheets have been checked and any errors remedied, UPS labels are printed (if applicable) and the bins are moved to the designated shipping/receiving area, where the blue shipping boxes are kept.

Filters are moved from bins to their appropriate blue boxes. A check is performed to make sure all loaded filters have been transferred from bins to boxes and prepared for shipping. UPS/USPS labels are affixed to the boxes, and then the boxes are secured with packaging tape. Boxes are then shipped via UPS or USPS. Detailed instructions for these procedures may be found in TI 251J and TI 251K.

F. Receipt of Boxes from the Site and Entry of Data into Computer

When sampled boxes arrive, lab personnel remove their contents and place them in their corresponding bins in the designated shipping/receiving area for the sample handling lab. Flashcards and log sheets are removed and stacked into piles, while bins are placed onto a cart and transported to the sample handling laboratory. Step-by-step directions for opening blue boxes can be found in TI 251D.

Flashcards and log sheets are given to lab technicians, who first download and review the data from the flashcard. Then, lab technicians enter the log sheet data into the system, which allows the filters to begin the process. More details on this process can be found in TI 251E.

G. Processing of the Exposed Filters for Ion Chromatography or Thermal Optical Reflectance Analysis and Terminal Status Filters

At the Post-Sample Processing station, lab personnel, prompted by the Lab Application, remove the sampled quartz and nylon filters, as well as any terminal status filters. The filters are inspected for flaws or damage and placed in labeled Petri dishes. "B" (nylon) and "C" (quartz) cartridges are cleaned and "C" loose screens are set aside for cleaning. Any problems during the process are noted and reported through the Lab Application. Processed nylon and quartz filters are placed in labeled boxes before they are sent for off-site analysis and evaluation. Step-by-step instructions for this procedure can be found in TI 251F.

H. Nylon and Quartz Filter Shipping

Although the "A" and "D" filters are analyzed on-site at Jungerman Hall, the "B" and "C" filters are sent offsite for analysis. The techniques used for shipping these filter to the Research Triangle Institute ("B" filters) and the Desert Research Institute ("C" filters) are explained in TI 251L.

I. Processing of the Exposed Filters for Post-Measurement of the Gravimetric Mass

The final step of this process is the post weighing of Teflon® filters. Lab personnel, prompted by the Lab Application, retrieve the appropriate bins. Lab personnel then open the cassettes and remove the sampled filters. Filters are inspected for damage and then weighed. The results are recorded by the computer program. Any problems that occur (including extreme or negative mass differences between pre-weigh and post-weigh) during this process are noted and reported to a lab technician. A lab technician checks the weights and weight differences of all samples weekly to ensure that valid weights were taken. If any weights are found to be invalid, filters are reweighed to check for filter swaps. More information on weight validation can be found in TI 251P.

"A" Teflon® filters are prepared and stored for future XRF analysis. "D" Teflon® filters are placed into containers and stored permanently. Loose screens are placed into a container for later cleaning. For instructions on preparing and storing Teflon® filters, see TI 251M. For instructions on how to clean loose screens, see TI 251N.

Once all the filters have been processed, bins are moved to the Cartridge Preparation

station shelf, where the process will begin again. Step-by-step instructions for the Post-Sample Weigh In procedures are located in TI 251G.

10. DATA AND RECORDS MANAGEMENT

The main program used in the sample handling laboratory is the Lab Application. This program prompts the user to weigh specific boxes and record the weights and other information for each sample in two databases, AnalysisWeights and Logs, respectively

A web application called IMPROVE Data Manager is also important for laboratory functions. It is used to ingest flashcard data into the SQL server.

For more information on these, please see TI 251R.

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 processing to prevent loading mistakes and to document possible contamination during the process.

11.1 Cleaning and Maintenance of the Sample Handling Laboratory

To reduce dust levels in the sample handling room and to prevent filter contamination, inlet air coming through the vents into the room must pass through high efficiency filters that are changed every 4-6 months. Air deflectors are placed on the vents to direct the air away from the balances. Entrance into the laboratory requires walking over a clean room floor mat to capture dust and foreign particles. The room is cleaned twice a week. This includes cleaning the floors with a high efficiency HEPA vacuum and wiping down all counter surfaces with reagent grade alcohol and Kimwipes™. One day a week the floor is also cleaned with a Swiffer® to help eliminate any residue that cannot be removed with the vacuum. These procedures reduce the possibility for contamination should a filter fall to the work surface. Following the cleaning, no analysis shall occur for at least 12 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 building. The temperature is set at or near 22°C and stays mostly within a ±3°C range.

Due to the central air processing system, relative humidity is not a controlled parameter and usually ranges between 20-50%. The sample handling lab has a humidifier and a dehumidifier that are utilized to keep the RH as stable as possible.

11.2 Balances

Several methods are employed to ensure that the balances are weighing accurately. Weighing is performed with a microbalance having a readability of 1µg. Laboratory quality control checks include lab blanks that are weighed weekly, replicate weighing of control filters, and daily weighing of test weights. These checks are detailed below:

11.2.1 Internal Calibration/Weighing Procedures

The Mettler balances have been programmed using the proFACT function to automatically do an internal calibration every morning. During an internal calibration, the balances generate a 3-point calibration equation with two internal masses. The weights of these internal masses combined are in the range of 5-6 grams. At the start of the work day, lab personnel manually redo the internal calibration. Please see Flow Chart 1 for a diagram of the calibration procedure. Detailed information can be found in TI 251A.

Several measures are in place to reduce the effects of static. Filters are passed through a Haug anti-static unit before they are placed in the balance. A small polonium strip is inside the weighing chamber of the balance in order to dispel any extra static. However, despite this protocol, static may at times cause the balance not to return to zero between filter weights. If this occurs, the balance is tared and an internal calibration test is performed. If the test is not passed, the balance is re-calibrated.

Each Mettler balance is placed on a Mettler XP Micro weighing table designed to prevent vibrations and therefore to decrease stabilization times for samples.

The balances are calibrated annually by Mettler technicians, who compare the internal calibration weights against Mettler's own traceable weights and make adjustments to the electronics as needed.

11.2.2 External Reference Weights

External reference weight tests are performed to track the validity of balance equations throughout the day and to check the consistency between the two balances. Two metallic calibration masses are weighed three times a day. At these times, a tare weight, two metallic reference weights, and a gauss meter reading are taken for each XP6 balance through the Lab Application. These external tests have shown that the balances maintain reliable balance equations throughout the day.

External reference weights must weigh within ± 0.003 mg of their expected averages. If a weight does not meet this criterion, it is reweighed. If it fails again, it is weighed on the other balance to see if the other balance agrees. If the test weight is out of range for only one balance, that balance is recalibrated. If both balances agree that a test weight is out of range, it is sent for recertification and another test weight is used in its place. Each test weight should also weigh within ± 0.003 mg when compared between balances. If this test fails, the mass is reweighed on each balance. If it continues to fail, the other test weight is checked on both balances. If both weights fail this criterion, the historical averages for each mass on each balance are noted. The balance that shows the most historical inconsistency is recalibrated and the masses are reweighed. If this does not solve the issue, the other balance is recalibrated as well. If the test continues to fail, a Mettler technician is contacted and one or both balances (depending on the issue) are designated as out of service until the issue is resolved. Please see Flow Chart 2 for a diagram of the external test weight procedure.

11.2.3 Lab Blanks

The lab blank checks the performances of the gravimetric analysis systems over the typical period between pre- and post-weighing of filter samples. Lab blanks monitor the artifact collection of filters in cassettes. They also provide a daily check of the correlation between the two microbalances used in filter processing.

A fresh control filter is weighed on both balances in the morning twice a week after reference weights are measured. It is then placed in a cassette and stored for three weeks. A second control filter, weighed three weeks 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 lab blanks facilitate determination of the following:

- Any change in the equivalency of the two balances. The balances should produce filter weights that are within ± 0.003 mg of each other. If the weights do not correlate well, the problem is noted and metallic test weights are checked.
- Any shift in readings between the pre-weights and post-weights for an ambient sample. As pre and post weights are performed about three weeks apart, a drift or shift in either balance could lead to erroneous gravimetric measurements. Lab blanks provide a daily record of balance consistency.
- The uncertainty of the analysis. The difference between the weights and re-weights provides an estimate of the precision of each microbalance.

11.2.4 Reference Filters

Twice a week, ten blank and four sampled reference filters are weighed on both balances. These filters are tracked to see how the mass differs over time for clean and sampled filters.

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-Sample Weigh In," "Q/A," "Quality Check," and "Blue Box Shipping" stations, which are detailed in the Procedures section, TI 251H, TI 251I, TI 251J, and TI 251K. New Teflon® filters received from the manufacturer are inspected for defects at the "Pre-Sample Weigh In" station and only defect-free filters are utilized and loaded into cartridges. The "Cartridge Preparation and Quality Control stations are in place to ensure that cartridges are configured and labeled properly and also to make sure that the clean filters sent out are free of any contamination and have been correctly loaded. A semi-automated check is performed when filters are transferred to their designated blue boxes to ensure that all loaded bins have been sent out into the field.

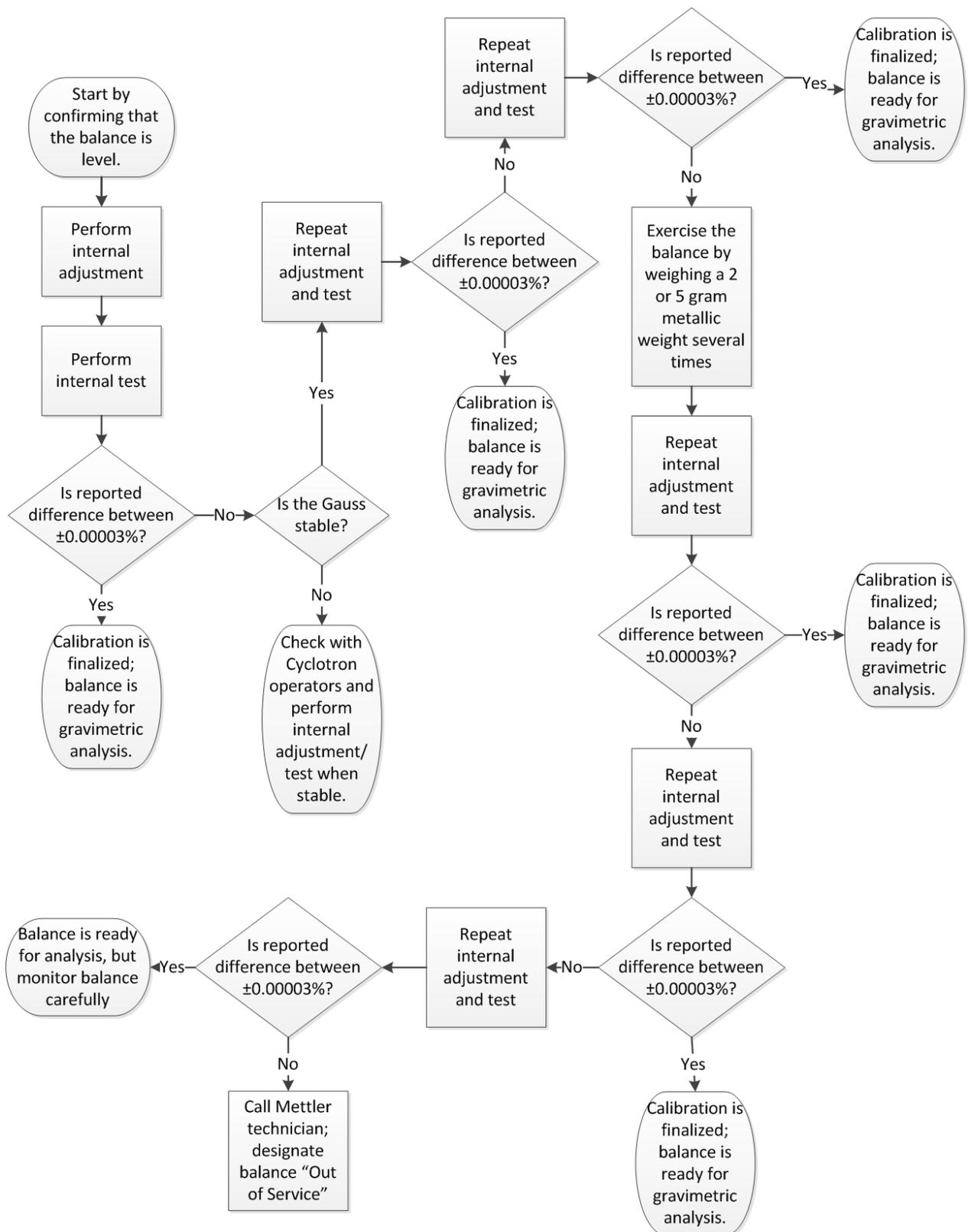
When samples return from the field and are processed, any potential contamination or filter damage is reported to a laboratory technician and/or the laboratory manager and noted in the LOGS database for review after further analysis.

12. REFERENCES

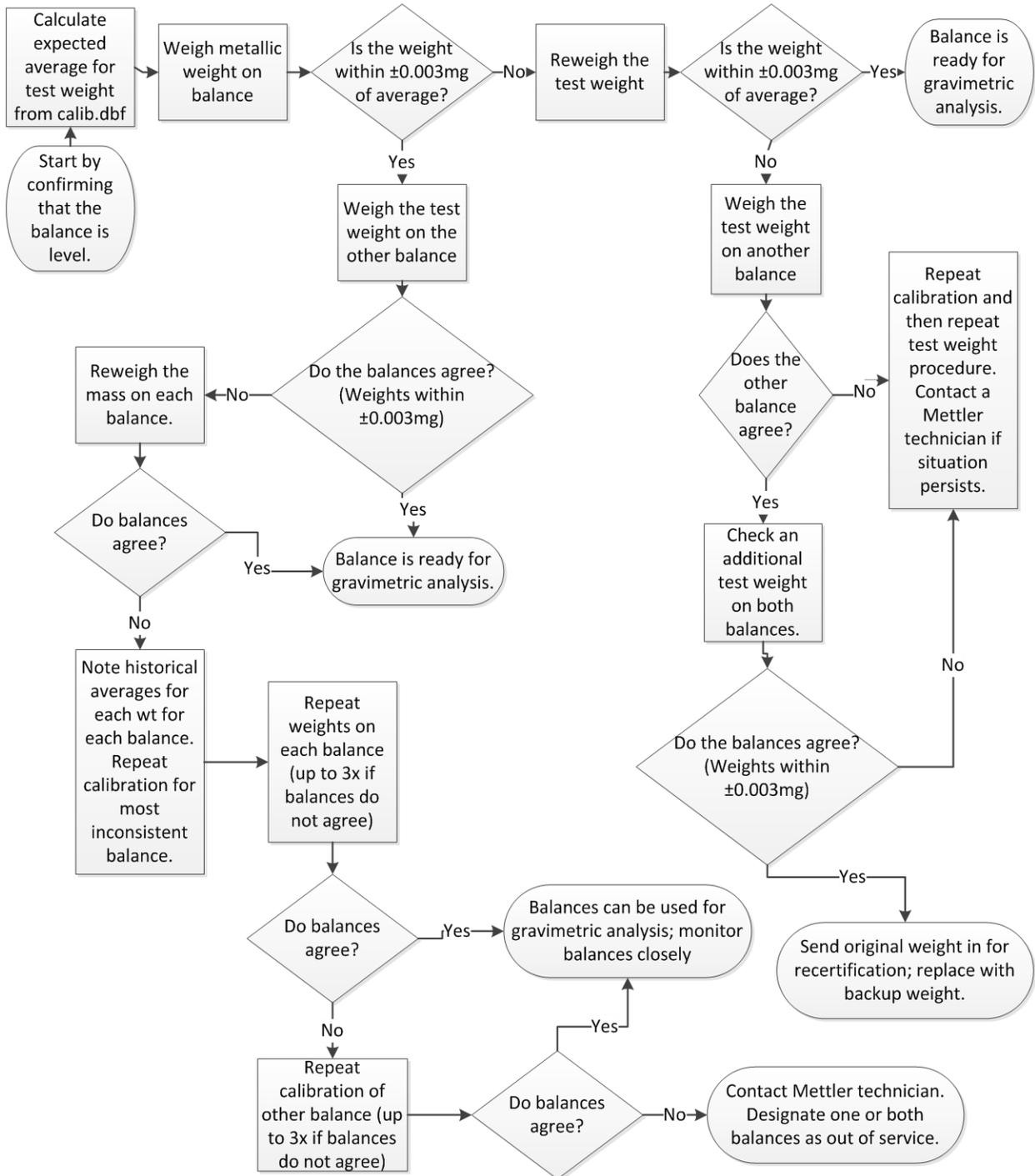
EH&S Website: <http://safetyservices.ucdavis.edu>
Mettler XP6 Microbalance Operations Manual
SOP 201: Sampler Maintenance by Site Operators
TI 251A: Reference Weights
TI 251B: Lab Blanks
TI 251C: Filter Inventory and Acceptance
TI 251D: Incoming Blue Boxes
TI 251E: Entering Log Sheets and Simple Problem Diagnosis
TI 251F: Post-Sample Processing
TI 251G: Post-Sample Weigh In
TI 251H: Cartridge Preparation
TI 251I: Pre-Sample Weigh In
TI 251J: Quality Check Station
TI 251K: Blue Box Shipping
TI 251L: BC Filter Shipments
TI 251M: A Filter Tray Check
TI 251N: Screen Cleaning
TI 251P: General Laboratory Procedures
TI 251Q: Box Cycles and Cartridge Orientation
TI 251R: Data and Records Management

Related SOPs:

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



Flow Chart 1. Calibration Procedure, including Acceptance Criteria and Troubleshooting Methods



Flow Chart 2. External Test Weight Procedure, including Acceptance Criteria and Troubleshooting Methods