UCD IMPROVE Standard Operating Procedure #251

Sample Handling Lab

Interagency Monitoring of Protected Visual Environments
Air Quality Research Center
University of California, Davis

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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 particulate matter (PM) collection and 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 provided 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 Air Quality Research Center (AQRC) sample handling laboratory in Davis, California.

2. SUMMARY OF THE METHOD

Clean PTFE, nylon, and quartz filters are loaded into cartridges to be sent out to approximately 160 IMPROVE sites. The following are the major steps in the process of handling the filters:

- The PTFE 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 PTFE filters are pre-weighed in environmentally-controlled weighing chambers (MTL AH500) on a Mettler XPR6UD5microbalances. Thereafter the values are recorded in a database.
- Filters are packed with corresponding log sheets and a flashcard, 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 electronic data and accompanying log sheets, processing and preparing the nylon and quartz filters to be shipped for further analysis, weighing the sampled PTFE filters in an environmentally-controlled weighing chambers (MTL AH500) on a Mettler XPR6UD5 balances, and recording the post-weights in a database.

- PTFE filters that have been sampled in a $PM_{2.5}$ module are prepared for elemental and light absorption analysis. PTFE filters sampled in a PM_{10} module are archived.

3. DEFINITIONS

- Gravimetric analysis: determination of particulate concentration based on the difference between pre- and post-weight of each sample.
- Mettler XPR6UD5 microbalance: microbalance with readability of 1 µg and a maximum capacity of 6.1 g.
- 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 25 mm or 37 mm 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 PTFE and quartz filters after they have been loaded into cassettes.
- $PM_{2.5}$: Particulate matter, aerodynamic diameter of 2.5 micrometers or less.
- PM_{10}: Particulate matter, aerodynamic diameter of 10 micrometers or less.
- 1A filters: 25 mm PTFE filters (3 µm pore size) that are sampled in modules that collect $PM_{2.5}$.
- 2B filters: 37 mm Nylon filters that are sampled in modules that collect $PM_{2.5}$.
- 3C filters: 25 mm quartz filters that are sampled in modules that collect $PM_{2.5}$.
- 4D filters: 25 mm PTFE filters (3 µm pore size) that are sampled in modules that collect PM_{10}.
- Cartridge Preparation station: Station at which 1A and 4D cartridges are cleaned, all cartridges are labeled, and 2B and 3C filters are loaded.
- Pre-Sample Weigh In: Station at which 1A and 4D PTFE filters are pre-weighed in an environmentally-controlled weighing chamber on a microbalance and loaded into cassettes.
- Quality Control (QC) 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 2B and 3C filters transferred into Petri dishes, then the 2B and 3C cartridges are cleaned.
- Post-weigh Chamber Prep: Station at which 1A and 4D filters are unloaded into carriers, 1A petri dishes are labeled, and filters are loaded into the environmentally controlled chambers.
- Post-Sample Weigh In: Station at which 1A and 4D filters are post-weighed in an environmentally-controlled weighing chamber and placed into containers for further analysis or archiving.

- Field blank (FB): a filter of any of the three substrates (quartz, nylon, PTFE 1A or 4D) that is sent out into the field but is not sampled.
- Neckties: thin stickers that have the module letter (1A, 2B, 3C, 4D, 5X) 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.
- Terminal-status 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.
- SW: status that stands for swapped sample dates, analyzed as normal.
- SP: status that stands for swapped sample dates, analyzed as normal.
- TO: status that stands for timing outside normal bounds, analyzed as normal.
- PC: status that implies the filter has possible contamination.

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, lab coats and safety glasses must be worn 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, visit the environment, health, and safety (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, safety glasses, 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. PTFE filters are weighed in the MTL chambers with strict environmental control, with temperatures set to 21.5 ºC +/- 1 º and RH set to 39% +/- 1.5%.

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 into the proper container 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 special care must 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. Laboratory wipes that have been used to clean quartz cassette bottoms are not to clean cassettes from any other filter type. Loose screens used in quartz cartridges are also cleaned and stored separately from those used in PTFE cartridges.

Special care must be used when processing and loading PTFE filters. Because the PTFE filters are sampled in either a $PM_{2.5}$ module (1A filters) or a PM_{10} module (4D filters), it is important to ensure that the correct weight is recorded for each PTFE 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 PTFE 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

The sample handling laboratory's personnel consists of laboratory supervisor, laboratory

assistants, and laboratory student assistants. All laboratory personnel perform under the general supervision of the laboratory manager.

All personnel employed in the sample handling laboratory must obtain extensive training in sample handling. They 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 assistant will:

- Oversee and train new lab assistants 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 PTFE filters with reagent grade ethanol
- Inventory and deliver 2B and 3C filters for analysis outside of the lab
- Inventory and deliver 1A filters for analysis outside of the lab
- Receive reanalysis requests from the data validation group

The laboratory assistants and student laboratory assistants will:

- Pre-weigh and individually identify filters for use at IMPROVE aerosol sampling sites
- Load filters into sampling cassettes
- QC 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 1A PTFE filters for XRF analysis into labeled petri dishes
- Process 4D PTFE filters into slides for storage
- Clean cassette bottoms, cartridge plates, and 37 mm screens thoroughly with laboratory wipes and reagent grade alcohol

Training in the IMPROVE sample handling laboratory follows a general pattern. New laboratory assistants and student 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 mastered, 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 an MTL AH500 automated weighing system with a Mettler XPR6UD5 microbalances to do the pre- and post-weighing of the PTFE filters. Documentation and technical specs are located in the Mettler XPR6UD5 User Manual, which is kept in the sample handling laboratory at all times.

Equipment and materials required for filter handling are listed below.

8.1 Filter and Cassette Requirements

- Quality tested and approved stretched PTFE 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)
  - 25 mm and 37 mm cassette bottoms
  - Cassette tops for 25 mm filters
  - Cassette tops with fixed screens for 37 mm filters
  - Clean 25 mm loose screens for PTFE and quartz cassettes
  - C-clips
- Red, yellow, green, blue, and orange dot stickers for cartridge plate labeling
- U-Line 8x10" 6 Mil re-closable bags
- Red, yellow, green, blue, and orange neckties for cassette tops
- Forceps, stainless steel
- Red protective caps for filter cassettes
- Assorted stickers for labeling boxes, bags, etc.
- Filter-slide mounts, 18 x 24, 2 mm
- Labeling supplies (Sharpies and highlighters, assorted colors)
- Blue site-specific shipping boxes, 3 sizes
- Reagent grade alcohol
- Laboratory wipes
- Memory Cards (Compact flashcards or SD cards)
- Small re-closable bags or plastic case for memory cards
- Log sheet paper
- Printer

8.2 Filter Weighing Equipment

- 1 computer with Lab Application and network connections
- 1 Mettler XP6 microbalances, sensitive to ± 1 μg
- 1 weighing table
- 2 filter-scanning matrix code reader
- 2 Mettler XPR6UD6 microbalances, sensitive to ± 1 μg
- 2 MTL AH500 weighing chambers
- Polonium strip ionization units
- 2 sets of stainless steel test weights, 100 mg, 200 mg, and 400 mg
- Forceps, stainless steel with ceramic tips

8.3 Exposed Filter Processing Equipment

- Computer for running Lab Application
- Stainless steel blunt tipped forceps
- Stainless steel forceps with ceramic tips
- Petri dishes for ion and carbon analysis filters
- Reagent grade ethanol
- Laboratory wipes
- Container for used loose quartz screens
- 3 numbered, 50-position petri dish trays (1 for quartz filters, 1 for nylon filters, 1 for terminal-status filters)
- Petri dish shipping trays for quartz and nylon filters
- Shipping boxes (12” cubed for quartz and 12” x 12” x 10” for nylon)
- Blue Ice cooler packs (for quartz shipments)
- Nitrile gloves
- Arbor presses
- Slides for PTFE filters
- Slide stickers
- Numbered, 50-position Petri dish trays for 1A PTFE filters
- Labeled slide tray per site for 4D PTFE filters (1 per site, per quarter)
- Labeled slide trays for 4D PTFE field blanks (2 per quarter)
- Petri dishes
- Container for used PTFE screens
- Temperature and RH probe

8.4 Blue Box Shipping Equipment

- Computer with Internet access and Lab Application
- Thermal printer
- UPS label rolls
- UPS pouches
- Blue boxes
- Box top 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 the AQRC Drew Avenue laboratory, offsite of the main UCD campus. Standard Operating Procedure #201 (*UCD SOP #201: Sampler Maintenance by Site Operators*) covers field operations used by the site operators.

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

1. Purchase and Preparation of the Filters

PTFE 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 251B.

2. Balance Calibration and Reference Weights

Two MTL AH500 automated weighing chambers, two Mettler Toledo XPR6UD microbalances, and one Mettler Toledo XPR6 microbalance are employed for gravimetric analysis of IMPROVE samples. The microbalances were certified upon initial installation by a Mettler technician and are 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 balance in the sample handling laboratory is calibrated in the morning and checked with reference weights. In the environmentally controlled MTL chambers; calibration, test weights, and reference filters are completed before any of the weighing for the network filters can begin. The balance must meet certain criteria during calibration 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 251A.

3. 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 251S, “Box Cycles and Cartridge Orientation.”

1A and 4D cartridges and cassette bottoms are cleaned at this station. Memory cards 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 251R, “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 refer to TI 251F.

4. Pre-Measurement of the Gravimetric Mass and Loading of PTFE Filters into Cassettes

The pre-weighing of PTFE is done at this station using the Lab Application. 1A and 4D 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 251J.

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

The Quality Check (QC) 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 or USPS labels are printed 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 251K and TI 251L.

6. 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. Memory cards 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.

Memory cards and log sheets are given to lab assistants, who first download and review the data from the memory card. Then, lab assistants 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.

7. 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 quartz or nylon filters. The filters are inspected for flaws or damage and placed in labeled Petri dishes. 2B (nylon) and 3C (quartz) cartridges are cleaned and 3C 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.

8. Nylon and Quartz Filter Shipping

Although the 1A and 4D filters are analyzed on-site at Drew Avenue, the 2B and 3C filters are sent offsite for analysis. The techniques used for shipping these filters to the Research

Triangle Institute (2B filters) and the Desert Research Institute (3C filters) are explained in TI 251M.

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

The final step of this process is the post weighing of PTFE 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 assistant. A lab assistant 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 251G and TI 251H.

1A PTFE filters are prepared and stored for future XRF analysis. 4D PTFE filters are placed into containers and stored permanently. Loose screens are placed into a container for later cleaning. For instructions on preparing and storing PTFE filters, see TI 251M. For instructions on how to clean loose screens, see TI 251O.

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 251H.

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 the SQL database.

A web application called IMPROVE Management Site is also important for laboratory functions. It is used to view all information related to the filters.

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

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 3 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 vacuum and wiping down all counter surfaces with reagent grade ethanol and laboratory wipes. One day a week the floor is also cleaned with a wet mop 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 ºC (72 ºF) and stays mostly within a ±3 ºC range.

Filters weighed in MTL's environmentally-controlled chamber equilibrate for a minimum of four hours with temperatures set at 21.5 ºC (+/- 1 ºC) and relative humidity at 39% (+/- 2.5%).

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 adjust 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. Three stainless steel metal reference weights are weighed prior to each weighing period and every four hours during said period. During the measurements, a tare weight, relative humidity, and temperature reading are taken for each XPR6UD5 microbalance. 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 another 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 multiple 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.

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 ± 0.003mg 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.

11.3 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 “Cartridge Preparation,” “Pre-Sample Weigh In,” “Quality Check,” and “Box Shipping” stations and are detailed in the Procedures section of TI 251I, TI 251J, TI251K, TI 251L, and TI 251 M. New PTFE 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. They are also to make sure 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 SQL database for review after further analysis.

12. REFERENCES

EH&S Website: http://safetyservices.ucdavis.edu

Mettler XP6 Microbalance Operations Manual

UCD SOP #201: Sampler Maintenance by Site Operators
UCD SOP #251: Technical Instruction —

- TI 251A: Reference Weights
- TI 251B: Filter Mass Quality Control and Validation
- TI 251C: Filter Inventory and Acceptance
- TI 251D: Box Receiving
- TI 251E: Entering Log Sheets and Simple Problem Diagnosis
- TI 251F: Post-Sample Processing
- TI 251 G: Post-weigh Chamber Prep
- TI 251H: Post-Sample Weigh-In
- TI 251I: Cartridge Preparation Station
- TI 251J: Pre-Sample Weigh-In
- TI 251K: Quality Check Station
- TI 251L: Box Shipping
- TI 251M: BC Filter Shipping
- TI 251N: 1A Tray Checking
- TI 251O: Cleaning Loose Screens
- TI 251P: Labeling and Organizing D-Slides
- TI 251Q: Cleaning Petri Dishes
- TI 251R: General Laboratory Procedures
- TI 251S: Box Cycles and Cartridge Orientation
- TI 251T: Data and Records Management
- TI 251U: Mass Data Validation of Reweigh Request

Related SOPs:

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

Figure 1. Flow chart of calibration procedure, including acceptance criteria and troubleshooting methods.

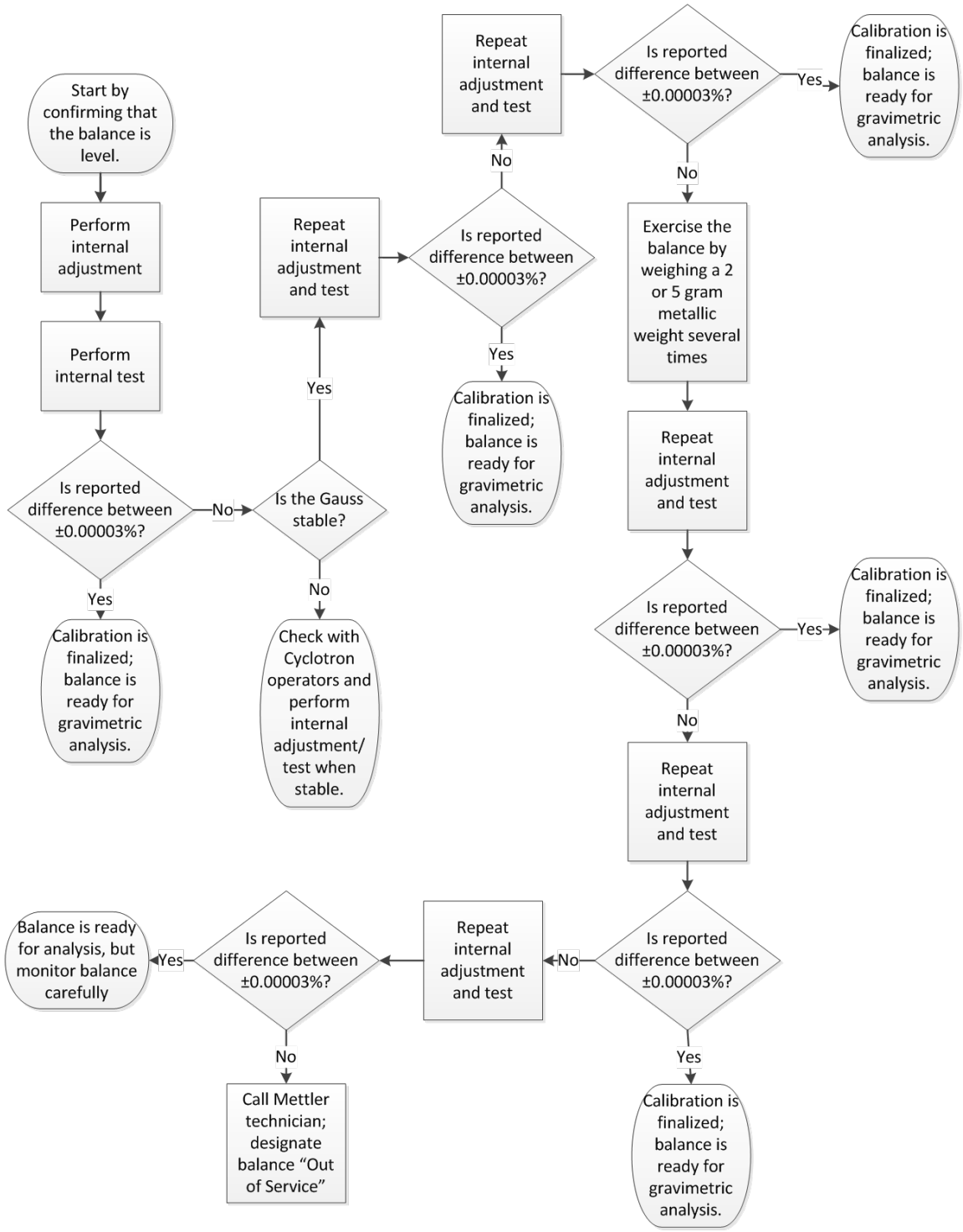

Figure 2. Flow chart of external test weight procedure, including acceptance criteria and troubleshooting methods.