UCD IMPROVE Standard Operating Procedure #251

Sample Handling Lab

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

> July 15, 2022 Version 3.5

	DocuSigned by:		
Prepared By:	Anthony trawamoto	Date:	10/4/2022
	DocuSigned by:		
Reviewed By:	Jason Giacomo	Date:	10/4/2022
	DocuSigned by:		
Approved By:	Marcus Langston	Date:	10/4/2022
••	0A10CFCF79B0452		



DOCUMENT HISTORY

Date Modified	Initials	Section/s Modified	Brief Description of Modifications
06/02/21	SRS	Document History	Document History table added.
05/04/2022	GRM	All	Updated wording and Figure 1. Created Figure 3 and Table 1 to accommodate new procedures.

TABLE OF CONTENTS

1.	Purpose and Applicability
2.	Summary of the Method
3.	Definitions
4.	Health and Safety Warnings7
5.	Cautions7
6.	Interferences
7.	Personnel Qualifications
8.	Equipment and Supplies
8.	1 Filter and Cassette Requirements
8.	2 Filter Weighing Equipment
8.	3 Exposed Filter Processing Equipment
8.	4 Blue Box Shipping Equipment
9.	Procedural Steps
10.	Data and Records Management
11.	Quality Assurance and Quality Control
11	1.1 Cleaning and Maintenance of the Sample Handling Laboratory
11	1.2 Balances
11	1.3 Quality Assurance Procedures in the Laboratory
12.	References

LIST OF FIGURES

Figure 1. Flow chart of calibration procedure	18
Figure 2. Flow chart of external certification weight procedure	19
Figure 3. Flow chart of external certification weight procedure for Manual Balance	20

LIST OF TABLES

Table 1. Definition of Filter Statuses, Terminal and Non-Terminal	6
Table 2. UCD Gravimetric routine QC and criterion.	21

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 and Laboratory Supervisor

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 Mettler XPR6UD5 ultrabalances. 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 Mettler

XPR6UD5 ultrabalances, 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 PM₁₀ 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 ultrabalance: ultrabalance with readability of 0.5 μ g 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.
- PM₁₀: 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 μ m pore size) that are sampled in modules that collect PM₁₀.
- Cartridge Preparation station: Station where all cartridges are labeled, cartridges and cassettes are checked for integrity, and Nylon and Quartz filters are loaded.
- Pre-Sample Weigh In: Station at which PTFE filters are pre-weighed in a environmentally-controlled weighing chambers on ultrabalances and loaded into cassettes.
- Quality Control (QC) station: Station at which loaded cartridges and log sheets are checked 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 nylon and quartz filters transferred into Petri dishes, then the B and C cartridges are cleaned.

- Post-weigh Chamber Prep: Station at which A and D cartridges are cleaned, A and D filters are unloaded into carriers, A petri dishes are labeled, and filters are loaded into the environmentally controlled chambers.
- Post-Sample Weigh In: Station at which A and D 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) that is sent out into the field but is not sampled.
- Neckties: 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.
- 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.

Table 1. Definition of Filter Statuses, Terminal and Non-Terminal.

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 or alternative
	status has not been assigned
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

- Site: physical location for the samplers.
- Sampler: a collection of air quality monitoring equipment. Site and sampler are used almost interchangeably, but sampler refers to the equipment while site refers to the location.
- Sampling date: the day that the sample is collected. IMPROVE equipment is scheduled to sample for 24 hours every three days for each module at the site.
- Module: a singular sampling unit.

- Controller: the computer unit that houses most of the electronics, dictating when the pumps will turn on, and which sampling position will run on each day.
- Ordinal Position: the port that the module is connected to on the Controller. Looking at the front-underside of the controller ports are identified as 1→5 from left to right.
- Module Type: ABC modules use the same physical module and collects PM_{2.5}, but each runs a different filter type. D modules are configured differently and collect PM₁₀.
 - A Modules sample on 25mm PTFE filters, collecting PM_{2.5} particulate.
 - \circ B Modules sample on 37mm nylon filters, collecting PM_{2.5} particulate.
 - C Modules sample on 25mm quartz filters, collecting PM_{2.5} particulate.
 - D Modules sample on 25mm PTFE filters, collecting PM₁₀ particulate.
- Collocated Module: Several IMPROVE sites have an additional module on top of the standard 1A, 2B, 3C, 4D. The 5[ABCD] module is a collocated module. It is a module plugged into the 5th controller port, but is a repeat of one of the other module types, using the same module, sampling on the same filter media, and sampling for the same duration. This serves as quality control and as a long-term study on the repeatability and consistency of our data collection procedures.

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 (<u>http://safetyservices.ucdavis.edu</u>).

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 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 °C +/- 1 °C and RH set to 39% +/- 2%.

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 crosscontamination 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 cassettes.

Special care must be used when processing and loading PTFE filters. Because the gravimetric measurements are taken, it is important to ensure that the correct mass 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 adhere 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 (I, II, and III), 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 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.
- Experience in a physical sciences discipline or equivalent combination of education and experience.

The laboratory assistant II and III (also known as lead lab technician in TIs) will:

- Oversee and train new lab assistants and student assistants
- Review filter readings and flow data.
- Review all log sheets for completeness, and check the validity of the Resolve any inconsistencies on the log sheet or in the samples
- Contact site operators regarding procedural problems
- Oversee filter handling procedures
- Track incoming and outgoing boxes for proper installation and limitation of lost boxes in transit.
- Request supplies, as necessary, for laboratory use
- 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

- Inventory and process terminal status filters
- Receive reanalysis requests from the data validation group
- Execute any swaps or creation of new boxes through the Web Application
- Review all Level 0 mass validation alerts

The laboratory assistants and student laboratory assistants will:

- Clean and maintain the sample handling laboratory
- Pre-weigh and individually identify filters for use at IMPROVE aerosol sampling sites
- Load and Unload the MTL AH500 weighing chambers.
- 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 A PTFE filters for FTIR analysis into labeled petri dishes
- Process D 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 is executed by either a laboratory assistant I, II, or III using the 'IMPROVE Sample Handling Lab Employee Training' document. New laboratory assistants and students are trained on each relevant station. The trainer and trainee sign and date that the trainee observed the trainer in the procedure, the trainer observed the trainee in the procedure, and the trainee was approved to perform procedure unsupervised. Detailed instructions for each station can be found in the TI documents for this SOP.

8. EQUIPMENT AND SUPPLIES

The sample handling laboratory employs the use of an MTL AH500 automated weighing system with a Mettler XPR6UD5 ultrabalances 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)
 - $\circ~~25~mm$ and 37 mm cassette bottoms
 - \circ 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
 - o C-clips
- Red, yellow, green, blue, and orange dot stickers for cartridge plate labeling
- U-Line 8 x 10" and 9 x 12" 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 ethanol
- Laboratory wipes
- Memory Cards (Compact flashcards or SD cards)
- Plastic cases for memory cards
- Log sheet paper
- Printer
- Arbor Press

8.2 Filter Weighing Equipment

- 1 computer with Lab Application and network connections
- 1 Mettler XP6 microbalances, sensitive to $\pm 1 \ \mu g$
- 1 weighing table
- 2 filter-scanning matrix code readers
- 2 Mettler XPR6UD6 microbalances, sensitive to $\pm 0.5 \ \mu g$

Sample Handling UCD SOP #251, Version 3.5 July 15, 2022 Page **12** of **21**

- 2 MTL AH500 weighing chambers
- Polonium ionization units
- Haug ionization unit
- 3 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 D PTFE filters (1 per site, per quarter)
- Labeled slide trays for D PTFE field blanks (2 per quarter)
- Petri dishes
- Container for used PTFE screens
- Temperature and RH probes external and internal to MTL AH500 chambers

8.4 Blue Box Shipping Equipment

- Computer with Internet access and Lab Application
- Thermal printer
- UPS label rolls
- UPS/USPS pouches
- USPS tracking barcode stickers
- Blue boxes

Sample Handling UCD SOP #251, Version 3.5 July 15, 2022 Page **13** of **21**

- Box top stickers
- Box labels

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 251C.

2. Balance Calibration and Reference Weights

Two MTL AH500 automated weighing chambers, two Mettler Toledo XPR6UD ultrabalances, and one Mettler Toledo XPR6 microbalance are employed for gravimetric analysis of IMPROVE samples. The microbalances were certified upon initial installation by a Mettler Toledo 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 electronic certifications are kept in the laboratory drive.

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, when in use, and checked with reference weights. In the environmentally controlled MTL chambers; balance adjustments, and test weights measurements, are completed before any of the weighing for the network filters can begin. The balance must meet certain criteria during adjustment and test weight measurements 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."

Flash cards are 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. 'Dummy' positions are checked. For specific, step-by-step instructions for the procedures at this station refer to TI 251I 'Cartridge Preparation Station'

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

The pre-weighing of PTFE filters is done at this station using the Lab Application. A and D filters are weighed and the values and filter codes 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. Flash 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, whoreview the electronic flow data. Then, lab assistants enter the log sheet data into the system. 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. 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.

8. Nylon and Quartz Filter Shipping

Although the A and D filters are analyzed on-site at Drew Avenue, the B and C filters are sent offsite for analysis. The techniques used for shipping these filters to the Research Triangle Institute (B filters) and the Desert Research Institute (C 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 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 251G and TI 251H.

A PTFE filters are prepared and stored for future XRF analysis. D 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 251N. 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. The room is cleaned bi-weekly by janitorial staff. 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 lab. The temperature is set at or near 22 °C (72 °F) and stays mostly within $a \pm 3$ °C range.

Filters weighed in MTL's environmentally-controlled chamber equilibrate for a minimum of four hours with temperatures set at 21.5 °C (+/- 1 °C) and relative humidity at 39% (+/- 2%).

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 $1\mu g$ or ultrabalance having a readability of 0.5 μg . Laboratory quality control checks include weekly intercomparison weighing of lab blanks and test weights, replicate weighing of select filters, and daily weighing of test weights. For more information on these, please see TI 251A.

11.3 Quality Assurance Procedures in the Laboratory

Multiple steps are taken to make sure that samples are loaded 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, and TI 251L. 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.

Sample Handling UCD SOP #251, Version 3.5 July 15, 2022 Page **17** of **21**

The quality control system is designed to provide confidence in the reported gravimetric values of the $PM_{2.5}$ and PM_{10} aerosol samples collected on PTFE filters. There are a variety of factors that could impact the accuracy of the instrument calibrations or contribute to contamination of the clean or sampled filters. The objective is to provide confidence that instrumentation is in control and provides notice when it is not. The quality control procedures are described in TI 251A.

QC procedures are used to monitor instrument performance daily and PTFE filter behavior weekly. Daily operation is monitored by weighing three external stainless-steel reference weights at the beginning of each weighing procedure, every four hours thereafter while the MTL chamber is in use, and at the end of each weighing procedure. Relative humidity and temperature values are recorded in near-real-time on the MTL database. The quality control criterion is summarized in the table below.

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 251C: Filter Inventory and Acceptance
- TI 251D: Box Receiving
- TI 251E: Entering Log Sheets and Simple Problem Diagnosis
- TI 251F: Post-Sample Processing
- TI 251G: 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: A Tray Checking
- TI 2510: 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
- TI 251V: Replacement Filters and Swaps

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 certification weight procedure for MTL AH500 units, including troubleshooting methods.



Sample Handling UCD SOP #251, Version 3.5 July 15, 2022 Page **20** of **21**

Figure 3. Flow chart of external certification weight procedure for Manual Balance, including criteria and troubleshooting methods.



Sample Handling UCD SOP #251, Version 3.5 July 15, 2022 Page **21** of **21**

Table 2. UCD Gravimetric routine QC and criterion.

Measurement	Frequency	Criterion
100mg metal reference weight	Daily, when gravimetric analysis is conducted	+/- 0.003mg of certified weight
200mg metal reference weight	Daily, when gravimetric analysis is conducted	+/- 0.003mg of certified weight
400mg metal reference weight	Daily, when gravimetric analysis is conducted	+/- 0.005mg of certified weight
Temperature	Daily, when gravimetric analysis is conducted	21.5 °C +/- 1 °C
Relative Humidity	Daily, when gravimetric analysis is conducted	39% +/- 2%.