Section 1 Filter Weighing Procedure



General summary: These are the instructions applicable to pre and post weighing of Teflon and Nuclepore filters. Steps 2 (on-site sampling) and 3 (unloading cartridge) must be completed before post-weighing filters.

All weighing is done in the HERC cleanroom (LSRI building). While in cleanroom, please log hour time in the logbook provided. We are charged a \$10/hour fee for its use and therefore require evidence of time spent.

Pre-Filter Weighing (To do upon entry of clean room)

- 1. Put on a clean pair of socks, if not already on, and step on sticky mats to ensure there is no dirt or dust on feet. Use lint brush to pull any lint of shirt, sweater and any other articles of clothing that will be reaching over workspace. Put on a pair of nitrile gloves.
- 2. Replace petri dish lids of filters that were previously left out to equilibrate. Stack in a convenient location before moving to next steps.

3. Spray anti-static spray solution on large Kim-wipe and use to wipe down tables, scale, computer and any other workspace or

instrument you will be using.

- Do not spray solution directly onto any of the instruments, use a Kim-wipe moistened with the antistatic solution.
- Ensure to wipe down areas which are closer to work before areas that are less likely to become in contact with your work (ie. Wipe down marble table and scale before the further away wooden table).
- 4. Ensure balance is level by the indicator bubble on the top of the balance. If the bubble is not in the indicator circle, use the leveling wheels at the **black circle as shown** base of the instrument to level it.



Figure 1: bubble should be inside

5. Turn on Ionizing Blower by moving the black switch to the right. A red light will flash when it is on.

- 6. Place a small Kim-wipe on marble table where it will be convenient for the forceps to rest (ie in front of scale display).
- 7. Spray anti-static solution onto small Kim-wipe and thoroughly wipe precision forceps, being careful not to get Kim-wipe stuck on point of forceps.
- 8. Open Weighing document on computer (**Dropbox**: HERC/Filter Masses/Masses for all locations) and record the date, temperature and the weigher's name. Be sure to save the document with a unique name.

Microbalance Calibration

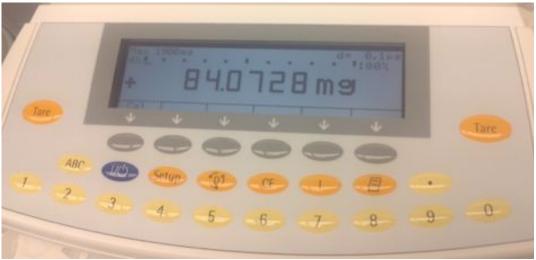


Figure 2: Example of scale display.

- 1. On the balance, press 202122 and then press 'setup'. Next, press '<<' and then 'S CAL'.
- 2. Press 'select' and continue to press 'select' until 'set preload' appears at bottom left of display. Press 'start'. A 'C' will appear in center of display, wait until this goes away and a weight comes onto screen (ie 0.0000mg). Now the preload procedure is finished.
- 3. Press 'select' until 'SCAL: Internal adjustment' appears, press 'start'. Wait until this procedure is finished and again a weight comes up on display.
- 4. Then press 'select' until 'SCAL: internal linearization' appears, press 'start'. Wait until the procedure is finished and a weight comes up on display.
- 5. Press the CF button, wait until a weight comes up on screen.

6. At this point the weight should be 0.0000mg. if not, consult scale manual or see the lab tech.

NOTE: make sure there is nothing on the scale and that the table is not bumped during calibration. Do not rest hands on the table or move things around on table while calibrating. Please see lab tech if any issues arise.

Weighing the Calibration Weights and the Lab Blank Standards

- 1. Ensure all surfaces and forceps have air-dried from the anti-static cleaning procedure.
- 2. Remove 200mg weight, 1mg weight and plastic forceps from the grey and red box on wooden table.
- 3. Begin by weighing the 200mg calibration weight
 - Pick up the ridge side of the weight with the clear forceps.



Figure 3: Grabbing a calibration weight



Figure 4: Example of placing item of scale

- Place approximately 15cm below the ionizing blower for approximately 3 seconds
- Open scale, place in center of pedestal and close lid.
- Allow weight measurement on display to remain constant for 20 seconds, before recording that final weight on spreadsheet on computer.

- 4. Remove weight from the scale and replace in its holder, be sure to close the lid.
 - At this point, there should be nothing on pedestal inside the microbalance
 - Wait until the display has remained at a constant measurement for at least 20 seconds.
 - If that constant measurement is 0.0000mg, proceed to next step.
 - If that constant measurement is not 0.0000mg, press the tare button and wait until the display button reads 0.0000mg before moving to the next step.
- 5. Repeat steps 3 and 4 for the 1 mg calibration weight.
- 6. To ensure the weight found is reliable, repeat the measurement two more times for each calibration weight.
 - The average of the three measurements for each calibration should be within +/- 0.0030 mg of the expected value with a standard deviation being less 0.0020 mg.
 - If not within these guidelines, try recalibrating the scale by pressing the start button underneath the 'SCAL' and repeat measurements.
 - If still not within these expectations, there is probably a problem with the microbalance and should consult the microbalance instruction manual.
- 7. Replace the calibration weights and the clear forceps to the holder.
- 8. Next weigh the lab blank standards.
 - Begin by using the precision forceps to pick up the first lab blank standard from the petri dish by grabbing the edge of the filter with the tips of the forceps.
 - Place filter approximately 15cm below the ionizing blower for approximately 3 seconds.
 - Open scale, place in center of pedestal and close lid.
 - Allow weight measurement on display to remain constant for 20 seconds, before recording that final weight on spreadsheet on computer.
- 9. Remove filter from pedestal, again by grabbing the edge of the filter with the precision forceps and replace in petri dish being careful not to accidentally grab the pedestal.
 - At this point, there should be nothing on pedestal inside the microbalance.
 - Wait until the display has remained at a constant measurement for at least 20 seconds.
 - If that constant measurement is 0.0000mg, proceed to next step.
 - If that constant measurement is not 0.0000mg, press the 'tare' button and wait until the display button reads 0.0000mg before moving to the next step.
- 10. Repeat steps 8 and 9 for all available lab blank standards.

- 11. Confirm the weights found for these lab blank standards are reliable by repeating measurements in a random order for a total of three measurements for each filter.
 - Compare the average of the measurements for each filter to previous measurements.
 - The measurements are acceptable if the average for each filter is within +/- $10 \mu g$ of the previously found averages and more importantly that the standard deviations are no larger than $10 \mu g$ and ideally below $5 \mu g$.
 - If these requirements are not met, ensure the microbalance is calibrated correctly and working properly before repeating steps 9-11.

NOTE: Please see lab tech if measurements are not stabilizing, if they are repeatedly inconsistent or if any other troubles arise.

Weighing the Filters

- 1. Make sure the filters to be weighed have been equilibrated (left exposed to clean room environment) for at least 24 hours.
- 2. Begin by using the precision forceps to pick up the first filter from the petri dish by grabbing the edge of the filter with the tips of the forceps.



Figure 5: example of holding the filter by its edge

- 3. Place filter approximately 15cm below the ionizing blower for approximately 3 seconds.
- 4. Open scale, place in center of pedestal and close lid.



Figure 6: example of filter being placed on middle of pedestal.

- 5. Allow weight measurement on display to remain constant for 20 seconds, before recording that final weight on spreadsheet on computer.
- 6. Remove filter from pedestal, again by grabbing the edge of the filter with the precision forceps and replace in petri dish. Be careful not to grab the pedestal with the forceps.
- 7. At this point, there should be nothing on pedestal inside the microbalance.
 - Wait until the display has remained at a constant measurement for at least 20 seconds.
 - If that constant measurement is 0.0000mg, proceed to next step.
 - If that constant measurement is not 0.0000mg, press the 'tare' button and wait until the display button reads 0.0000mg before moving to the next step.
- 8. Repeat steps 2-7 for all filters to be weighed.
- 9. Confirm the weights found for these filters are reliable by repeating measurements in a random order for a total of three measurements for each filter.
 - **For filters**: The measurements are acceptable if the standard deviations are no larger than 0.0100mg and ideally below 0.0050mg. If these requirements are not met, ensure the microbalance is calibrated correctly and working properly before repeating steps 2-9.
 - **For field blanks**: The measurements are acceptable if the standard deviations are no larger than 0.0150mg. The field blanks should not increase or decrease more than 0.0300mg from the pre-sampled weights. If these

requirements are not met, ensure the microbalance is calibrated correctly and working properly before repeating steps 2-9.

NOTE 1: Field blanks are only specified after a sampling process. Before sampling, field blanks and other filters are both treated in the same way.

NOTE 2: Please see lab tech if measurements are not stabilizing, if they are repeatedly inconsistent or if any other troubles arise.

TIP: It is helpful to weigh filters in groups of four at a time. This way, you can faster observe whether the measurements are appropriate or if the microbalance should be recalibrated or adjusted.

After Weighing Event

- 1. Replace green forceps in holder, and put weighed filters back in appropriate tray and cover.
- 2. Take the next filters to be weighed out of the tray and lay out one-by-one on table. Open each petri dish lid of these filters so that the cut out notch on lip is exposed to the air.
- 3. Leave these filters exposed to the clean room conditions for at least 24 hours.

More information on filter mass measurements:

Electrostatic & humidity effects, and filter aging see Lawless and Rodes (1999)

Humidity range (35%) and weighing similar to Yang et al. (2011)

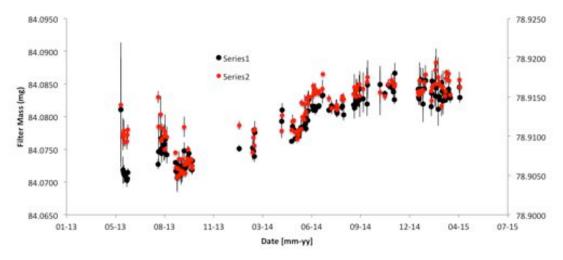


Figure 7: Two filters (Teflon) weighted repeatedly at HERC for two years

Logging filter mass data

Pre and post-weighed data are recorded in

Dropbox: SPARTAN_DB/HERC/Filter Masses/Filter blanks and calibration_[date].xlsx

And

Dropbox: SPARTAN_DB/HERC/Filter Masses/Masses for all locations_[date].xlsx

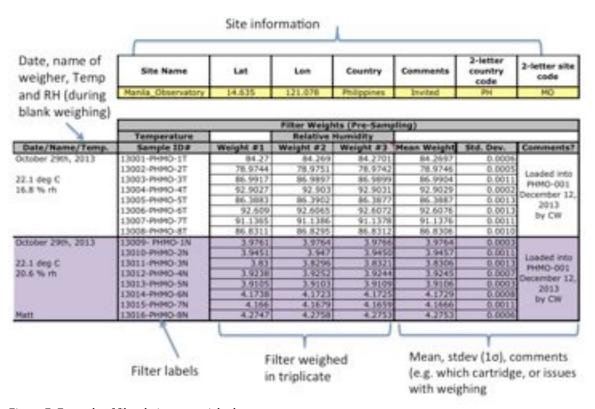


Figure 7: Example of filter being preweighed.

There is one spreadsheet tab per site. Weights are recorded in triplicate both for pre and post-weighing, hence each filter is weighed 6 times.

- Teflon filters are SKC brand (part No. 225-2726 FILTER-PTFE, 25MM, 100PK).
 About \$500 per pack, ordered via www.conceptcontrols.com
- Nuclepore filters are SPI Supplies brand (part no. E2508G-MB, Grease Coated Polycarbonate Membrane Filters 8um, 25mm 100PK). About \$550 per pack, special ordered (give 4-6 months notice) via www.2spi.com.
- Petri dishes are purchased from New Star Environmental (NS-1504700) \$45 per box of 100 via http://www.newstarenvironmental.com/product/air-sampling-filter-storage-shipping-dish

REFERENCES

Lawless, P. A. and Rodes, C. E.: Maximizing Data Quality in the Gravimetric Analysis of Personal Exposure Sample Filters, J. Air Waste Manage. Assoc., 49(9), 1039–1049, doi:10.1080/10473289.1999.10463877, 1999.

Yang, F., Tan, J., Zhao, Q., Du, Z., He, K., Ma, Y., Duan, F. and Chen, G.: Characteristics of PM2.5 speciation in representative megacities and across China, Atmos. Chem. Phys., 11(11), 5207–5219, doi:10.5194/acp-11-5207-2011, 2011.