APPENDIX 4

HONG KONG ENVIRONMENTAL PROTECTION DEPARTMENT

STANDARD OPERATING PROCEDURES

FOR

PM2.5 FILTER WEIGHING

Standard Operating Procedures for PM2.5 Filter Preparation, Conditioning & Weighing for the 12-month Chemical Speciation Project (Version 1, Oct 2000)

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APPENDIX

1. INTRODUCTION

The Standard Operating Procedures (SOP) for PM2.5 Filter Preparation, Conditioning and Weighing is to provide a guidance for handling, inspecting, conditioning, weighing and qualitatively controlling the 47mm diameter filters inside the EPD's Balance Laboratory (here called the Laboratory) for the determination of PM2.5 mass concentration in unit of microgram per cubic meter ($\mu g/m^3$). The Laboratory itself has been accredited for PM10 weight measurement by HOKLAS* since 1996, being located at 47/F, Revenue Tower, 5 Gloucester Road, Wan Chai, Hong Kong.

The goal of this speciation project is to monitor the daily concentration of particulates with aerodynamic diameters less than or equal to 2.5 micrometers (μm) at roadside, urban and rural areas in Hong Kong throughout a year, and meanwhile to examine its seasonal variation, as well as identifying the major elements, particularly the organic compound species, through the chemical speciation process. Samples are to be collected on the 47mm filters by a USEPA designated PM2.5 sampler, namely, Partisol-Plus Model 2025 Sequential Air Sampler. This SOP only covers the parts of filter preparation, conditioning and weighing in the Laboratory. The standard operating procedures for the Sampler should be referred to another SOP, namely, "Field Operating Procedures for Partisol-Plus Model 2025 Sequential Air Sampler".

The 47mm Quartz and Teflon filters are selected for sample collection in the project. All virgin quartz filters upon receipt from the local supplier, will have been fired in an oven at 900^{o}C for at least 3 hours to get rid of the background carbon resided on filters before use. Around 5% of the pre-fired quartz filters will then be randomly picked out and examined the carbon residue by a carbon analyzer of thermal optical transmission type as one of the quality control processes. Details of filter specifications are given in Section 2. Quartz filters are analyzed for nitrate and sulfate by ion chromatography (IC), for ammonium by automated colorimetry (AC), for organic and elemental carbon by thermal optical reflectance (TOR) and thermal optical transmission (TOT), for soluble sodium, magnesium and potassium by atomic absorption (AA), and for organic carbon speciation by water soluble extraction and solvent extraction methods. Whereas, Teflon filter should be the type of 46.2 mm polytetrafluoroethylene (PTFE) with a plastic support ring at edge. They are analyzed for elements by Energy Dispersive X-ray Fluorescence (EDXRF), and are used for the determination of mass concentration in accordance with the USEPA's PM2.5 reference method.

* : HOKLAS = The Hong Kong Laboratory Accreditation Scheme operated by the Hong Kong Accreditation Service (HKAS) of the Innovation and Technology Commission of the Government of the Hong Kong Special Administrative Region of China

2. APPARATUS AND MATERIALS

The following apparatus and materials are used to perform the mass weighing activities.

Item	Model No.	Supplier	Quantity	Unit
Microbalance	MT-5	Mettler Toledo	2	No.
100 and 200 mg Standard Weights (Class M1 or above) with independent weight-handling forceps	ME-15930	Mettler Toledo	1	No.
Relative humidity (RH) & temperature sensing and chart recording system	80000-00	Cole-Parmer	1	No.
NIST traceable glass thermometer	-	ICL Cal. Inc.	1	No.
Assmann Psychrometer	5230	Qualimetrics Inc.	1	No.
Refrigerator	N350	Philco	2	No.
Freezer	MIR-553	Sanyo	1	No.
Light box	-	Mega-Pro	1	No.
Powder-free anti-static gloves, M/size	96-233	Oak	10	Bag of 100
210-Polonium strip	2U500	NRD	2	No.
PetriSlide	PD1504700	Millipore	10	Box of 100
Low-lint disposable laboratory wipe	34155	Kimwipes	1	Case of 15 packs
Chart paper & pen	-	-	10	Set
Anti-static laboratory coat	-	China Scientific Ltd.	2	No.
Filter-handling Forceps (SS)	35a-SA	Aven	2	No.
Anti-static zip lock bag , 305x406mm	663-005	RS Components Ltd.	12	10 bags / pack
Anti-static Zip-lock bag , 101x152mm	290-9404	RS Components Ltd.	3	100 bags / pack
Anti-static Zip-lock bag , 152x203mm	290-9410	RS Components Ltd.	10	100 bags / pack
47 mm Teflon Membrane Filter (PTFE) with PMP support ring	R2PJ047	Pall Gelman	6	50 pcs / pack
47 mm Quartz (QMA) Filter *	1851047	Whatman	6	100 pcs / pack
Survey Count Rate Meter	190-489-110 C	Davis	1	No.
Mask	1730	Gerson	12	20 pcs / box

* Deposit area of QMA filters sampled with Partisol-Plus 2025 Sequential Sampler are found to be consistent, 11.98 cm^2

3. BALANCE LABORATORY

3.1 Facilities of Laboratory

3.1.1 The Laboratory itself has been a HOKLAS accredited laboratory for PM10 weight measurement since 1996. It is positively pressurized and 24-hour air-conditioned. An independent air-conditioning system equipped with high-efficiency particulate air (HEPA) filters is specially designed to meet the stringent requirements for PM conditioning and weighing.

3.1.2 The Laboratory is completely isolated from the outside by two entrance doors. When a laboratory staff (LS) enters the Laboratory, the first door need to be closed before the second one is opened. Similarly, when he leaves, the second door need to be closed before the first one is opened. Sticky taps are put on the floor between these two entrances in order to remove loose objects adhered on the bottom of shoes.

3.1.3 A light box and working benches are installed inside the Laboratory for light testing and filter preparation respectively. In addition, a filter conditioning and weighing chamber is specially designed to meet the stringent requirements as stipulated in Section 3.2 below for PM2.5 filter conditioning and weighing.

3.1.4 A microbalance, a RH& temperature sensing and recording system and two filter conditioning cabinets are installed inside the conditioning and weighing chamber. One of the cabinets is specially assigned for pre-sampling filter conditioning while another one for post-sampling filter conditioning. Temperature and RH inside the chamber is continuously monitored and recorded by the electronic sensing and chart recording system which shall be traceable to the primary standards of the Standards and Calibration Laboratory (SCL) of the Innovation and Technology Commission of the Government of the Hong Kong Special Administrative Region of China.

3.2 Environmental Requirements for PM2.5 Filter Conditioning and Weighing

To ensure that the weight measurements for pre-and post-sampling filters will be unbiased and precise, the following environmental requirements for the conditioning and weighing chamber in the Laboratory have been established. Continuous monitoring on these two parameters by an electronic sensing and recording system is required.

i) Temperature : Prior to a weighing session, the mean of the 5-minute temperature averages over the preceding 24 hours must be a value in between 20 and 23℃, controlled to ±2℃.

ii) Relative Humidity (RH) : Prior to a weighing session, the mean of the 5-minute relative humidity averages over the preceding 24 hours must be a value in between 30 and 40%, controlled to ±5%.

3.3 Requirements of Microbalance

The following summarizes the requirements of microbalance for assurance of balance stability and sensitivity during and between pre- and post-sampling measurements on the same filter:

i) a readability and repeatability of at least ±1 μg;
ii) operated with the leveling bubble at the level position;
iii) automatically calibrated;
iv) not automatically zeroed (for PM2.5 mass weighing application);
v) manual external verification of working standards according to the manufacturer's instructions;
vi) internally calibrate according to the manufacturer's instructions;
vii) located in controlled environment;
viii) located on a clean, vibration-free surface;
ix) located in an environment free of air pulses of turbulence that might prolong of disrupt efforts to achieve stable weights;
x) the balance's weighing chamber module grounded to universal ground;
xi) left plugged into power and with power on at all times (LCD screen can be left off);
xii) maintained and operated strictly according to the manufacturer's instructions;
xiii) calibrated the balance with the NIST standards twice a year by an independent audit team.

3.4 Requirements of Electrostatic Charge Neutralization

i) A small amount of polonium (^{210}Po) anti-static (500 microcuries) strips should be used to reduce electrostatic buildup in the microbalance chamber and on individual filters by filter neutralization. They will neutralize electrostatic charges on items brought within an inch of them.
ii) The ^{210}Po strip is attached to the Positioner by means of a leaf spring which has two small projections, one at each end. Flex this spring so that the projections can be engaged in the holes at each end of the ^{210}Po strip. The grid of the ^{210}Po strip should face outward.
iii) Direct the radiation emanating through the grid of the ^{210}Po strip by bending the flexible arm.
iv) For most effective results the grid of the ^{210}Po strip should be from ½" to ¾" away from the surface to be neutralized. The alpha particles produced by the ^{210}Po element travel a maximum distance of 1⅜" in still air, and the greatest ionization occurs at the end of their path. Air movements must be considered, so it may be necessary to try the strip at several distances from the surface to be neutralized in order to determine a location for maximum efficiency under prevailing conditions
v) Change the anti-static strips every 6 months and return the old strips to the manufacturer for safety disposal.

Safety Precautions for handling ^{210}Po strips :

Precaution 1 : Do NOT directly touch the surface of ^{210}Po strip by bare hands or skin. Put on anti-static gloves and use forceps to insert filters near the strips for neutralizing.

Precaution 2 : If the ^{210}Po strip is accidentally handled or touched, wash hands thoroughly as soon as possible.

Precaution 3 : When the strips are not in use, lock them inside an air-sealed, rigid box with a warning label sticking on the box surfaces; and/or

Precaution 4 : Use a radiation detector to monitor the exposure level to laboratory staff. According to the Radiation Board's guideline [Reference 6], the dose limit for a normal person and a pregnant female should be limited to 20 mSv and 1 mSv respectively in a calendar year.

Precaution 5 : Safely pack and return the expired strips to the manufacturer/suppler for safety disposal.

4. FILTER PREPARATION

4.1 Filter Handling

The following procedures provide a general guidance for performing work inside the Laboratory without contaminating filters:

4.1.1 Avoid equilibrating filters from refrigerator in the Filter Balance Laboratory at all times as it may interfere the stability of temperature and relative humidity of the Laboratory. Allow filters from the refrigerator to equilibrate to around 25^{o}C in a separate air-conditioned room for 12 to 24 hours before weighing. Filters should not be removed from the PetriSlides/cassette magazine and/or the air-sealed, anti-static Zip-lock bag during this period.

4.1.2 Thoroughly clean up the surface of working bench in the Laboratory with low-lint, disposable laboratory wipes. Place new clean wipes on the bench surface where filters handling will be proceeded.

4.1.3 Wear powder free anti-static gloves, mask and laboratory coat whenever handling filters. Gloves that are packed in a box can carry an electrostatic charge. Discharge this charge by touching a good electrical ground with the back of the hand after putting on the gloves.

4.1.4 Use clean, smooth, non-serrated forceps. Mark the filter-handling forceps to distinguish it from the forceps used to handle the reference standard weights.

4.1.5 Clean the forceps with low lint disposable laboratory wipes before handling filter.

4.1.6 Carefully handle the filters with the filter-handling forceps, holding the support ring (for Teflon filter), rather than the filter media.

4.1.7 Keep forceps in a clean Zip-lock bag when not in use.

4.1.8 Clean the forceps with low lint disposable laboratory wipes if they ever touch the filter material of an exposed filter to avoid cross-contamination of filters.

4.1.9 If filters come into contact with foreign objects and they become contaminated, they must be either flagged (if the contamination occurred during post-sampling activities) or rejected from further sampling activities (if contamination occurred during pre-sampling activities).

4.1.10 Once a filter is inspected and accepted, it should only come in contact with the conditioned PetriSlide, forceps, anti-static strip, or weighing pan until a pre-weight is established.

4.1.11 Once a pre-sampling weight is established, the filter should only come in contact with clean forceps, PetriSlide, and sampling cassette until it is inspected before sampling.

4.1.12 Once the filter has been inspected after sampling, it should only come in contact with the conditioned PetriSlide, forceps, anti-static strips, or weighing pan.

4.2 Filter Inventory

4.2.1 The shipment should contain a number of filter boxes, each box containing 50 filters for Teflon or 100 filters for Quartz. In most cases, the filter box will arrive shrink-wrapped. Before opening the boxes, the LS should inventory the filter boxes.

4.2.2 The lot number of the filter box is labeled on the shrink-wrap of each filter box. Do not open a filter box until the lot numbers and the box numbers are recorded as follows:

(i) Take a Filter Inventory / Inspection Form INV-1.

(ii) Take all filter boxes from the shipment. Locate the lot number on the Zip-lock shrink-wrap.

(iii) Group the boxes by lot number.

(iv) For each lot number, group the filters consecutively by box number.

(v) Fill in Form INV-1 and keep in file.

4.3 Filter Inspection

Filter will be inspected when the LS gets ready to put individual filters out of conditioning. Inspection will only occur in the conditioning environment of the Laboratory. Make sure the quartz filters have been pre-fired and examined for the carbon content by the Government Laboratory before inspection.

4.3.1 Put on a laboratory coat and new pair of anti-static gloves.

4.3.2 Select the appropriate Filter Inventory / Inspection Form INV-1.

4.3.3 Select the next filter box, based on the first-in / first-out sequence of acceptable filter lots, on Form INV-1 and open it.

4.3.4 With clean filter forceps, select a filter.

4.3.5 Visually inspect the filter for the following specific filter defects:

i) Pinhole – a small hole appearing as a distinct and obvious bright point of light when examined over the light table. If light appears turn filter over, if light still appears then a pinhole is identified.

ii) Separation of ring – any separation or lack of seal between the filter border reinforcing the ring.

iii) Chaff or flashing – any extra material on the reinforcing polyolefin ring, if any, or on the heat seal area that would prevent an airtight seal during sampling.

iv) Loose material – any extra loose material or dirt particles on the filter.

v) Discoloration – any obvious discoloration that might be evidence of contamination.

vi) Filter non-uniformity – any obvious visible non-uniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity or density across the face of the filter.

vii) Other – a filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

4.3.6 If any of these defects are discovered on the filter, reject the filter and place it in a reject pile (not in a PetriSlide).

4.3.7 If the filter is acceptable, put it in a PetriSlide and close the slide. It will undergo further conditioning.

4.3.8 Repeat Steps 4.3.4 - 4.3.6 until a filter box is completely emptied. Count the number of filters accepted from the filter box. Add this value, the date, and the LS's initials to Form INV-1.

4.3.9 Repeat Steps 4.3.3 - 4.3.8 for another filter box.

At the end of the inspection process, place the rejected filters into a reject envelope. Record the information from the shrink-wrap onto a label along with the LS's initials and the date, and put the label on the envelope. This label will facilitate later evaluation of the rejection's cause and the details, if necessary. If the rejection rate is greater than 15%, contact the Local Supplier/ Dealer to verify.

4.4 Sample Labeling System

A sample labeling system should be created for identifying the sample's nature consistently and distinguishing a sample from the others. Each sample must be allocated a unique Filter Identify (ID) and Sample ID. Stick adequate labels onto the lid and the flat part of the base of a PetriSlide to identify each filter.

The following labeling system is hence developed, reporting details of Site ID, Filter Lot Number, Filter ID, Weighing Batch Number and Sample ID of a unique sample.

4.4.1 Site ID :

TW – Tsuen Wan Air Quality Monitoring Station (Urban Station)

MK – Mong Kok Roadside Air Quality Monitoring Station (Roadside Station)

RU – Rural Hok Tsui Station (Rural Station)

4.4.2 Filter Lot Number :

A filter lot is defined as a single shipment of filters from a filter manufacturer or another source of filters.

i) Lot no. for QMA filter is allocated as
Q001 for the 1st lot, Q002 for the 2nd lot, etc. where Q = Quartz filter

ii) Lot no. for Teflon filter is allocated as
T001 for the 1st lot, T002 for the 2nd lot, etc. where T = Teflon filter

4.4.3 Filter ID :

i) For quartz filter, Filter ID is presented by QXXX YYYY where X & Y are numerical numbers and Q represents Whatman quartz filter. The first 3 digits immediately after Q (i.e. XXX) is the filter lot no. while the last 4 digits (i.e. YYYY) show the unique sequence no. for that particular filter.

e.g. Q001 0020

means Q001 = the 1st lot of quartz filter shipment
0020 = the 20th filter in the Quartz Filter Shipment Lot No. 001

ii) For Teflon filter :

(a) Similarly, filter ID of Teflon filter manufactured by PALL Gelman Science is presented as TXXX YYY where T = Gelman Teflon Filter

e.g. T003 0010

means T003 = the 3rd lot of Teflon filter shipment
0010 = the 10th filter in Teflon Filter Shipment Lot No. 003

(b) Filter ID of Whatman Teflon filters simply follows the ID provided by the manufacturer.

e.g. P 0001960

Filter lot no. is not shown in Filter ID., but will be shown on the data sheet.
The first digit shows the filter medium code, i.e. P.
The second to eighth digits show the unique sequence no. for Teflon filters, i.e. 0001960.

4.4.4 Weighing Batch Number :

The batch no. for each weighing session is allocated this way :

e.g. A001 for the first weighing session,
A002 for the second weighing session, etc.

4.4.5 Sample ID :

Each sample should be allocated a Sample ID as follows:

e.g. TW 00 12 31 S F01 Q

(from left to right)

i) The first two alphabets = Site ID where

TW = Tsuen Wan Air Quality Monitoring Station (Urban)

MK = Mong Kok Roadside Air Quality Monitoring Station (Roadside)

RU = Rural Hok Tsui Station (Rural)

ii) The 3rd & 4th places = Year

e.g. 00 = 2000, 01 = 2001, etc.

iii) The 5th & 6th places = Month

e.g. 01 = January, 02 = February, 03 = March, etc.

iv) The 7th & 8th places = Day

e.g. 01 = the first day of the month, 02 = the second day of the month, etc.

v) The 9th place = Sample Type where S = Ambient Sample and F = Field Blank

vi) The 10th to 12th places = Equipment Number

e.g., F01, F02, R10, H01, etc.

vii) The 13th place = Filter Medium where Q = Quartz and T = Teflon

In the above example, the Sample ID means that the sample is an ambient sample collecting dusts on a quartz filter by Equipment No. F01 at Tsuen Wan Air Quality Monitoring Station on Dec 31, 2002.

5. STABILITY TESTS

5.1 Balance Stability Test

Prior to the implementation of routine filter activities, establish the laboratory to meet the microbalance requirements mentioned before. The following balance stability test procedures should then be implemented:

5.1.1 Turn on the microbalance and let it warm up for 24 hours.

5.1.2 Select the 5, 100 and 200 mg working standard weights.

5.1.3 Zero and calibrate the microbalance using the “Menu” key according to the microbalance’s operating manual.

5.1.4 Open and close the draft shield of microbalance (circular arrow key) two times to equilibrate the air in the draft shield chamber with the conditioning room.

5.1.5 Using smooth, nonserrated, nonmetallic forceps gently place each working mass

reference standard on the sample pan. Close the draft shield. Wait until the microbalance's display indicates that a stable reading has been obtained (around 20 seconds after stable reading indicated) before recording a gravimetric measurement.

5.1.6 Record the measured value in mg to 3 decimal places in the Laboratory Balance Stability Test Form (LBS-01).

5.1.7 Each hour repeat Steps 5.1.1-5.1.6 until seven measurements of each working mass have been recorded.

5.1.8 Calculate the difference between paired consecutive measurements and the mean difference of all seven and the mean and standard deviation of each standard weight.

5.1.9 For each standard, provide a detection limit by multiplying the standard deviation of each weight by 3 and entering it in the "DL" row.

5.1.10 Provide an overall detection limit by averaging the three detection limits and placing the value in the "Overall Detection Limit" row.

5.1.11 If the mean difference is less than 3 μg for each standard and the overall detection limit is less than 5 μg and the balance achieves a stable weight in 30 seconds, then balance stability has been demonstrated. If not, troubleshoot the microbalance system and the laboratory environment until stability is established.

5.2 Lot Blank Filter Stability Test

The following procedures describe the method to determine the minimum length of time to condition filters that have been opened from their shrink-wrapped boxes for initial exposure to laboratory conditions. **This procedure only needs to be accomplished with each distinct lot of filters.**

5.2.1 Follow the "Filter Inventory" procedures in Section 4.2.

5.2.2 Randomly select 3 filter boxes from the inventory of filter boxes.

5.2.3 From each filter box, randomly select 3 filters. These filters will be identified as **lot blanks**. This will provide for a total of 9 filters.

5.2.4 Inspect these 9 filters as described in Section 4.3.

5.2.5 If the filters are acceptable, place each filter in a PetriSlide and identify each filter by a Filter ID on the flat part of the base and the lid of PetriSlide.

5.2.6 Take a Lot Blank Filter Stability Test Form FST-01 and record each filter.

5.2.7 Allow these nine filters to condition in the Laboratory for 24 hours.

5.2.8 After 24 hours, weigh the filter following the procedures in Section 6.2 every 24 hours for a minimum of 5 consecutive days and until the difference between one weight and the consecutive weight of all 9 filters is less than 15μg. If the test goes longer than 5 days, take another Lot Blank Filter Stability Test Form FST-01 and continue to record the data.

5.2.9 Record the length of time (in days) that it takes each filter to stabilize.

5.2.10 Calculate the average length of time for the 9 filters to stabilize. This average will

provide the LS with an estimate of time that filters will take to achieve a constant weight after opening of box.

Note: It is important to note any trends during this test. Control charting this data can help. Upon weighing, if decreasing weights continuously occur over the 5 days period and longer, even though it may be less than 15µg, this will indicate outgasing which is why conditioning is important. The LS is required to continue this test until this download trend stops or is negligible (5µg difference). If the trend is an increase in weight, it may indicate laboratory contamination, which must be rectified immediately.

5.3 Lot Exposure Blank Filter Stability Test

Section 5.2 establishes the typical length of time that a box of filters, once opened, should be set out into the conditioning environment. These procedures describe the method for determining that a set of filters which will be used for a pre-sampling weighing session have been sufficiently conditioned (stable). The LS can proceed with weighing them for field sampling.

5.3.1 From the Filter Inventory/Inspection Form INV-01, select the next set of filter boxes for individual filter conditioning.

5.3.2 Inspect filters by following the procedures in Section 4.3.

5.3.3 Place the acceptable filters in individual PetriSlides and place the PetriSlides in the conditioning environment. Place the lids ¾ over the slide bottom (i.e. filter locating area) or the lids under the slide bottom.

5.3.4 Group these filters in an area where the LS can identify when these filters were set out.

5.3.5 Allow the filters to condition for the length of time established in Section 5.2 above.

5.3.6 From the set of filters that have been placed out for conditioning, randomly select three filters and identify them as **lot exposure blanks.**

5.3.7 Weigh the lot exposure blank filters and check the weights by following procedures in Section 6.2. Record the initial weight of each blank filter in a Lot Exposure Blank Filter Stability Form FST-02.

5.3.8 Maintain the required temperature and RH of the Laboratory. Measure temperature and RH on a continuous basis during filter weight stability experimentation using electronic instrument which outputs are recorded by a data acquisition system. Calculate the means and ranges of temperature and RH of the conditioning environment over each 24-hour period, and record these values.

5.3.9 Wait 24 hours and then weigh the three lot exposure blanks again. Record the difference in micrograms from the consecutive weights.

5.3.10 Weigh the lot exposure blanks every 24 hours until the average difference between all three weights is 5µg and no filter difference is greater than 15µg. Once this occurs, the filters associated with the lot exposure blanks are ready for weighing.

5.4 Post-sampling Filter Stability Test

The following procedures describe the method for conditioning a batch of exposed / sampled filters that are returned from the sampling sites. Filters may or may not have been stored in a refrigerator.

5.4.1 Follow the filter receiving procedures in Section 7.2.

5.4.2 Condition the exposed filters in the PetriSlide for at least 24 hours in the Laboratory.

5.4.3 After 24 hours, select 3 routine filters.

5.4.4 Select a Post-sampling Routine Filter Stability Test Form FST-03.

5.4.5 Weigh the 3 routine filters and check weights by following the procedures in Section 6. Record the initial weight in FST-03.

5.4.6 Maintain the required temperature and RH of the conditioning and weighing chamber. Measure temperature and RH on a continuous basis during filter weight stability experimentation using electronic instrument which outputs are recorded by a data acquisition system. Calculate the means and ranges of temperature and RH of the conditioning environment over each 24-hour period, and record these values.

5.4.7 Wait a minimum of 8 hours and then weigh the routine samples again. Record the difference in micrograms from the consecutive weights.

5.4.8 Weigh the 3 routine sample filters every 12-24 hours until the difference between two out of the three filters consecutive weights is less than 15μg. Once this occurs, the batch associated with the 3 filters is ready for weighing.

5.4.9 Follow the post-sampling filter weighing procedures in Section 6.4.

6. FILTER CONDITIONING AND WEIGHING

Follow the filter handling procedures in Section 4.1 before implementing the following procedures in Sections 6.1 - 6.4.

6.1 Pre-sampling Filter Conditioning

6.1.1 Particular attention must be paid to Section 4.1.1 prior to conditioning any refrigerated filters.

6.1.2 From the Filter Inventory/Inspection Form INV-01, select a number of filters from the same lot for conditioning.

6.1.3 Inspect the filters as described in Section 4.3.

6.1.4 Place the acceptable filters in individual PetriSlides and place the slides in the pre-sampling conditioning chamber. Place the lids ¾ over the slide bottom plate (i.e. filter holding area) or the lids under the slide bottom plate.

Note : An open-type cabinet should be assigned for conditioning the pre-sampling

filters. Post-sampling filter should NOT be allowed to condition in this cabinet. A separate open-type conditioning cabinet should be assigned for holding the post-sampling filters.

6.1.5 Group and condition these filters in the assigned pre-sampling conditioning cabinet.

6.1.6 Allow the filters to condition in the conditioning and weighing chamber for the length of time established in Section 5.3.

6.1.7 Maintain the required temperature and RH inside the conditioning & weighing chamber. Measure and record the temperature and RH of the chamber on a continuous basis using an electronic instrument to determine the stability of conditioning environment.

6.2 Pre-sampling Filter Weighing

6.2.1 After pre-sampling filter conditioning, calculate the means and standard deviations of the temperature and RH over the preceding 24-hour period.

6.2.2 Verify that the mean temperature and RH in the conditioning environment have remained within the acceptance criteria and that the temperature and RH during the weighing session is controlled to within ±2°C and ±5% respectively. Record these values in the Filter Conditioning and Weighing Logbook. If the conditioning environment had not remained within these specifications, the filters should not be weighed. Take appropriate trouble shooting and corrective action in that event.

6.2.3 If necessary, clean the weighing pan and draft shield of microbalance with lint-free, anti-static, disposable wipes.

6.2.4 Clean the surface near the microbalance with lint-free, anti-static, disposable wipes.

6.2.5 Clean the forceps for handling mass reference standards and the filter forceps with an ethanol-dampened, lint-free cloth with the pre-moistened wipes. Allow the forceps to air-dry after cleaning. Make sure that the forceps are thoroughly dry before use because even a small amount of moisture can cause a significant measurement bias.

Note : Cleaning and air-drying the forceps inside or close to the conditioning and weighing chamber is prohibited.

6.2.6 Leave the microbalance plugged in and the power turned on at all times. This procedure enables the microbalance to be operational at any time and eliminates the need for a preliminary warm up period before analyses can be performed.

6.2.7 Have all equipment clean and ready to prepare filter cassettes: caps, cassettes, stainless steel screen, anti-static zip-lock bags, log sheets, and indelible in markers or labels.

The following procedures describe the method for warming up a microbalance with a load, called “exercising the balance”.

6.2.8 Open and close the microbalance’s draft shield (circular arrow key) two times to equilibrate the air.

6.2.9 Zero and internally calibrate (using the "Menu" key) the microbalance according to the microbalance's operating manual. This may take 1-2 minutes.

6.2.10 Open the draft shield.

6.2.11 Using working standard forceps, gently place the 100 mg working standard weight on the weighing pan of microbalance. Then close the draft shield.

6.2.12 Wait until the symbol "□" at the upper left corner of microbalance's display disappears. That means a stable reading is obtained.

6.2.13 After obtaining a stable reading, wait for another 20 seconds. If the reading remains stable, record the measured value in the Filter Conditioning and Weighing Logbook and go to Step 6.2.15.

6.2.14 If the reading fluctuates within these 20 seconds, wait for next 20 seconds and record the reading again. If after three attempts the microbalance still does not provide a stable reading, take off the working standard weight and repeat Steps 6.2.9 - 6.2.12.

6.2.15 Repeat Steps 6.2.11 – 6.2.14 with the 200 mg working standard weight.

6.2.16 If the verified and measured values of either working standard disagree by more than ±3μg, repeat Steps 6.2.8 – 6.2.15. If the measured values are acceptable, move to Step 6.2.18.

6.2.17 If the measured values still disagree with their corresponding standards by more than ±3μg, halt the routine weighing until the problem has been fixed. Troubleshoot the entire measurement system and take appropriate corrective action. Corrective action may include:

i) conducting the calibration sensitivity test,

ii) calibrating the microbalance using the internal standard of microbalance,

iii) re-certifying the working standard weights against its primary standard weights, and/or asking a service technician to adjust or repair the microbalance. In any case, LS should not attempt to repair the microbalance himself,

iv) checking temperature and RH of the conditioning and weighing chamber with independent standards,

v) calibrating the microbalance using an external laboratory primary standards.

6.2.18 Close the draft shield and allow the microbalance to come to zero. Wait at least 20 seconds to ensure zero is achieved. If not achieved by then, the instrument can be manually zeroed using the "Re-zero" key.

6.2.19 Select a filter for weighing and record its ID as indicated on the lid of PetriSlide in the Filter Conditioning and Weighing Logbook. Indicate the filter type as either "RO" for a routine filter, "LB" for a laboratory blank, "FB" for a field blank, "CO" for a collocated sample, "BD" for a batch duplicate, or "PD" for a duplicate from a previous (day) batch.

6.2.20 Take the filter from its PetriSlide by gently pushing down (with the filter-handling forceps on the outer polyolefin support ring) one side of the filter. This should raise the other end of the filter and with a slight budge, rest the filters edge on the PetriSlide. LS can then access the outer polyolefin support ring with the filter-handling forceps.

6.2.21 Lay each filter, with its support-ring side upward, in the ^{210}Po antistatic strip for 30 to 60 seconds immediately prior to weighing for neutralizing electrostatic charge.

6.2.22 Open the microbalance's draft shield.

6.2.23 Immediately transfer the filter to the center of the microbalance's weighing pan and then close the draft shield.

6.2.24 After the symbol "□" at the upper left corner of the microbalance's display disappears, it means a stable reading is obtained, wait for another 20 seconds and record the value in the "Weight" column on Logbook. If this process takes much longer than 20-30 seconds, evaluate conditions of the weighing room to identify a possible reason.

6.2.25 Remove the filter and put it back into the PetriSlide and set it aside.

6.2.26 Take another filter and repeat Steps 6.2.19 – 6.2.25.

6.2.27 After weighing around 10 filters, or at the end of each weighing session, reweigh the first (routine) filter as a duplicate filter. Mark the filter type "BD" for a batch duplicate.

6.2.28 If the measured value of duplicate filter (BD) is within ±15μg of its previously measured weight, go to Step 6.2.31.

6.2.29 If the result of duplicate filter (BD) measurement disagrees with that of previous measurement by more than ±15μg, flag the filter "FLD" in the Logbook. Set it back into the PetriSlide and close it. Mark the PetriSlide "FLD" and set it aside from the other routine samples.

6.2.30 Reweigh the second and third filters. If either of these measurements also disagrees with their previous measurements by >±15μg, place all samples back into the conditioning environment for at least another 12 hours and thereof repeat all weighing procedures. If the results are still unsatisfactory, troubleshoot the entire measurement system and take appropriate corrective action.

6.2.31 After weighing the first 10 filters and a duplicate filter, reweigh the 100 mg working standard weight by repeating Steps 6.2.11-6.2.13 before weighing another 10 filters in that weighing session. Weigh more filters (around 10) by following Steps 6.2.18-6.2.31.

6.2.32 After all filters have been weighed in the weighing session, reweigh both working standard weights by repeating Steps 6.2.11-6.2.17. Compare the verified and measured values with the working standards. Record the measurement results in the Filter Conditioning and Weighing Logbook. If they disagree by more than ±3μg, reweigh again. If these measurements still disagree by >±3μg, place all samples back into the conditioning environment for at least another 12 hours and repeat the weighing procedures. If the results are still unsatisfactory, troubleshoot the entire measurement system and take appropriate corrective action.

Note 1: Pre-sampling filters and their PetriSlides should be separated from the post-sampling filters and their PetriSlides. Assign different filter holding cabinets for these two types of filters.

Note 2: Keep the routine sample that was used for duplicate weighing, and place it with the batch for the next weighing session. Mark a "PD" and the weighing

batch number on the lid of PetriSlide and the Conditioning and Weighing Logbook. Do not make this sample one of the first three filters in the next batch.

Note 3: Weigh the filter again if it has not been used for sampling within 30 calendar days since the last weighing.

6.2.33 After successful weighing, each pre-sampling filter should be kept in its unique PetriSlide with the dust collecting side up, and then pack tightly together with the other weighed pre-sampling filters. Air-seal the PetriSlides in an anti-static Zip-lock bag. Air inside the Zip-lock bag should be removed as much as possible before sealing.

6.2.34 Keep the Zip-lock bags mentioned in Step 6.2.33 in the Laboratory. Do NOT refrigerate them.

6.3 Post-sampling Filter Conditioning

6.3.1 Particular attention must be paid to Section 4.1.1 prior to conditioning any refrigerated filters.

6.3.2 Follow the filter receiving procedures in Section 7.2.

6.3.3 Place the filters and their PetriSlides in the post-sampling cabinet inside the conditioning and weighing chamber. Place the lids ¾ over the slide bottom plate (i.e. filter holding area) or the lids under the slide bottom plate.

Note : An open-type cabinet should be assigned for conditioning the post-sampling filters. Pre-sampling filter should NOT be allowed to condition in this cabinet. A separate open-type conditioning cabinet should be assigned for the pre-sampling filters.

6.3.4 Group and condition these filters in the assigned post-sampling conditioning cabinet.

6.3.5 Allow the exposed / sampled filters to condition for the length of time established in Section 5.4 under the same environmental conditions as existed during pre-sampling conditioning.

6.3.6 Maintain the required temperature and RH inside the conditioning and weighing chamber. Measure temperature and RH on a continuous basis during filter weight stability experimentation using electronic instrument which outputs are recorded by a data acquisition system.

6.4 Post-sampling Filter Weighing

6.4.1 Weigh the post-sampling filters within 10 calendar days after sampling or 30 calendar days if they are refrigerated at $4^{o}C$ or less.

6.4.2 After post-sampling filter conditioning, calculate the means and standard deviations of the temperature and RH over the preceding 24-hour period. Verify that the mean temperature and RH in the conditioning environment have remained within the acceptance criteria as stipulated in Section 8.1 and that the temperature and RH during

the weighing session is controlled to within ±2°C and ±5% respectively. Record these values in the Filter Conditioning and Weighing Logbook. If the conditioning environment is not properly maintained within these specifications, filters shall not be weighed. Take appropriate troubleshooting and corrective action in that event.

6.4.3 Place the filters to be weighed in order.

6.4.4 Open and close the microbalance's draft shield (circular arrow key) two times to equilibrate the air in the draft shield with the chamber air.

6.4.5 Zero (using the Re-zero key) and internally calibrate (using "Menu" key) the microbalance according to the microbalance's operating manual. This may take 1 to 2 minutes.

6.4.6 Open the microbalance's draft shield.

6.4.7 Using the working standard forceps, gently place the 100 mg working reference standard weight on the weighing pan. Close the microbalance's draft shield.

6.4.8 Wait until the symbol "□" at the upper left corner of the microbalance's display disappears. That means a stable reading is obtained.

6.4.9 After a stable reading is obtained, wait for another 20 seconds. If the reading remains stable, record the measured value in the Filter Conditioning and Weighing Logbook and go to Step 6.4.11.

6.4.10 If the reading fluctuates within these 20 seconds, wait for another 20 seconds and record the reading. If after three attempts the microbalance still does not provide a stable reading, take away the standard weight and repeat Steps 6.4.6 - 6.4.9.

6.4.11 Repeat Steps 6.4.7-6.4.10 with the 200 mg working standard weight.

6.4.12 If the verified and measured values of either working standard disagree by more than ±3μg, repeat Steps 6.4.3-6.4.11. If the results are acceptable, move to Step 6.4.14.

6.4.13 If the two values still disagree with their corresponding standard weights, halt the routine weighing and troubleshoot the entire measurement system. Take appropriate corrective action, including the following :

i) conducting the calibration sensitivity test,

ii) calibrating the microbalance using the microbalance's internal standard,

iii) re-certifying the working standard weights against the primary standard weights, and/or asking a service technician to adjust or repair the microbalance. LS shall not attempt to adjust the microbalance himself.

iv) checking temperature and relative humidity with independent standards,

v) check the working weights with the external laboratory primary weights.

6.4.14 Close the draft shield and allow the microbalance to come to zero. Wait at least 20 seconds to ensure zero is achieved. If not achieved by then, the instrument can be manually zeroed by using the Re-zero key.

6.4.15 Select a filter for weighing and record the filter ID marked on the cover of PetriSlide into the Filter Conditioning and Weighing Logbook. Indicate the filter type as either "RO" for a routine filter, "LB" for a laboratory blank, "FB" for a field blank, "CO" for

a collocated sample, "BD" for a batch duplicate, or "PD" for a duplicate from a previous (day) batch.

6.4.16 Take the filter from its PetriSlide by gently pushing down (with the filter-handling forceps on the outer polyolefin support ring) one side of the filter. This should raise the other end of the filter and with a slight budge, rest the filters edge on the PetriSlide. You can then access the outer polyolefin support ring with the filter-handling forceps.

6.4.17 Lay each filter, with its support ring side upward, in the ^{210}Po anti-static strips for around 30-60 seconds immediately prior to weighing for neutralizing electrostatic charge.

6.4.18 Open the microbalance draft shield.

6.4.19 Immediately transfer the filter to the center of the microbalance's weighing pan and then close the draft shield.

6.4.20 After the symbol "□" at the upper left corner of the microbalance's display disappears, it means a stable reading is obtained. Wait for 20 seconds and record this value in the "Weight" column on Logbook. If this process takes much longer than 20-30 seconds, evaluate the conditions of weighing room to identify a possible reason.

6.4.21 Remove the filter and put it back into the PetriSlide and set it aside.

6.4.22 Take another filter and repeat Steps 6.4.14 – 6.4.21.

6.4.23 After weighting around 10 filters, or at the end of each weighing session, reweigh the first (routine) filter as a duplicate filter. Mark "BD" in the filter type for a batch duplicate.

6.4.24 If the reading of duplicate filter (BD) measurement is within ±15μg of the previously measured value, go to Step 6.4.27.

6.4.25 If the duplicate filter measurement disagrees from the previous measurement by more than ±15μg, flag the filter "FLD" in the duplicate area. Set it back into the PetriSlide and close it. Mark the PetriSlide "FLD" and set it aside from the other routine samples.

6.4.26 Reweigh the second and third filters on Form BAT-01. If either of these measurements also disagree by >±15μg, place all samples back into the conditioning environment for at least another 12 hours and repeat the weighing procedures. Troubleshoot the entire measurement system and take appropriate corrective action.

6.4.27 After weighing the first 10 filters and a duplicate filter, reweigh the 100mg working standard by repeating Steps 6.4.6 – 6.4.9 before weighing another 10 filters in that weighing session. Weigh more filters (around 10 filters), follow Steps 6.4.15 – 6.4.26.

6.4.28 After all filters have been weighed in that weighing session, reweigh both working standards by repeating Steps 6.4.6 - 6.4.12. Compare the verified and measured values with the working standards. Record the measurement results in the Logbook. If they disagree by more than ±3μg, reweigh again. If these measurements still disagree by >±3μg, place all samples back into the conditioning environment for at least another 12 hours and repeat the weighing procedures. If the results are still unsatisfactory, troubleshoot the entire measurement system and take appropriate corrective action.

6.4.29 Review data. In particular, look at field and laboratory blanks and determine if they meet the acceptance criteria (±30μg and ±15μg respectively). If they do not, mark "FFB or FLB respectively in the flag field column of Logbook.

Note 1: Some filters may lose weight due to volatilization of particulates. It is important to keep the initial measured weights of these samples.

Note 2: Keep the routine sample that was used for the duplicate weighing and place it with the batch for the next weighing session. Mark the PetriSlide's lid and the Filter Conditioning and Weighing Logbook with a PD and the weighing batch number. Do not make this sample one of the first three filters in the next batch.

6.4.30 Place each post-sampling filter in its unique PetriSlide immediately after successful weighing. Package the PetriSlides by rubber-banding them tightly together, and then put them in a labeled, air-sealed, anti-static Zip-lock bag. Remove as much air as possible from the bag before sealing.

6.4.31 Place the field blanks in their unique PetriSlides immediately after successful weighing. Package the PetriSlides by rubber-banding them tightly together, and then put them in a labeled, air-sealed, anti-static Zip-lock bags. Remove as much air as possible from the bag before sealing.

6.4.32 Place the laboratory blanks in their unique PetriSlides immediately after successful weighing. Package the PetriSlides by rubber-banding them tightly together, and then put them in a labeled, air-sealed, anti-static Zip-lock bags. Remove as much air as possible from the bag before sealing.

Note : Separate the blanks from the samples by keeping them in different bags.

6.4.33 Refrigerate the blanks and the post-sampling filters with their deposit side up at $4^{o}C$ or less immediately after packing.

7. FILTER CASSETTE AND MAGAZINE HANDLING

Filter cassette and magazine handling should ONLY be done in the Balance Laboratory, but shall be away from the weighing and conditioning chamber. Follow the filter handling procedures in Section 4.1 before starting procedures in Sections 7.1-7.2.

7.1 Insert of Clean Filter into Filter Cassette and Magazine

7.1.1 Clean and inspect the filter cassettes and magazines. Use "clean air duster" to blow out the dust on cassette screen, if necessary.

Note : Do NOT perform this kind of activity inside or close to the conditioning and weighing chamber.

7.1.1 Place each weighed filter into an open filter cassette (R&P part number 59-004648) with serialized screen and then close the filter cassette by snapping its top part onto the

bottom section. Ensure that the top and bottom pieces of the cassette are pushed together completely.

7.1.2 Record the filter ID, sample ID and the serial number of the filter cassette in the Field Operation Log Sheet.

7.1.3 Insert the filter cassettes into a supply magazine (59-004733) which is to be installed at site for sampling. Carefully check the order of the serial numbers of filter cassettes when they are inserted in the magazine.

Note 1 : To avoid cross-contamination of samples, make sure that each pre-sampling filter is separated from the others by the clean dummy filters.

Note 2 : Make sure that the pre-sampling filters are valid for sampling, i.e. used within 30 calendar days since the last weighing. If not, they have to be re-weighed before use.

Note 3 : The sequence of cassettes from top to bottom of magazine should be in this order: dummy filter cassette (top), sample filter cassette, dummy filter cassette, sample filter cassette, …, field blank cassette, dummy filter cassette (bottom). Field blank(s) should always be placed on top of the last dummy filter at bottom.

7.1.4 Cap the magazine and put it inside a magazine transport container (R&P part number 20-004997). Properly seal the container and the Field Operation Log Sheet in an air-sealed Zip-lock bag. Preparation for pre-sampling filters is completed.

7.1.5 Store the prepared pre-sampling filters inside the Laboratory. Do NOT refrigerate these filters.

7.1.6 Do NOT refrigerate the pre-sampling filters during transport. Water condensation on the filter surfaces will easily occur if they are suddenly exposed to a humid outdoor environment, especially in summer season.

7.2 Removal of Sampled Filter from Filter Cassette and Magazine

7.2.1 Refrigerate the capped storage magazine at 4^{o}C or less if the post-sampling filters are not going to be removed from the magazine after being returned from site. Keep the capped magazine inside the air-sealed Zip-lock bag before refrigeration, and try to remove as much as possible the air inside the bag before sealing.

7.2.2 Before weighing, equilibrate the refrigerated storage magazine and the Zip-lock bag for 12~24 hours in an air-conditioned room (at around 25^{o}C) before taking it into the Balance Laboratory.

Note : Do NOT equilibrate the refrigerated filters inside the Laboratory.

7.2.3 After equilibrating the magazine/filters, take it to the Balance Laboratory for filter removal.

7.2.4 The most convenient means of removing filter cassettes from a filter cassette magazine is to make use of the R&P Cassette Removal Sleeve (36-004734).

7.2.5 Carefully remove the caps over the open and end of the filter cassette magazine.

7.2.6 Insert the four pins on the inner surface of the metal cap of the Cassette Removal Sleeve into four slots at the top of the filter cassette magazine.

7.2.7 Attach the bulb pump hose to the magazine's air connector fitting with the quick connect fitting on the end of the hose.

7.2.8 Squeeze the bulb gently until the topmost filter cassette is positioned in the slot of the metal cap.

7.2.9 Carefully slide the filter cassette out of the slot. Continue to push all filter cassettes out the slot by squeezing gently on the bulb. When all filter cassettes have been removed from the magazine, remove the metal cap from the top of the magazine and disconnect the hose from the magazine air connector.

7.2.10 Carefully separate the filter cassette with the Cassette Separator Tool (R&P part number 38-004892).

7.2.11 Use a clean filter-handling forceps to remove the post-sampling filter from the cassette. Then carefully set the filter in its unique PetriSlide with the filter deposit side up.

Note : The cassettes and magazine can be used to hold other filters in the next sampling cycle once it has been cleaned thoroughly.

7.2.12 Examine the filter for defects that may have occurred during sampling, as well as for evidence of leaks in the filter cassette. Leaks manifest themselves as pronounced radial streaks that extend beyond the exposed area of the filter.

7.2.13 Place each filter in its unique PetriSlide with the deposit side up, and then cover it with the lid.

7.2.14 Refrigerate the post-sampling filters/PetriSlides as mentioned in Step 7.2.13 at 4^{o}C or less when they are not in use. All PetriSlides shall have been tightly packed by rubber band and kept inside an air-sealed, anti-static Zip-lock bag before refrigeration. Always try to remove as much air as possible from the Zip-log bag before proper sealing and to keep the filter deposit side up during storage/refrigeration.

7.2.15 Regularly monitor and record the indoor temperature of refrigerators and freezer. Make sure that the indoor temperatures are always maintained below 4^{o}C. Wherever possible, they should be connected to the emergency power supply of the building so that unnecessary power interpretation due to accidental breakdown of city-lines can be avoided.

8 QUALITY CONTROL

8.1 Acceptance Criteria for Laboratory QC Check

Requirements	Measurement Frequency adopted by Hong Kong EPD	Acceptance Criteria adopted by Hong Kong EPD	Relevant USEPA's Handbook : Document 2.12
Conditioning Environment			
High-efficiency particulate air (HEPA) filter	Bimonthly	Replace a clean filter	Not described
Temperature	Each weighing session	24-hour mean temp between 20 and 23 ^{o}C, control ±2^{o}C (SD)	2.12 Sec. 7.6
Relative humidity (RH)	Each weighing session	24-hour mean RH between 30% and 40% RH, control ±5% RH (SD)	2.12 Sec. 7.6
Filter Blanks			
Lot blanks	3/box, 3 boxes/lot, 9 total (Daily for at least 5 days)	Max. diff of ± 15μg between weighings	2.12 Sec. 7.6
Lot exposure blanks	3 filters for 1 pre-, 1 post-sampling weighing session/lot	Avg. diff of ≤5μg, No filter diff > 15μg	Not described
Field blanks	One per sampler/wk	± 30μg difference	Not described
Laboratory blanks	One per weighing session	± 15μg difference	2.12 Sec. 7.8
Calibration and Verification			
Working mass standards verification	Quarterly	± 0.025mg	2.12 Sec. 7.3
Mass standards	Yearly	Traceable to NIST primary reference standard	2.12 Sec. 7.3
Balance calibration	Annually or as needed	Not applicable	Not described
Temperature/ humidity verification	Quarterly	± 2 ^{o}C / ± 2 %	Not described
Microbalance QC Checks			
Working standard QC check	Start, around every 10th filter and end of each weighing session	± 3μg	2.12 Sec. 7.8
Duplicate filter weighing	1 filter at end of weighing session; 1 carried over to next session with lab blank	± 15μg	Not described
Performance Evaluations			
Inter-laboratory Comparisons	Quarterly	Audit by EPD's audit team. Details refer to Quarterly Calibration Log Sheet	Not described

8.2 Acceptance Criteria for Blank Filters

There are four types of blank filters assigned for quality control: lot blank, lot exposure blank, laboratory blank and field blank.

8.2.1 **Lot blanks** representing a new lot or shipment of filters are unconditioned, unsampled virgin filters used to determine if effects on filter weight stability have occurred due to the volatilization of material from the filter or the absorption of atmospheric gaseous material into the filter. Measure the weight stability and the time required to achieve weight stability of each new shipment of filters by opening three new boxes from each shipment and randomly selecting 3 filters from each box to be used as lot blanks. A filter lot is defined as a single shipment of filters from a filter manufacturer, or other source of filters. After an initial 24-hour conditioning, re-weigh these filters periodically (daily for at least 5 days) and store them in the conditioning environment between weighings. Continue these weighings until the average weight change of these 9 filters is less than 5 μg /day and ±15μg.

8.2.2 **Lot exposure blanks** are blank filters from a new lot that are part of a batch of filters that will be conditioned together before weighing, and are included to show the effects of any event associated with a particular conditioning period of laboratory exposure. These blanks are only used with the batch of filters with which they are conditioned.

8.2.3 **Laboratory blanks** are conditioned, unsampled filters used to determine any weight change between pre-sampling and post-sampling weighings due to contamination in the microbalance and conditioning environment. Keep laboratory blanks inside the conditioning environment. Weigh enough laboratory blanks during a pre-sampling weighing session to provide at least one single-use laboratory blank during each subsequent post-sampling weighing session. Record the pre-sampling and post-sampling weights in the Logbook. If the weight change in the laboratory blank exceeds ±15μg, contamination in the conditioning environment may be occurring. Take appropriate troubleshooting and corrective action in that event.

8.2.4 **Field blanks** are conditioned, unsampled filters used to determine whether similar contamination occurs during sampling. Weigh enough field blanks during a pre-sampling weighing session to provide one single-use field blank for each sampler. Record the pre-sampling and post-sampling weights in the Logbook. If the weight change in the field blank exceeds ±30μg, contamination during transportation or at the evaluation site may be occurring. Take appropriate troubleshooting and corrective action in that event.

8.3 Cautions

To maintain a good quality control for filter weighing, the following handling procedures should be cautioned:

8.3.1 Pre-sampling Condition

8.3.1.1 The pre-sampling filters should be prepared in the Balance Laboratory. Laboratory staff shall follow the handling procedures described in section 4.1.

8.3.1.2 Cap the supply magazine in which pre-sampling filters are stored. Put the magazine inside a metal box and then keep the box inside an air-sealed Zip-lock bag.

8.3.1.3 Supply magazine/ PetriSlides which keep the pre-sampling filters should be stored in the Balance Laboratory before use.

Note 1: Do **NOT** refrigerate the pre-sampling filters/ PetriSlides/ supply magazine to avoid water condensation on filter surface during exposure to humid outdoor environment.

Note 2: Pack and store the unused PetriSlides in an air-sealed Zip-lock bag when they are not in use.

8.3.1.4 Reweigh the pre-sampling filters if they are not used for sampling within 30 calendar days after weighing.

8.3.1.5 Electrostatic charges on filters should be removed by moving it over ^{210}Po strips for 30~60 seconds before weighing.

Note : The ^{210}Po strip should be replaced every 6 months and returned to the manufacturer for safe disposal.

8.3.2 Post-sampling Condition

8.3.2.1 The post-sampling filters should be collected within 4 calendar days after the end of sampling period.

8.3.2.2 A clean, dummy cassette should be put on top of the storage magazine immediately after being removed from the sampler. It helps reduce the clearance between the cap and the cassette, and minimize the movement of cassettes during delivery. Then cap the storage magazine and keep it inside a metal box. Properly seal the metal box by air-sealed Zip-lock bag and put it in an ice box with frozen blue ice inside. Return the ice box to EPD's laboratory within 8 hours.

8.3.2.3 The post-sampling filters should be conditioned and weighed within 10 calendar days after the end of the sampling period. Unless the filters are refrigerated at 4^{o}C or less during the entire time between retrieval from the sampler and start of the conditioning, in which case the period shall not exceed 30 calendar days.

8.3.2.4 Allow filters from the refrigerator to equilibrate to room temperature for 12 ~ 24 hours in an air-conditioned environment. During the equilibrating stage, filters shall not be

removed from the supply magazines/ PetriSlides and the air-sealed Zip-lock bags to avoid contamination.

Note : Since filters and/or containers from the refrigerator are probably colder and/or more or less humid than the conditioning room/chamber, do **NOT** place them, immediately after retrieving from the refrigerator, in the Balance Laboratory to avoid influencing the stability of controlled temperature and relative humidity of the conditioning environment.

8.3.2.5 Electrostatic charges on filters should be removed by moving it under ^{210}Po strip for 30~60 seconds before weighing.

8.3.3 Others

8.3.3.1 Always keep the microbalance in the Balance Laboratory.

8.3.3.2 Ensure the Balance Laboratory has a HEPA-filtered air supply system on its inlet air system to minimize airborne contaminants.

8.3.3.3 Change the HEPA-filter regularly.

8.3.3.4 Maintain the Laboratory at a slightly positive pressure so that air flows away from the balance and conditioning area.

8.3.3.5 Minimize ingress to and egress from the room.

8.3.3.6 Minimize dust contamination by cleaning the weighing area daily; by installing, using, and as needed, replacing sticky floor coverings (minimum weekly) at the entrance of Laboratory; and by wearing clean laboratory clothing over any clothing exposed to uncontrolled environment.

8.3.3.7 Regularly clean the floor and table surfaces by lint-free wipes/sticking papers.

8.3.3.8 Place the microbalance on a balance table to reduce vibrations.

8.3.3.9 Avoid bumping the microbalance to prevent disturbing its calibration.

8.3.3.10 Verify that analytical microbalance has been calibrated by an authorized microbalance service representative within the past 6 months.

8.3.3.11 If more than one microbalance is used, make the pre- and post-sampling measurements of each filter on the same microbalance. (not applicable in this project, but a reminder for future application)

8.3.3.12 Verify that the working standards have been either recertified within the past year against NIST-traceable mass standards at a State weights and measures laboratory holding a NIST Certificate of Traceability or at a calibration laboratory accredited by NVLAP and verified within the past 3 months against the laboratory primary standards at the weighing laboratory.

8.3.3.13 Check the working standards' masses, after any incident of rough handling, and compare to their verified value.

8.3.3.14 Select working standards so that they will bracket the mass of a blank or loaded filter (that is, 100 and 200 mg).

8.3.3.15 When weighing filters, hold the filter (with filter forceps) only by the outer polyolefin

supporting ring, not by the filter material.

8.3.3.16 If the forceps ever touch the filter material of an exposed filter, flag the sample and thoroughly clean the forceps with disposable laboratory wipes to avoid cross-contamination.

8.3.3.17 Place lint-free, anti-static papers/wipes on the table surface before performing filter preparation or removal.

9 SAMPLE SHIPMENT

Ship the samples and blanks by courier service if the analytical laboratory is an overseas one. Follow the shipment instructions below for filter delivery :

9.1 Contact the analytical laboratory prior to shipping samples. Filter samples should be individually stored in Zip-lock PetriSlides (e.g. Millipore Corporation catalog number PD1504700) which have a rim on the inside of the lid that holds the filter securely by its outer edge. This prevents the deposit surface of the filter from coming in contact with the PetriSlide during shipment.

9.2 Label each filter sample with a unique ID of 8 characters or less. Ask the analytical laboratory to track the samples and report results referenced to this ID. Put the ID label on the flat part of the base.

9.3 Separate the sampled filters from blanks for packing.

9.4 Package the PetriSlides by rubber-banding them tightly together or otherwise securing them so the lids do not come off during shipment. Wrap in bubble-wrap or other packing material to minimize vibration and potential loss of particles during shipment. Do not use Styrofoam as it outgases components that may interfere with analysis. Place filters deposit side up in a shipping container. For Teflon filters scheduled for EDXRF analysis, include laboratory blanks (i.e. unused filters); 5% lab blanks or at least 3 or 4 filters of the same manufacturing lot as the samples are required. Include field blanks (filters loaded in the sampler and left for the same period of time as the samples, but with no air drawn through them) as well, if they were collected.

9.5 For quartz fiber filters that are scheduled for carbon analysis, or for other filters where loss of volatile components such as organic carbon or nitrate is not desired, ship cold. Use an insulated shipping container (such as a soda cooler) and place enough frozen “Blue Ice” packs in the cooler to keep the samples cold during shipment. Seal the Blue Ice packs in Zip-Lock bags to prevent damage in case of leaks. Ship via overnight delivery on Monday, Tuesday or Wednesday to minimize the chance of filters warming up during shipment or over the weekend. Include field blanks if they were collected. Ask for analyzing quartz lab blanks for background subtraction if field blanks are not available. However, concentrations of some species such as organic carbon are typically much higher on field blanks than lab blanks.

9.6 Deposit mass should range between 200 and 2000 micrograms for best results. It is hard to analyze samples with certain unusual chemical composition or with heavy loading.

9.7 Include a cover letter that identifies the project and a contract or purchase order number. Include a description of the samples, noting whether they are ambient or source samples, the sampler type, and the purpose of the laboratory analyses, Specify which samples receive analysis for chemical species. Attach a listing of the samples by ID number and any chain-of-custody forms that are required.

9.8 Prepare a "field data" file that contains sample ID, particle size (e.g. PM2.5 or PM10), sampling site, date and time, air volume, mass, or any parameter need. Send a copy of the field data file via e-mail to the analytical laboratory and include a disk copy in the shipment.

10. REFERENCES

(1) The USEPA Quality Assurance Guidance Document 2.12: Monitoring PM2.5 in Ambient Air Using Designated Reference of Class I Equivalent Methods, April 1998

(2) Federal Register / USEPA Appendix L of 40 CFR Part 50 (EPA 1997a)

(3) Rupprecht & Patashnick's Operating Manual for Partisol-Plus Model 2025 Sequential Air Sampler.

(4) The USEPA Quality Assurance Document: Quality Assurance Project Plan for the PM2.5 Performance Evaluation Program, Feb 1999

(5) The USEPA Quality Assurance Guidance Document: PM2.5 Mass Weighing Laboratory Standard Operating Procedures for the Performance Evaluation Program, Oct 1998.

(6) Radiation Report, Health Department, Hong Kong SAR Government.

Filter Inventory / Inspection Form

Lot No.	Box No.	Filter Medium	Brand Name	Cat. No. or P/N No.	Date Received	Date Pre-fired	Range of Filter ID		Date Inspected	No. Accepted	Lab. Analyst Initials
							From	To			

Form: INV-1

Lot Blank Filter Stability Test

Filter Medium: QMA / Teflon * **Lot No.:** _____ **Operator's Initial :** _____

Starting Filter Conditioning Date: _____ Time: _____

Weighing Date												
Weighing Time												
Past 24 hrs. Ave. Temp.												
Past 24 hrs. Ave. RH												
	Filter ID	Day1 (mg)	1 - 2 Diff (µg)	Day2 (mg)	2 - 3 Diff (µg)	Day3 (mg)	3 - 4 Diff (µg)	Day4 (mg)	4 - 5 Diff (µg)	Day5 (mg)	Total Diff (µg)	Average (µg)
QC1 100mg												
QC2 200mg												
Filter1												
Filter2												
Filter3												
Filter4												
Filter5												
Filter6												
Filter7												
Filter8												
Filter9												
QC1 100mg												
QC2 200mg												

Remarks:

Acceptance criterion is maximum difference of ±15µg between weighings.

Form: FT01

Lot Exposure Blank Filter Stability Test

Filter Medium: QMA / Teflon * **Lot No.:** _____ **Operator's Initial :** _____

Starting Filter Conditioning Date: _____ Time: _____

Weighing Date										
Weighing Time										
Past 24 hrs. Ave. Temp.										
Past 24 hrs. Ave. RH										
	Filter ID	**(1)** Initial Weight (mg)	1 - 2 Diff (µg)	**(2)** 24 hrs (mg)	2 - 3 Diff (µg)	**(3)** 48 hrs (mg)	3 - 4 Diff (µg)	**(4)** 72 hrs (mg)	4 - 5 Diff (µg)	**(5)** 96 hrs (mg)
QC1 100mg										
QC2 200mg										
Filter1										
Filter2										
Filter3										
QC1 100mg										
QC2 200mg										

Remarks:
Acceptance criteria are the average difference between all three weights of 5µg or less and no filter difference greater than 15µg.

Form: FT02

Post-sampling Routine Filter Stability Test

Filter Medium: QMA / Teflon * **Lot No.:** _____ **Operator's Initial :** _____

Starting Filter Conditioning Date: _____ Time: _____

Weighing Date											
Weighing Time											
Past 24 hrs. Ave. Temp.											
Past 24 hrs. Ave. RH											
	Filter ID	**(1)** Initial Weight (mg)	1 - 2 Diff (μg)	**(2)** 12~24 hrs (mg)	2 - 3 Diff (μg)	**(3)** 12~24 hrs (mg)	3 - 4 Diff (μg)	**(4)** 12~24 hrs (mg)	4 - 5 Diff (μg)	**(5)** 12~24 hrs (mg)	
QC1 100mg											
QC2 200mg											
Filter1											
Filter2											
Filter3											
QC1 100mg											
QC2 200mg											

Remarks:
Acceptance criteria is the difference between two out of the three filters consecutive weights less than 15μg.

Form: FT03

Filter Conditioning / Weighing Record

FILTER CONDITIONING								FILTER WEIGHING								
Date (yyyy/mm/dd)	Time (hh:mm)	Filter ID / working standard	Pre-sampling / Post-sampling	Batch No.	Filter Type*	Filter Inspection (✔ for OK)	Operator (Initials)	Date (yyyy/mm/dd)	Time (hh:mm)	Mean Temp[1] (^{o}C)	SD of Temp (^{o}C)	Mean RH[1] (%)	SD of RH (%)	Weight (mg)	Flag	Operator (Initials)
			Pre / Post **													
			Pre / Post **													
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* Indicate working standard (WS), routine filter (RO), laboratory blank (LB), field blank (FB), collocated sample (CO), batch duplicate (BD), previous batch (PD).

** Delete as appropriate;

1 - Mean Temp & RH for Past 24 hours.