California Environmental Protection Agency

Air Resources Board

STANDARD OPERATING PROCEDURE (SOP) FOR THE DETERMINATION OF PM2.5 MASS IN AMBIENT AIR BY GRAVIMETRIC ANALYSIS

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STANDARD OPERATING PROCEDURE (SOP) FOR THE DETERMINATION OF PM2.5 MASS IN AMBIENT AIR BY GRAVIMETRIC ANALYSIS

1.0 SCOPE

This document describes the methodology used by Monitoring and Laboratory Division (MLD) Inorganics Laboratory Section (ILS) staff to analyze the mass of particulate matter less than 2.5 μm in diameter (PM2.5) samples collected on Teflon filters.

2.0 SUMMARY OF METHOD

Individual Teflon filters (46.2 mm diameter) are weighed on an electronic micro-balance before and after field sampling. Particulate matter less than 2.5 μm in diameter is collected from ambient air over a 24-hour period. PM2.5 filters are sampled on two different types of samplers: Rupprecht and Patashnick (R&P) (either a single channel or sequential), or a Speciation Air Sampling System (SASS™) low-volume sampler. The net difference between pre- and post-sampling filter weights is used to calculate the ambient air mass concentration. After post-weighing, all filters are archived; however, samples from the SASS™ sampler are stored for subsequent analysis.

3.0 INTERFERENCES

3.1 The potential effect of body moisture or oils contacting the filters is minimized by using non-serrated forceps and gloves to handle the filters at all times. This measure also moderates interference due to static electricity.

3.2 Teflon or Polytetrafluoroethylene (PTFE) filters accumulate a surface electrical charge, which may affect filter weight. Static electricity is controlled by treating filters with a "Static Master" static charge neutralizer prior to weighing. Static Master static strips must also be taped inside each balance chamber.

3.3 Moisture content can affect filter weight. Filters must be equilibrated for a minimum of 24 hours in a controlled environment prior to pre- and post-weighing. See Section 6.1 for limits.

3.4 Airborne particulate can adversely affect accurate mass measurement of the filter. Filters undergoing conditioning should not be placed within an airflow path created by air conditioning ductwork, computer printers, or frequently opened doorways. Cleaning laboratory bench-tops and weighing areas daily, installing "sticky" floor mats at doorway entrances to the balance room and wearing clean lab coats over regular clothing can further minimize dust contamination.

4.0 PERSONNEL QUALIFICATIONS

Personnel should be trained to operate a micro-balance, to properly handle 46.2 mm Teflon or PTFE filters, to operate the Mettler Toledo FA/747 Automatic Filter Weighing System, and to operate the Laboratory Information Management System (LIMS).

5.0 SAFETY STATEMENT

All personnel shall follow the general health and safety requirements found in the Air Resources Board's (CARB or ARB) Chemical Hygiene Plan.

6.0 EQUIPMENT

6.1 Environmentally controlled laboratory. Per Code of Federal Regulations (CFR), Title 40, Part 50, Appendix L, the balance room's relative humidity must be maintained at a mean value range of 30-40% and its air temperature must be maintained at a mean value range of 20.0-23.0°C. If the balance room is out of range for temperature or humidity, no weighing can occur until the room is back in range for 24 hours.

6.2 Laboratory Information Management System (LIMS).

6.3 Mettler Toledo XP6 electronic micro-balance with a minimum resolution of 0.001 mg and a precision of ± 0.001 mg, supplied with a balance pan. The micro-balance must be positioned on a vibration-damping balance support table and should be interfaced with the LIMS database system.

6.4 Mettler Toledo FA/747 Automatic Filter Weighing System (Robot) that includes the XP2U electronic micro-balance. The system should be interfaced with the LIMS database system.

6.5 Calibration weights, utilized as Mass Reference Standards, should be non-corroding, and be certified as traceable to National Institute of Standards and Technology (NIST) mass standards. Lower and upper control weights that "bracket" the expected filter masses are needed (i.e. 100 mg and 500 mg). Two sets are needed, one set as a working standard and one set as the primary standard. The weights should be American Society for Testing and Materials (ASTM) Class 1 category.

6.6 Laboratory ionizer stand (positioner) for use with the Static Master antistatic strips. This stand positions the antistatic strips for optimal static charge neutralization.

6.7 Radioactive (alpha particle) Polonium-210 (Static Master) antistatic strips for static charge neutralization. At least eleven (11) strips are needed per balance: six (6) 1"x1" strips for inside the balance chamber, two (2) 3"x1" strips for the positioner, and three (3) 3"x1" strips for use on the workbench.

6.8 Non-serrated forceps for handling filters.

6.9 Non-metallic, non-serrated forceps for handling weights.

6.10 Digital timer/stopwatch.

6.11 Filter: Teflon membrane or PTFE membrane, 46.2 mm diameter with a polypropylene support ring.

6.12 Filter support cassettes and covers. The support cassettes must be compatible with the air samplers.

6.13 Filter cassette opener.

6.14 Filter equilibration racks.

6.15 Relative humidity/temperature recorders—one probe must be placed in the balance room and one must be placed inside the robot. An additional recorder is used as a primary which must be annually certified as NIST traceable.

6.16 Antistatic, nitrate-free, phosphate-free, sulfate-free, and powder free vinyl gloves.

6.17 Plastic petri-slide filter containers.

6.18 Zip-lock plastic bags, 6"x 9".

6.19 Disposable laboratory wipes.

6.20 Filter equilibration cabinets.

6.21 "Sticky" floor mats.

6.22 Time/Temperature tags, 5°C and 25°C.

6.23 Metal filter-shipping canisters.

6.24 Light source for inspecting un-sampled filters.

6.25 Rubber air dust blower for removing particles from filters.

7.0 PROCEDURES

7.1 Filter Inspection and Stability

7.1.1 For equilibration, the filters are transferred from their sealed manufacturer's packaging to a filter-handling container such as a plastic petri-slide. The filters are handled with non-serrated forceps. Lab personnel must wear vinyl gloves that are free of contaminant ions, powder-free, and anti-static when filters are being prepared for conditioning and weighing. Before any filter is placed in a filter-handling container, it must be inspected for defects. This is done by an examination of the filter by holding it to a light source. A filter must be discarded if any defects are identified. Specific defects to look for are the following:

7.1.1.1 **Pinhole**—A small hole appearing as a distinct and obvious bright point of light when examined over a light source.

7.1.1.2 **Separation of ring**—Any separation or lack of seal between the filter and the filter support ring.

7.1.1.3 **Chaff or flashing**—Any extra material on the reinforcing ring or on the heat-seal area that would prevent an airtight seal during sampling.

7.1.1.4 **Loose material**—Any extra loose material or dirt particles on the filter.

7.1.1.5 **Discoloration**—Any obvious discoloration that might be evidence of contamination.

7.1.1.6 **Other**—A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

7.1.2 Maintain an adequate supply of filters equilibrating so that the minimum equilibration period is always met before the filters are pre-weighed.

7.2 PRE-WEIGHING OF UNSAMPLED FILTERS

7.2.1 Manual Balance

7.2.1.1 Ensure that the temperature and the relative humidity of the Balance Room have remained and are currently within the allowable limits (see Section 6.1 for limits) throughout the preceding 24 hours. Also, make certain the selected filters have been conditioned for at least the minimum time needed to attain mass stability, as determined from the filter lab blanks.

7.2.1.2 Clean the micro-balance weighing chamber with a fine brush, if necessary. Clean the surfaces near the micro-balance with antistatic solution or disposable lab wipes moistened with isopropanol. Clean the forceps used for handling the mass reference weights and the

filters prior to each weighing session. Ensure that all forceps in use are dry.

7.2.1.3 Perform an internal and external calibration of the micro-balance as described in Section 8.1.1.1 prior to beginning the weighing session. Transfer the external calibration values into LIMS.

7.2.1.4 Weigh the lab blanks. See section 8.2.2 for limits.

7.2.1.4.1 Using forceps, grip a filter only by the outer polypropylene support ring and place the filter onto a static neutralizer strip for a minimum of 30 seconds. Then, using forceps, pass the filter through the static strip ionizer stand 3 times before placing on the balance.

7.2.1.4.2 Leave the lab blank on the balance for 30 seconds. Transfer the weight value into LIMS. Using forceps, remove the lab blank and return it to the petri-slide.

7.2.1.5 Prepare an appropriate number of CARB 24-hour - Field Sample Report (24-Hr Report) forms with site name(s) and barcode labels. Assign appropriate number of field and trip blanks and place labels on the 24-Hr Report form. For filters designate for SASS™ samplers, use the appropriate SASS sample and field blank forms.

7.2.1.6 Gather the appropriate filter support cassettes and metal covers. For filters being sent to monitoring sites using SASS™ samplers, use petri-slides.

7.2.1.7 Create a worklist in LIMS using the barcode numbers from the 24-Hr Report forms. Insert duplicates and upper and lower control working standards into the worklist.

7.2.1.8 There must be a duplicate mass rate of ≥10%. Duplicates are automatically inserted by LIMS after every nine filters using the first filter of each batch.

7.2.1.9 Working standards are automatically inserted by LIMS at the beginning and at the end of the worklist. After each duplicate sample, the upper and lower control working standards are alternately inserted automatically by LIMS.

7.2.1.10 Place the lower control working standard on the balance pan using a pair of non-metal forceps. Transfer the weight reading into LIMS after

30 seconds. Repeat these steps with the upper control working standard.

7.2.1.11 Using forceps, place a filter on the balance pan and close the chamber. At the end of 30 seconds, transfer the mass data into the LIMS worksheet. Record on the 24-Hr Report form the following:

7.2.1.11.1 Cassette identification (I.D.) number (each support cassette rim is marked with an I.D. number).

7.2.1.11.2 Pre-weight mass of the filter.

7.2.1.11.3 Date of pre-weight measurement.

7.2.1.11.4 Analyst's initials.

7.2.1.12 Place the weighed filter into the appropriate filter support cassette.

7.2.1.13 Repeat above steps (7.2.1.1.11 and 7.2.1.12) for subsequent filters and duplicate weighings.

7.2.1.14 Duplicate weighings—If the original and duplicate mass values differ by ±15 µg, check the two mass results, and reweigh the filter, if necessary. If the difference is still not within ±15 µg, consult with the lab supervisor.

7.2.1.15 Control weighings—After each duplicate weighing, alternately weigh the working standards. If a control weight is out of range by ±0.003mg, reweigh the control. If the control is still out of range, reweigh the control and the entire set of filters between that control and the next control that is in range. If the control is still out of range, consult with the lab supervisor.

7.2.1.16 Repeat above steps (7.2.1.14 and 7.2.1.15) subsequent batches of filters.

7.2.1.17 At the end of the weighing session, weigh the upper and lower control working standards. After the last control has been transferred to the worklist, examine the data for errors and correct, if necessary.

7.2.1.18 Transfer data to LIMS, print a pre-weight summary report, and log trip blanks into LIMS.

7.2.1.19 After the set of filters has been weighed for R&P samplers, snap on the top of the plastic cassette, then place the protective metal covers on

the bottom and top of the cassette. With an appropriate labeling system, designate whether or not a filter is a "field blank" or a "trip blank". If any filter(s) is labeled blank(s), make the appropriate notation on the corresponding 24-Hr Report form. If the filters are for a SASS™ sampler, place the filter into a petri slide and apply a speciation sample label. If the filter is for a SASS™ sampler and is a blank, a "field blank" 24-Hr Report form is used.

7.2.1.20 After pre-weighing for R&P samplers is complete, obtain a metal shipping canister for each site that filters were weighed for. Place the cassettes containing the pre-weighed filter and the metal covers in the slots inside the metal filter shipping canister and screw on the cover. The 24-Hr Reports should be folded in a manner that easily identifies the site. Place these folded reports in a 6 in x 9 in zip-lock bag and wrap the bag around the metal shipping canister, securing it with a large rubber band. Canisters are shipped to the site operator, or left for pick up by the site operator. Any canisters being shipped out are to be taken to the stockroom where they will be shipped to the designated site.

7.2.1.21 When the pre-weighing for SASS™ samplers is done, deliver the filters contained in labeled petri slides along with the corresponding custody/field data sheets to the bin in the speciation sample processing lab.

7.2.2 Robot Balance

7.2.2.1 Ensure that the temperature and the relative humidity of the balance room have remained (and are currently) within the allowable limits (see Section 6.1 for limits) throughout the preceding 24 hours. Also, make certain the selected filters have been conditioned for at least the minimum time needed to attain mass stability, as determined from the filter lot blanks.

7.2.2.2 Clean the surfaces near the robot with antistatic solution or disposable lab wipes moistened with isopropanol. Clean the forceps used for handling the mass reference weights and the filters prior to each weighing session. Ensure that all forceps in use are dry.

7.2.2.3 Perform an internal and external calibration of the micro-balance as described in Section 8.1.1.2 prior to beginning the weighing session. Transfer the values to LIMS.

7.2.2.4 Perform the Calibration/Lab Blank weighing:

7.2.2.4.1 Load the lab blank filters into the robot. Refer to Section 7.2.2.8 for filter loading procedure.

7.2.2.4.2 Use robot software and LIMS to set up the run and ensure that the physical filter positions match up with the software filter positions.

7.2.2.5 Prepare an appropriate number of 24-Hr Report forms with site name(s) and barcode labels.

7.2.2.6 Obtain the appropriate filter support cassettes and metal covers. For filters being sent to monitoring sites using SASS™ samplers, use petri slides.

7.2.2.7 Import the preweight worklist into the robot table. There must be a duplicate mass rate of ≥10%. Duplicates are automatically inserted by LIMS after every nine filters using the first filter of each batch. Working standards are automatically inserted by LIMS at the beginning and at the end of the worklist. After each duplicate sample, the working standards are alternately inserted automatically by LIMS.

7.2.2.8 Using forceps, grip a filter only by the outer polypropylene support ring and place the filter onto a static neutralizer strip for a minimum of 30 seconds. Then, using the forceps, pass the filter through the static strip ionizer stand 3 times. Place the filter into the appropriate metal filter holder located inside the robot.

7.2.2.9 Perform the weighing

7.2.2.9.1 Load the filters into the robot (Section 7.2.2.8).

7.2.2.9.2 Use robot software and LIMS to set up the run and ensure that the physical filter positions match up with the software filter positions.

7.2.2.9.3 Duplicate weighing—If the original and duplicate mass values differ by ±15 µg, check the two mass results, and reweigh the filter, if necessary. If the difference is still not ±15 µg, consult with the lab supervisor.

7.2.2.9.4 Control weighing—After each duplicate weighing, the working control standards will be automatically weighed. If a control weight is out of range by ±0.003 mg, reweigh the control. If the control is still out of range, reweigh the control and the entire set of filters between that control and the next

control that is in range. If the control is still out of range, consult with the lab supervisor.

7.2.2.10 Place the weighed filter into the appropriate filter support cassette. Ensure that the barcodes from worksheet and robot position match the cassette number on the 24-Hr Report form.

7.2.2.11 Record on the 24-Hr Report form the following:

7.2.2.11.1 Cassette I.D.

7.2.2.11.2 Pre-weight mass of the filter.

7.2.2.11.3 Date of pre-weight measurement.

7.2.2.11.4 Analyst's initials.

7.2.2.12 Perform steps Sections 7.2.1.18 through 7.2.1.21 to transfer data into LIMS, print a pre-weight summary report, log trip blanks into LIMS, and ship the filters to the sites.

7.3 TRACKING DOCUMENTATION AND INSPECTION OF SAMPLED FILTERS

7.3.1 Upon receipt of samples from the field, examine the Temperature Threshold Indicators inside the filter-shipping canister. These determine what receiving temperature is entered on the 24-Hr Reports with this canister. If both indicators have not turned pink, enter "<4." If the 5°C indicator is pink, but the 25°C indicator has not changed color, enter "4-25." If both have turned pink, enter ">25". Also enter the current date, time, and your initials in the appropriate spaces for the "Sample Received at Lab" line for each of the 24-Hr Reports. Refer to section 7.3.10 for post weigh time limits.

7.3.2 Verify that each 24-Hr Report has a corresponding cassette. Inspect the condition of the exterior and interior walls of the canister for evidence of moisture accumulation on the filters.

7.3.3 Examine the 24-Hr Report form for completeness. The Sample Summary section must be fully completed by the site operator. The Chain of Custody section of the 24-Hr Report must have the following information and signatures:

INFORMATION	SIGNATURES
Sample Summary Section	None required
Date, time filter loaded onto sampler	Site Operator
Date, time filter removed from sampler	Site Operator
Date, time filter placed in freezer	Site Operator
Date, time, temperature conditions when filter shipped to lab	Site Operator
Date, time, temperature conditions when filter received	Balance Room Staff
Date, time, temperature conditions when filter begins equilibrating	Balance Room Staff

If any of the above information and/or signatures is not available before login, these samples and their attached 24-Hr Reports are to be "flagged" and the site operator contacted to provide the missing information.

7.3.4 Remove each filter individually from the metal shipping canisters. Detach the protective metal covers but leave each filter in its filter support cassette for identification purposes. Check the physical appearance of the filters for contamination and/or filter damage which may invalidate the sample. Filters from SASS™ samplers will be in petri slides. 24-Hr Report forms for these samples are found in a folder in the balance room. These filters are handled the same as R&P filters.

7.3.5 If there is evidence of contamination and/or damage to the filter, make note of it in the "lab comments" section of the 24-Hr Report form and write "INVALID" at the top of the report form. The sample may remain valid in LIMS until after post-weigh. For directions on invalidating a sample in LIMS, refer to the LIMS SOP.

7.3.6 Remove the cassette top and allow each filter to condition on a designated rack for at least 24 hours. Assemble the 24-Hr Reports in such a manner as to facilitate login into the LIMS database.

7.3.7 All samples, whether designated as routine sample, field blank, or trip blank, will be logged into LIMS.

7.3.8 Each filter logged in using this program will generate a LIMS I.D. number (i.e. 200391546). Write the LIMS I.D. number on the 24-Hr Report.

7.3.9 Use a LIMS report to create a summary of PM2.5 samples logged into LIMS for the selected date. The analyst reviews this report, checking for incomplete and flagged entries.

7.3.10 R&P samples shipped and stored at a constant 4°C or lower before equilibration must be weighed within 30 days of the sampling date. Samples shipped and stored at a constant range between 4°C and 25°C before equilibration must be weighed within 10 days of the sampling date, unless the average ambient temperature during sampling was above 25°C, as reported on the 24-Hr Report form by the site operator. In that case, the sample must be weighed within 30 days of the sampling date. Samples received at temperatures above 25°C must be weighed within 10 days of the sampling date. Any samples exceeding the time limit between sampling and post-weighing must be noted on the matching 24-Hr Report and reported to the lab supervisor. LIMS will automatically invalidate these samples.

Temperature at Receipt	Days Allowed Between Sampling and Weighing
4°C or lower	30
Between 4°C and 25°C Average ambient temperature >25°C	30
Between 4°C and 25°C Average ambient temperature <25°C	10
25°C or higher	10

7.3.11 The temperature of SASS™ samples received in the laboratory is monitored by the speciation program personnel. SASS™ samples must be weighed within 10 business days of receipt in the laboratory.

7.4 POST-WEIGHING OF FIELD SAMPLES

7.4.1 Manual Balance

7.4.1.1 Ensure that the temperature and the relative humidity of the balance room have remained (and are currently) within the allowable limits (see Section 6.1 for limits) throughout the preceding 24 hours. Also, make certain the selected filters have equilibrated in the balance room for at least 24 hours.

7.4.1.2 Clean the micro-balance weighing chamber with a fine brush, if necessary. Clean the surfaces near the micro-balance with antistatic solution or disposable lab wipes moistened with isopropanol. Clean the forceps used for handling the mass reference weights and the

filters prior to each weighing session. Ensure that all forceps in use are dry.

7.4.1.3 Generate a work list from LIMS. Upper and lower control working standards are automatically inserted by LIMS at the beginning and at the end of the worklist. After each duplicate sample, the working standards are alternately inserted automatically by LIMS. There must be a duplicate mass rate of ≥10%. Duplicates are automatically inserted by LIMS after every nine filters using the first filter of each batch.

7.4.1.4 After the samples have equilibrated for at least 24 hours, arrange the 24-Hr Reports of the samples that are to be post-weighed in the same order as the LIMS work list. Affix matching barcodes labels on an equal number of petri slides. Remove the sampled filters from the conditioning cabinet. Match up the filter cassette I.D. numbers with the correct 24-Hr Reports and petri slides. For samples received from SASS™ samplers, arrange the 24-Hr Report forms and petri slides in same order as the LIMS worklist.

7.4.1.5 Perform an internal and external calibration of the micro-balance as described in Section 8.1.1.1. prior to beginning the weighing session.

7.4.1.6 Weigh the four lab blanks. See section 8.2.2 for limits.

7.4.1.6.1 Using forceps, grip a filter only by the outer polypropylene support ring and place the filter onto a static neutralizer strip for a minimum of 30 seconds. Then, using forceps, pass the filter through the static strip ionizer stand 3 times before placing on the balance.

7.4.1.6.2 Leave the lab blank on the balance for 30 seconds. Transfer the weight value into LIMS. Using forceps, remove the lab blank and return it to the petri-slide. Transfer all data to LIMS.

7.4.1.7 Post weigh the samples in the same order as the worklist. Using forceps, place a filter on the balance pan and close the chamber. At the end of 30 seconds, transfer the mass data into the LIMS worksheet. Record on the 24-Hr Report form the following:

7.4.1.7.1 Post-weight mass of the filter.

7.4.1.7.2 Date of post-weight measurement.

7.4.1.7.3 Analyst's initials.

7.4.1.8 Place the weighed filter into the petri dish.

7.4.1.9 Repeat for subsequent filters and duplicate weighings. If the original and duplicate mass values differ by ±15 µg, check the two mass results, and reweigh the filter, if necessary. If the difference is still not ±15 µg, consult with the lab supervisor.

7.4.1.10 Working standards are automatically inserted by LIMS at the beginning and at the end of the worklist. After each duplicate sample, the upper and lower control working standards are alternately inserted automatically by LIMS into the worklist.

7.4.1.11 Repeat steps for subsequent batches of filters.

7.4.1.12 At the end of the weighing session, weigh the lower and upper control working standard weights. After the last control has been transferred to the worklist, examine the data for errors and correct, if necessary.

7.4.1.13 After the data transfer into LIMS is complete, run the PM2.5-Post Weight Summary Report. Confirm that the percentage of duplicates to filters is not less than 10 percent and that there are no duplicates out of range.

7.4.1.14 Make sure field and/or trip blanks are within control. If not, reweigh the blanks and indicate that the post weight was verified in the comments section of the 24-Hr report form. Refer to sections 8.2.3 and 8.2.4 for limits and actions.

7.4.1.15 Exceedences of the National Ambient Air Quality Standard for PM2.5 should be reported to the lab supervisor as soon as possible. If the mass variation between the pre-weight and post-weight of any blank filter is greater than 30 µg, notify the site operator and lab supervisor.

7.4.1.16 Place the weighed filter into a petri slide, close tightly, and store at 4°C for at least one year after sampling. After one year, the samples can be stored at room temperature. Samples must be kept for the current year plus five years.

7.4.2 Robot Balance

7.4.2.1 Ensure that the temperature and the relative humidity of the Balance Room have remained (and are currently) within the allowable limits

(see Section 6.1 for limits) throughout the preceding 24 hours. Also, make certain the selected filters have equilibrated in the balance room for at least 24 hours.

7.4.2.2 Clean the micro-balance weighing chamber with a fine brush, if necessary. Clean the surfaces near the micro-balance with antistatic solution or disposable lab wipes moistened with isopropanol. Clean the forceps used for handling the mass reference weights and the filters prior to each weighing session. Ensure that all forceps in use are dry.

7.4.2.3 Generate a work list from LIMS. The upper and lower control working standards are automatically inserted by LIMS at the beginning and at the end of the worklist. After each duplicate sample, the working standards are alternately inserted automatically by LIMS. There must be a duplicate mass rate of ≥10%. Duplicates are automatically inserted by LIMS after every nine filters using the first filter of each batch.

7.4.2.4 After the samples have equilibrated for at least 24 hours, arrange the 24-Hr Reports of the samples that are to be post-weighed in the same order as the LIMS work list. Affix matching barcodes labels on an equal number of petri slides. Remove the sampled filters from the conditioning cabinet. Match up the filter cassette I.D. numbers with the correct 24-Hr Reports and petri slides.

7.4.2.5 Perform an internal and external calibration of the micro-balance as described in Section 8.1.1.2 prior to beginning the weighing session. Transfer the values to LIMS.

7.4.2.6 Perform the Calibration/Lab blank weighing. Refer to Section 7.2.2.4. Send the values to LIMS.

7.4.2.7 Perform the post weight weighing.

7.4.2.7.1 Load the filters into the robot. Refer to Section 7.2.2.8 for filter loading procedure.

7.4.2.7.2 Use robot software and LIMS to set up the run and ensure that the physical filter positions match up with the software filter positions.

7.4.2.7.3 Examine and check for errors. Make any necessary corrections or appropriate reweighs.

7.4.2.7.4 Transfer the data into LIMS.

7.4.2.7.5 Make sure field and/or trip blanks are within control. If not, reweigh the blanks and indicate that the post weight was verified in the comments section of the 24-Hr report form. Refer to sections 8.2.3 and 8.2.4 for limits and actions.

7.4.2.7.6 Exceedences of the National Ambient Air Quality Standard for PM2.5 should be reported to the lab supervisor as soon as possible. If the mass variation between the pre-weight and post-weight of any blank filter is greater than 30 μg, notify the site operator and lab supervisor.

7.4.2.7.7 Record on the 24-Hr Report form the following:

7.4.2.7.7.1 Post-weight mass of the filter.

7.4.2.7.7.2 Date of post-weight.

7.4.2.7.7.3 Analyst's initials.

7.4.2.7.7 Place the weighed filter into a petri slide, close tightly, and store at 4°C for at least one year after sampling. After one year, the samples can be stored at room temperature. Samples must be kept for the current year plus five years.

7.4.2.8 After the data transfer into LIMS is complete, run the PM2.5-Post Weight Summary Report. Confirm that the percentage of duplicates to filters is not less than 10 percent and that there are no duplicates out of range.

8.0 QUALITY CONTROL

8.1 Instrument Calibration and Verification

8.1.1 Analytical Micro-Balance

Annual Calibration

The micro-balance must be calibrated and certified annually. The calibration will be traceable to NIST traceable mass reference standards. If the micro-balance is found to be out of calibration, it will be recalibrated according to the manufacturer's directions.

Daily Calibration

The micro-balance must be calibrated prior to and during each weighing session. This will be done by internal and external micro-balance calibrations and by alternating mass reference standards.

Quarterly Calibration

Each quarter, the balance must be checked against certified mass reference standards (see Section 8.1.2). The primary standards are weighed on the balance five times and their average mass readings are compared to their certified mass values. If the average mass readings are not within ±0.003 mg of the certified masses, the balance must be recalibrated, and the primary standards must be weighed again. If the difference in mass is still greater than ±0.003 mg, the balance needs to be adjusted and recertified.

If the value of a mass reference standard differs by more than ±0.003 mg, reweigh the reference standards. If the discrepancy remains, contact the ILS supervisor for direction.

Below are the procedures for calibrating the manual and the robot micro-balances:

8.1.1.1 Manual Balance

8.1.1.1.1 Prior to any filter weighing session, the micro-balance must be calibrated. First, check the micro-balance base level and adjust as needed. To ensure maximum stability, the micro-balance must remain on at all times.

8.1.1.1.1.2 Internal Calibration: Press the "Internal Calibration" key on the balance liquid crystal display (LCD). The LCD should display "OK" when the balance has finished the internal calibration. Press the "OK" button and the balance is now ready for external calibration.

8.1.1.1.1.3 External Calibration: Place the lower working mass reference standard control weight onto the micro-balance pan with non-metallic forceps. After 30 seconds, send the mass value to LIMS. Remove the lower reference standard weight and place the upper reference standard weight on the balance for 30 seconds. Send the mass value to LIMS. Also, record

and send the date, temperature and relative humidity of the balance room to LIMS.. Remove the upper calibration weight. External calibration must be performed daily for each day that filters are pre-weighed and/or post-weighed.

8.1.1.2 Robot Balance

8.1.1.2.1 Prior to any filter weighing session, the micro-balance must be calibrated. To ensure maximum stability, the micro-balance must remain on at all times.

8.1.1.2.2 Internal Calibration: The Robot will automatically conduct an internal calibration once the "Calibration/Lot Blank" run is activated.

8.1.1.2.3 External Calibration: The Robot will automatically conduct an external calibration once the Calibration/Lab Blank run is activated. The Robot will weigh the both the lower and upper control standard weights for 30 seconds each. Record the date, temperature, relative humidity into LIMS.

8.1.2 Mass Reference Standards

Mass reference standards should be non-corroding and be certified as traceable to NIST. Lower and upper control weights that "bracket" the expected filter masses (i.e. 100 mg and 500 mg) are needed for each balance. Two sets are needed, one set as working standards and one set as primary standards. The weights should be ASTM Class 1 category.

The working mass reference standards will remain in the balance room. The primary mass reference standards will be sent out to be recertified by an outside source annually. The outside source must provide a NIST certificate for each mass reference standard.

The working mass reference standards will be used during each weighing sessions as controls. The two standards that are selected to "bracket" the expected mass of the samples will be weighed at the beginning and the end of each session. The standards will also alternately be weighed after each duplicate (or tenth filter) weighing.

Each quarter, the masses of the working mass reference standards are compared to the primary mass reference standards. Weigh each mass standard five times and record an average. The primary mass standard average is used

to check the performance of the micro-balance (see Section 5.1.1). The working mass standard average is used to determine the certified working mass range for the mass reference standards. This range will be used as controls during weighing sessions. During the quarterly calibration check, if the working standard masses are outside of ±0.003 mg of the certified value, submit a request to the ILS laboratory supervisor that the certified working mass be adjusted.

8.1.3 Temperature and Percent Relative Humidity Recorder

The balance room's percent relative humidity must be maintained at a mean value range of 30-40% and its air temperature must be maintained at a mean value range of 20.0-23.0°C. If the balance room does not meet these specifications, notify the lab supervisor and schedule a repair visit. No weighing can occur until balance room is back in range for 24 hours.

Each quarter the temperature and humidity recorder must be checked against a NIST traceable temperature and humidity instrument (standard). The standard must be recertified annually by an outside source. Record the temperature and humidity readings about every two minutes from both the record and the standard at least ten times. Determine the averages of the temperature and humidity for both the recorder and the standard. Subtract the averages for the recorder from the averages for the standard. The differences should not be greater than ±2.0°C for temperature and ±2.0% for relative humidity. If the differences are greater than ±2.0, notify the lab supervisor and schedule a repair visit.

8.2 BLANKS

Four types of blanks are used in the method: Lot Blanks, Laboratory Blanks, Field Blanks and Trip Blanks.

8.2.1 Lot Blanks:

Randomly select one set of nine filters as lot blanks from each new lot received for each micro-balance. Place the filters in individual containers. Equilibrate the exposed filters in a filter equilibration cabinet in the balance room that allows air circulation, but still reduces extraneous airborne particles from settling on filters. Weigh lot blanks every 24 hours on each manual balance and the robot balance. Record the lot number, filter number, mass, and dates of the lot blanks and send them to the designated LIMS staff to set up a Lot Blank Determination worksheet. Once the mass difference between weighing is less than 0.015 mg for all nine lot blanks, the filters have stabilized. Note the time taken from initial exposure of the filters to attainment of mass stability. This information is designated as the minimum equilibration period required before filters from the same lot can be pre-

weighed and used for routine sampling. Once this minimum equilibration period is determined, designate four of the nine lot blanks to become lab blanks which are set aside for long term exposure in the same equilibration cabinet where routine samples, field blanks, and trip blanks are equilibrated prior to pre- or post-weighing. Set aside the five remaining lot blanks as back up lab blanks, in case a working lab blank is damaged.

8.2.2 Lab Blanks:

Weigh the four lab blanks prior to each weighing session day. The lab blanks are used to determine if there is any contamination in the conditioning area. Acceptable range is "expected lot blank weight" ± 0.015 mg. Lab blank mass values are reviewed as needed and the reference values are adjusted if the lot blank weight is outside the acceptable range. The new "expected lot blank weight" is based on the average of the previous week's lot blank weighings, or as determined by the analyst.

8.2.3 Field Blanks:

Field blanks are conditioned, unexposed filters that are used to determine whether contamination occurs during sampling. Field blanks are included in weighing sessions and handled in the same manner as a filter destined for sampling. Site operators load the field blanks and run them through a sampler without flow.

Ensure that field blanks are included in at least 10 percent of the sampler's operating frequency. A good rule is the following:

for daily schedule—10 field and trip blanks per quarter
for 1 in 3 schedule—4 field and trip blanks per quarter
for 1 in 6 schedule—3 field and trip blanks per quarter
for 1 in 12 schedule—1 field and trip blank per quarter

If the weight change between pre- and post- field blank weighings exceeds 0.030 mg, contamination may have occurred. Verify the mass value by reweighing the filter. If the mass difference still occurs, notify the site operator.

8.2.4 Trip Blanks:

Trip blanks are conditioned, unexposed filters that are used to determine whether contamination occurs during the transportation of the filters. Trip blanks are included in weighing sessions and handled in the same manner as a filter destined for sampling. The trip blanks travel with the filters, but are not loaded into the sampler.

Ensure that trip blanks are included in at least 10 percent of the sampler's operating frequency (see Section 8.2.3 Field Blanks for suggested scheduling).

If the weight change between pre- and post- trip blank weighings exceeds 0.030 mg, contamination may have occurred. Verify the mass value by reweighing the filter. If the mass difference still occurs, notify the site operator.

9.0 DATA HANDLING

9.1 Data handling for this method assumes a familiarity with LIMS. Follow the procedures outlined in the LIMS SOP to accurately transfer mass values.

9.2 After post-weighing the samples, file the 24-Hr Report forms. Archive the 24-Hr Report forms for 5 years, plus the current year.

9.2 After each pre-weigh and post-weigh session, print a summary report from LIMS. Review, initial and date the report. Archive the reports for five years, plus the current year.

10.0 REVISION HISTORY

The change from Revision 0.0 to Revision 1.0 addresses the addition of the Automatic Filter Weighing System, updates to the filter static charge neutralization procedure, new relative humidity/temperature recorders, updates in 40 CFR, Appendix L, and clarification of procedures.

11.0 CALCULATIONS

The equation to calculate the mass of fine particulate matter collected on a Teflon filter is seen below:

$$M_{2.5} = (M_f - M_i) \times 10^3 \qquad \text{Equation 1}$$

where,

$M_{2.5}$	=	total mass of fine particulate collected during sampling period (μg)
M_f	=	final mass of the conditioned filter after sample collection (mg)
M_i	=	initial mass of the conditioned filter before sample collection (mg)
10^3	=	unit conversion factor for milligrams (mg) to micrograms (μg)

According to 40 CFR Part 50, Appendix L, PM2.5 samplers are required to provide measurements of the total volume of ambient air passing through the sampler (V) in cubic meters at the actual temperatures and pressures measured during sampling. Use the following formula if V is not available directly from the sampler:

$$V = Q_{avg} \times t \times 10^{-3} \qquad \text{Equation 2}$$

where,

V	=	total sample volume (m^3)
Q_{avg}	=	average flow rate over the entire duration of the sampling period (L/min)
t	=	duration of sampling period (min)
10^{-3}	=	unit conversion factor for liters (L) into cubic meters (m^3)

The equation outlined below can be used to determine $PM_{2.5}$ mass concentration:

$$PM_{2.5} = \frac{M_{2.5}}{V} \qquad \text{Equation 3}$$

where,

$PM_{2.5}$	=	mass concentration of PM2.5 particulates ($\mu g/m^3$)
$M_{2.5}$	=	total mass of fine particulate collected during sampling period (μg)
V	=	total volume of air sampled (m^3)

12.0 REFERENCES

Code of Federal Regulations, Title 40, Part 50, Appendix L.

Chemical Hygiene Plan for 13th and T Facility Monitoring and Laboratory Division. California Air Resources Board, July 2005.

Laboratory Information Management System Standard Operating Procedure. California Air Resources Board, 2014

Quality Assurance Guidance Document 2.12, Monitoring $PM_{2.5}$ in Ambient Air Using Designated Reference or Class I Equivalent Methods. U.S. Environmental Protection Agency, November 1998.

Quality Assurance Guidance Document EPA-454/B-12-003, PM2.5 Chemical Speciation Sampling, U.S. Environmental Protection Agency, June 2012.

Quality Assurance Handbook Volume II, Appendix D. U.S. Environmental Protection Agency, May 2013.

Air Resources Board

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Sacramento, California 95812 • www.arb.ca.gov

Matthew Rodriquez
Secretary for
Environmental Protection

Edmund G. Brown Jr.
Governor

TO: Brenda Saldana, Manager
Inorganics Laboratory Section

FROM: Patrick Rainey, Manager
Quality Management Section

DATE: September 2, 2015

SUBJECT: STANDARD OPERATING PROCEDURE (SOP) FOR THE DETERMINATION OF PM2.5 MASS IN AMBIENT AIR BY GRAVIMETRIC ANALYSIS

Thank you for your submission of the addendum (see attached) to the Standard Operating Procedure (SOP) for the Determination of PM2.5 Mass in Ambient Air by Gravimetric Analysis. The Quality Management Section has reviewed the addendum along with the SOP and determined that it covers all of the required elements. The addendum is approved.

Please direct comments or questions to Aman Bains at (916) 323-0032 or by email at aman.bains@arb.ca.gov.

Attachment

cc: Mike Miguel, Chief
Quality Management Branch

Aman Bains
Quality Management Section

The energy challenge facing California is real. Every Californian needs to take immediate action to reduce energy consumption. For a list of simple ways you can reduce demand and cut your energy costs, see our website: http://www.arb.ca.gov.

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State of California
California Environmental Protection Agency
Air Resources Board
MLD/QMS-066 (NEW 4/14)

QUALITY MANAGEMENT DOCUMENT ADDENDUM

Section 1. ARB Document
☐ Quality Management Plan (QMP)
☐ Quality Assurance Project Plan (QAPP)
☒ Standard Operating Procedure (SOP)

Section 2. Information		
Submitter Name:	Alicia Adams	
Submitter Signature/Date:		9/1/2015

Section 3. Document Title *(specify exact title, revision #, and date of ARB Document(s) that your District proposes to modify)*	Date
SOP MLD 055 Revision Number 1.0	3/24/14

Section 4. Revision(s)
(specify exact section(s), page number(s) and language in existing ARB document that will be modified and then specify modification (including any spreadsheets or forms).

Section 6.0 EQUIPMENT

ARB modification to section 6.0 – Add 6.26 Temperature measuring device, such as a temperature probe. Each year, the device should be certified as traceable to National Institute of Standards and Technology (NIST).

Section 7.3.1, page 9, states "Upon receipt of samples from the field, examine the Temperature Threshold Indicators inside the filter-shipping canister. These determine what receiving temperature is entered on the 24-Hr Reports with this canister. If both indicators have not turned pink, enter "<4." If the 5°C indicator is pink, but the 25°C indicator has not changed color, enter "4-25." If both have turned pink, enter ">25." Also enter the current date, time, and your initials in the appropriate spaces for the "Sample Received at Lab" line for each of the 24-Hr Reports."

ARB modification to Section 7.3.1 – Add 7.3.1.1 Upon receipt of samples from the field, if Temperature Threshold Indicators are missing, inactivated, or reacted (pink), place temperature measuring device in shipping box, next to the canister. Quickly replace the box lid and allow the thermometer reading to stabilize, about 5 to 10

minutes. If the temperature reading is below 4.0 °C, write "<4" in the appropriate space on the 24-Hr Report. If the temperature is between 4 and 25 °C, enter "4-25." If the temperature is greater than 25 °C, enter ">25." Also enter the current date, time, and your initials in the appropriate spaces for the "Sample Received at Lab" line for each of the 24-Hr Reports. In this instance, use of the thermometer will indicate the official receiving shipment temperature.

Section 5. Justification for Deviation(s)
(provide explanation of why modification(s) to existing ARB document is necessary)

ILS studies have shown no discrepancies between the temperature strips and the thermometer readings. All incoming boxes received during the study time frame were shipped with properly activated temperature strips at or below 4 degrees Celsius.

Section 6. Attachment(s) ☐ *(specify attachment titles and number of pages, include modified spreadsheets or forms)*	**# of Pages**

Section 7. ARB Approval		
Name/Phone Number:	Patrick Rainey	916 327-4756
Title:	Manager, Quality Management Section	
Signature/Date:		9/1/2015
Addendum Number	A07 MLD055.1	

Completed form must be scanned/emailed or mailed to:

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Air Resources Board

Mary D. Nichols, Chair
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Sacramento, California 95812 • www.arb.ca.gov

Matthew Rodriquez
Secretary for
Environmental Protection

Edmund G. Brown Jr.
Governor

TO: Brenda Saldana, Manager
Inorganic Laboratory Section

FROM: Patrick Rainey, Manager
Quality Management Section

DATE: November 22, 2016

SUBJECT: Standard Operating Procedure (SOP) for the Determination of PM2.5 Mass in Ambient Air by Gravimetric Analysis

Thank you for your submission of the addendum (see attached) to the Standard Operating Procedure (SOP) for the Determination of PM2.5 Mass in Ambient Air by Gravimetric Analysis (MLD 055 R1). The Air Resources Board's (ARB) Quality Management Branch reviewed the SOP addendum and determined that it covers all of the required elements. The addendum is approved for use in conjunction with SOP MLD 055 R1.

Please direct comments or questions to Kyle Vagadori at 916-445-9391 or by email at kyle.vagadori@arb.ca.gov.

Attachment

cc: Mike Miguel, Chief
Quality Management Branch

Kyle Vagadori
Quality Management Section

The energy challenge facing California is real. Every Californian needs to take immediate action to reduce energy consumption. For a list of simple ways you can reduce demand and cut your energy costs, see our website: http://www.arb.ca.gov.

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State of California
California Environmental Protection Agency
Air Resources Board
MLD/QMS-066 (NEW 4/14)

QUALITY MANAGEMENT DOCUMENT ADDENDUM

Section 1. ARB Document
☐ Quality Management Plan (QMP)
☐ Quality Assurance Project Plan (QAPP)
☒ Standard Operating Procedure (SOP)

Section 2. Information		
Submitter Name:	Alicia Adams	
Submitter Signature/Date:	[signature] 11-14-16	11/14/16

Section 3. Document Title *(specify exact title, revision #, and date of ARB Document(s) that your District proposes to modify)*	Date
SOP MLD 055 Revision Number 1.0	3/4/14

Section 4. Revision(s)
(specify exact section(s), page number(s) and language in existing ARB document that will be modified and then specify modification (including any spreadsheets or forms).

Section 7.2 PRE-WEIGHING OF UNSAMPLED FILTERS

Section 7.2.1 Manual Balance

Current Section 7.2.1.1 Ensure that the temperature and the relative humidity of the Balance Room have remained and are currently within the allowable limits (see Section 6.1 for limits) throughout the preceding 24 hours. Also, make certain the selected filters have been conditioned for at least the minimum time needed to attain mass stability, as determined from the filter lab blanks.

ARB Modification to Section 7.2.1.1 Ensure that the temperature and the relative humidity (RH) of the Balance Room have remained, and are currently, within the allowable limits throughout the preceding 24 hours prior to the start of the weigh session. The allowable limits for temperature averages are 20.0-23.0 °C, with a temperature control of ±2 °C. The allowable limits for RH averages are 30-40%, with a RH control of ±5%. The average values are determined by using the average formula in Excel. The control values are determined by using the standard deviation formula in Excel. Also, make certain the selected filters have been conditioned in the Balance Room for at least 24 hours (See Section 7.1.1).

Following are the procedures for using the Omega data logger to ensure temperature and RH

conditions are met and for calculating the preceding 24-hour temperature and RH averages and standard deviations:

7.2.1.1.1 From the sensor set up page, select "Chart". Visually review the chart to determine if the temperature and RH conditions are met. Print the chart, write initials and date, and file the chart in the appropriate binder.

7.2.1.1.2 To calculate the preceding 24-hour temperature and RH, from the sensor URL setup page, select "Recording" then "Data Retrieval."
Retrieve data for the prior and current day through two separate files by selecting the correct days and "Start Data Retrieval." When asked to open or save "downloadRecordData.data," select "Save." The file is now saved in the computer downloads file. The current and prior day both have to be retrieved in order for the sensor data to be downloaded for the specific range of time needed (i.e. 10:00 am 1/4/17 to 10:00 am 1/5/17).

7.2.1.1.3 Open Excel, and select to "Open" a file. Retrieve the extracted data from the first computer download file and "Open." This will prompt the delimited "Text Import Wizard."

7.2.1.1.4 Choose file type as "Delimited." Select "Next." For Delimiters, uncheck any auto populated box, and only check "Semicolon." Select "Next" and "Finish." Follow this same process for the second data file.

7.2.1.1.5 Copy and paste the two files together within one spreadsheet. The amount of data will far exceed what's needed for the 24-hour calculations. The recorder takes a reading every 2.5 minutes, which equates to 576 data points in a 24-hour period. This is the amount of data points to be used in the calculations. Take the most current data point, and preceding data points to cover the full preceding 24-hour time frame. For example, if a weigh session is set to start at 10:00 am on 1/5/17, data points would need to be collected from 10:00 am on 1/4/17 through 10:00 am on 1/5/17. This would cover the preceding 24-hour time frame.

7.2.1.1.6 Delete Dew Point column and remove the letter "C" from the temperature column.

7.2.1.1.7 Using Excel formulas (i.e. AVERAGE[Cell Start:Cell Stop] and STDEV[Cell Start:Cell Stop]), calculate averages and standard deviations for both temperature and relative humidity for the 576 data points to cover the preceding 24-hour period.

7.2.1.1.8 Save the calculated data in the Temp/RH File on the computer Desktop.

7.2.1.1.9 Enter these calculated averages for temperature and relative humidity in the daily calibration worksheet in LIMSLink.

If the temperature and RH conditions are met per section 7.2.1.1, start the weighing session. Continually monitor the temperature and RH during the weighing session. If either the temperature or RH appears to be trending towards being out of compliance or a spike occurs, assess if the weighing session should be stopped. Stop the weigh session if the temperature and/or RH move out of compliance.

Section 7.2.2 Robot Balance

Current Section 7.2.2.1 Ensure that the temperature and the relative humidity of the balance room have remained (and are currently) within the allowable limits (see Section 6.1 for limits) throughout the preceding 24 hours. Also, make certain the selected filters have been conditioned for at least the minimum time needed to attain mass stability, as determined from the filter lot blanks.

ARB Modification to Section 7.2.2.1 Ensure that the temperature and the relative humidity (RH) of the Balance Room have remained, and are currently, within the allowable limits throughout the preceding 24 hours prior to the start of the weigh session. The allowable limits for temperature averages are 20.0-23.0 °C, and a temperature control of ±2 °C. The allowable limits for RH averages are 30-40%, and a RH control of ±5%. Also, make certain the selected filters have been conditioned in the Balance Room for at least 24 hours (See Section 7.1.1).

To calculate preceding 24-hour temperature and relative humidity, see section 7.2.1.1.1 through 7.2.1.1.9.

Section 7.4 POST-WEIGHING OF FIELD SAMPLES

Section 7.4.1 Manual Balance

Current Section 7.4.1.1 Ensure that the temperature and the relative humidity of the balance room have remained (and are currently) within the allowable limits (see Section 6.1 for limits) throughout the preceding 24 hours. Also, make certain the selected filters have equilibrated in the balance room for at least 24 hours.

ARB Modification to Section 7.4.1.1 Ensure that the temperature and the relative humidity (RH) of the Balance Room have remained, and are currently, within the allowable limits throughout the preceding 24 hours prior to the start of the weigh session. The allowable limits for temperature averages are 20.0-23.0 °C, with a temperature control of ±2 °C. The allowable limits for RH averages are 30-40%, with a RH control of ±5%. The average values are determined by using the average formula in Excel. The control values are determined by using the standard deviation formula in Excel. Also, make certain the selected filters have been conditioned in the Balance Room for at least 24 hours (See Section 7.1.1).

To calculate prior 24-hour temperature and relative humidity, see section 7.2.1.1.1 through 7.2.1.1.9.

If the temperature and RH conditions are met per section 7.2.1.1, start the weighing session. Continually monitor the temperature and RH during the weighing session. If either the temperature or RH appears to be trending towards being out of compliance or a spike occurs, assess if the weighing session should be stopped. Stop the weigh session if the temperature and/or RH move out of compliance.

7.4.1.1.1 Once the LIMSLink post-weigh worksheet is created, LIMSLink will automatically upload the calculated temperature and humidity values for each sample from their pre-weigh session. LIMSLink identifies the post-weight sample by its barcode, and retrieves the calculated temperature and humidity from that filter's pre-weight. These values were entered into LIMSLink during the pre-weight session (See Section 7.2.1.1.9). Filters must be conditioned at the same conditions (humidity within ±5 relative humidity percent) before both pre- and post-sampling weighings. LIMSLink will automatically compare the pre- and post-weight calculated values for each sample. If the pre- and post-sampling RH conditions are not within ±5% RH, LIMSLink will flag the sample. In the event a sample exceeds the control requirement, the filter should be removed from the worksheet and weighed another day. If subsequent daily

calculations still don't meet the control requirements for a filter, the filter should be weighed by the weigh-by date. If the filter doesn't meet the relative humidity control requirements, it should be weighed, but will be invalid.

Section 7.4.2 Robot Balance

Current Section 7.4.2.1 Ensure that the temperature and the relative humidity of the Balance Room have remained (and are currently) within the allowable limits (see Section 6.1 for limits) throughout the preceding 24 hours. Also, make certain the selected filters have equilibrated in the balance room for at least 24 hours.

ARB Modification to Section 7.4.2.1 Ensure that the temperature and the relative humidity of the Balance Room have remained, and are currently, within the allowable limits throughout the preceding 24 hours. This is done by manually extracting the data from the Omega data logger and calculating the average and standard deviation of temperature and relative humidity for the preceding 24 hours prior to the weigh session. Additionally, daily temperature and relative humidity charts are reviewed, printed, initialed, dated, and filed. Charts are checked for visible trends residing outside the required limits, in which case any weighing would be postponed. The allowable limits for temperature averages are 20.0-23.0 °C, and a temperature control of ±2 °C. The allowable limits for relative humidity averages are 30-40%, and a relative humidity control of ±5%. Also, make certain the selected filters have been conditioned in the Balance Room for at least 24 hours. To calculate prior 24-hour temperature and relative humidity, see section 7.2.1.1.1 through 7.2.1.1.9.

7.4.2.1.1 Once the LIMSLink post-weigh worksheet is created, upon loading the pre-weight information, LIMSLink will automatically upload the calculated temperature and humidity values for each sample from their pre-weigh session. LIMSLink identifies the post-weight sample by its barcode, and retrieves the calculated temperature and humidity from that filter's pre-weight. These values were entered into LIMSLink during the pre-weight session (See Section 7.2.1.1.9). Filters must be conditioned at the same conditions (humidity within ±5 relative humidity percent) before both pre- and post-sampling weighings. LIMSLink will automatically compare the pre- and post-weight calculated values for each sample. If the pre- and post-sampling RH conditions are not within ±5% RH, LIMSLink will flag the sample. In the event a sample exceeds the control requirement, the filter should be removed from the worksheet and weighed another day. If subsequent daily calculations still don't meet the control requirements for a filter, the filter should be weighed by the weigh-by date. If the filter doesn't meet the relative humidity control requirements, it should be weighed, but will be invalid.

Section 5. Justification for Deviation(s)
(provide explanation of why modification(s) to existing ARB document is necessary)

SOP Amendment to further explain and detail current procedures for calculating Balance Room temperature and humidity requirements per current EPA recommendations and revisions to the QA Guidance Document 2.12, January 2016.

State of California
California Environmental Protection Agency
Air Resources Board
MLD/QMS-066 (NEW 4/14)

Section 6. Attachment(s) ☐ *(specify attachment titles and number of pages, include modified spreadsheets or forms)*	**# of Pages**

Section 7. ARB Approval		
Name/Phone Number:	Patrick Rainey	916 327-4756
Title:	Manager, Quality Management Section	
Signature/Date:		11/22/16
Addendum Number	A15 MLD055	

Completed form must be scanned/emailed or mailed to:

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prainey@arb.ca.gov