<u>PM Test Method Workgroup Summary of Recommendations</u>
March 30, 2017 (revised with priority ranking and proportionality)

To the EPA certified test lab community: we are seeking comments and feedback on recommendations for modifying particulate matter (PM) dilution tunnel measurements contained in this document. The Cordwood Test Method Steering Committee convened a PM test method workgroup that held several calls over the last few months to discuss opportunities to increase the precision of dilution tunnel PM measurement methods. The workgroup developed a list of summary recommendations. Specific recommendations are grouped by topic areas. Topics that the workgroup discussed but did not need develop recommendations for modifications are not listed here but can be provided, if requested. If labs are interested, call(s) can be convened to discuss the rationale behind the recommendations and discuss the feasibility of implementing these recommendations.

After this list of recommendations was developed, the workgroup assigned a priority to each of them on a scale of 1 (most important to implement) to 3 (least important). None of the recommendations were ranked as 3. All but the following listed below were ranked as 1 except as noted.

1. **Water/Humidity/Temperature control in dilution tunnel and at the sample filter.**

Liquid water should not be present anywhere in the sampling system for a valid sample – both in the dilution tunnel and at the sample filter / filter holder or anywhere in the sampling train. Temperature and RH (and calculated dew point temperature) should be measured in the dilution tunnel near the location of the sample probe. A high quality sensor should be used that is rated above the highest expected tunnel temperature. The sensor accuracy specifications should be at least +/- 0.5 ºC for temperature and +/- 1% RH (between 5 and 95%). Dew point temperature (calculated from T and RH) should be reported by the sensor. One example of a suitable sensor is the Omega HX-80A series, specifically the HX85A (probe only) or HX86A (probe and display). Saturated salt solutions can be used for RH calibration checks (saturated potassium chloride yields 85% RH at room temperature).

The workgroup recommends a maximum RH measured in the tunnel of 90% to protect against possible condensation anywhere in the tunnel. Since tunnel temperature may be hotter than laboratory temperature, it is also necessary to maintain the filter temperature at least 2 ºC higher than the tunnel dew point temperature to avoid condensation at the filter (a 2 ºC difference between dew point temperature and air (dry bulb) temperature is ~ 89% RH at ~ 85 ºF).

Filter temperature should be measured with a device that has a specified accuracy of 0.5º C (1º F) or better. If this cannot be achieved with a thermocouple, a thermistor or RTD sensor can be used. The workgroup agrees with the filter temperature range under consideration by EPA of 80º to 90 ºF, using a temperature sensor with 0.5 ºC accuracy or better.

A maximum tunnel temperature of 100 ºF is recommended; this is 10 ºF above the existing maximum filter temperature (90 ºF) limit. (Priority of 2)

Any exceedances of these limits should be explicitly noted in the test report. When the sampling train is disassembled, it should be inspected for any signs of liquid water in the probe, the filter holders, or on the filters. If either the tunnel RH limit or the tunnel dew point/filter temperature minimum difference are not met, the test report should explicitly note the results of the sampling train liquid water inspection.

2. **Filter Media:** To minimize interferences from adsorbed organic and acidic gases and water vapor, Pallflex® Emfab™ (TX40) is recommended over the glass fiber filter media usually used. Cost (~ $1.70 per filter) is more than glass fiber.

3. **Additional operational parameters to be included in a test report but not controlled for at this time** (* indicates to logged electronically where possible):

*Pump vacuum (pressure drop across the filter)
*Filter sample volumetric flow rate (actual temperature and pressure not STP)
*Filter temperature
*Tunnel temperature and RH (and dew point temperature)
Filter face velocity (flow rate divided by filter's actual particle loading area)
Front filter net mass loading (mg)
Average tunnel PM concentration (mg/m^3)
*Tunnel flow rate
Tunnel residence time (should be in the range of 1 to 3 seconds)
Estimate of stack flow
Estimated tunnel dilution ratio range

4. **Filter equilibration / conditioning post-sample collection:**

General recommendation: use conditions and times similar to those used for the ambient PM2.5 FRM method (40 CFR Part 50, Appendix L). Do not use desiccation; equilibrate at RH between 30 and 40% for at least 24 hours. A saturated salt solution of magnesium chloride (33% RH) can be used to achieve this in a small chamber if a controlled weighing room that meets the PM2.5 FRM specifications is unavailable. When possible (priority of 2), front filter mass measurements (mg) should be made and reported soon after end of sampling ("0-day") and the next day ("1-day", ~ 24 to 30 hours after sampling) to document filter mass loss over time. The date and time of all filter weighings should be recorded and reported with test results. Final filter weights would still be made as described in EPA method 5G.

Loss of filter mass over time during post-sample equilibration could be water and/or semi-volatile PM. It would be useful to do some tests to determine if water is a major component of the loss or not, but this is beyond the scope of the workgroup.

5. **Size-cut Cyclone:** a PM10 (10 µm) size selective inlet upstream of the filter is recommended to exclude large ash particles from the measurement. Method 201 (U.S.EPA, 2010) available from several source testing vendors (Apex Instruments, 2016; Environmental Supply Co, 2016) are acceptable, as are ambient PM10 inlets available from several ambient sampler manufacturers (Mesa Labs, 2016; Tisch, 2016; URG, 2016). Kenny and Gussman (2000) and Kenny et al. (2000) provide design criteria that allow for manufacture of PM10 inlets that accommodate a range of flow rates.

6. **"Blanks":** Four types of blanks should be done. (1) a lab blank, which is removed from each filter batch, stored in a protective environment, and weighed during each weighing session; (2) a "loaded blank", which is placed into the filter holder, then removed, stored, and handled like a sample filter; and (3) a dynamic tunnel blank, which is run as a sample but with a particle filter over the inlet of the dilution tunnel (not done during a test run). A "room blank" PM sample should also be collected during every test run (as recommended by ASTM 2515-11).

7. **Balance Resolution:** a balance with 0.01 mg resolution or better should be used to provide sufficient net mass resolution for lightly loaded filters and to better characterize mass loss during equilibration. With .01 mg resolution, a front filter should have a minimum loading of 0.20 mg for a valid test.

8. **Filter Sample Flow Measurement:** recommend use of an automated volumetric flow controller to eliminate manual flow "tweaking" as the filter loads. Target flow rates should match what is needed by the PM10 inlet to achieve an approximately 10 micron cut-point, but can vary as needed to maintain proportionality within limits (#13 below).

9. **Probe Catch:** recommend reporting sampling system "catch" as a separate number instead of combining it into a single mass value, to keep track of it. Catch is any mass from the sample probe system other than the PM on the front filter. The workgroup also recommends rinsing as a better approach to determine probe catch rather than weighing the probe to avoid catch mass precision issues.

10. **Filter Weighing Static Control:** recommend using an active ionizing air blower or ^{210}Po alpha sources to neutralize charge associated with the filter. Residual charge is indicated by erratic or unstable balance readings. Ion blowers are generally thought to be more effective in removing static charge. Two examples of suitable ionizing air blowers are:

http://desco.descoindustries.com/DescoCatalog/Ionization/Bench-Top-Ionizers/High-Output/60505/ and

https://technology-ionization.simco-ion.com/Products/IonizingBlowers/BenchtopIonizingBlowers/FastDischargeSingle-fanBenchtopIonizingBlower.aspx

11. **Weigh Room Environmental Conditions:** recommend a temperature range of 68º to 78º F and an RH cap of 45% (i.e., RH ≤ 45%).

12. **Sample Flow Corrections for Water Vapor:** recommend that corrections should be done using the average of actual tunnel dew point measurements during a sample run, rather than an assumed dew point value. This is a priority of 2.

13. **Limits on Proportionality** (the ratio between sample filter flow and tunnel flow over the duration of a test run): recommend the specification for maintaining the ratio between sample filter flow and tunnel flow be tightened to ± 5% (from 10%).

Inlet Cyclone References.

Apex Instruments, (2016). PM10 cyclone kit, http://www.apexinst.com/product/pm10-cyclone-kit

Environmental Supply Co, (2016). PM10-SS PM10 stainless steel cyclone set, http://www.environsupply.com/product/pm10-ss-pm10-stainless-steel-nozzle-set/

Kenny, L.C., Gussman, R.A., (2000). A direct approach to the design of cyclones for aerosol-monitoring applications. J. Aerosol Sci. 31, 1407-1420.

Kenny, L.C., Gussman, R.A., Meyer, M.B., (2000). Development of a sharp-cut cyclone for ambient aerosol monitoring applications. Aerosol Science and Technology 32, 338-358.

Mesa Labs, (2016). Cyclone fractionators, http://bgi.mesalabs.com/cyclone-fractionators/

Tisch, (2016). https://tisch-env.com/

U.S.EPA, (2010). Method 201A - Determination of PM10 and PM2.5 Emissions From Stationary Sources (Constant Sampling Rate Procedure): 55 FR 14246 04/17/90 (Appendix M of 40 CFR 51), U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards, Technical Support Division, Research Triangle Park, NC. http://www.epa.gov/ttn/emc/promgate/m-201a.pdf

URG, (2016). http://urgcorp.com/index.php/products/inlets/stainless-steel-cyclones