PM Test Method Workgroup Summary of Recommendations
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 used. (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/
- 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%).

Summary of Recommendations from the PM Measurement Method Workgroup

NESCAUM

George Allen, NESCAUM

July 19, 2017

PM Measurement Method Workgroup Charge:

To address issues specifically related to PM measurement methods included in 5G (and related methods)

Section 6 of: https://www.epa.gov/burnwise/process-developing-improved-cordwood-test-methods-wood-heaters

Workgroup Product:

List of recommendations that could improve PM measurement reproducibility (2020 NSPS Step 2 tighter limits)

5G method clean up (2017?)

Workgroup Members:

Selected by the Test Method Steering Committee

Academic Researchers with source testing expertise

Not stakeholders (S/L air agencies, EPA, commercial)

Workgroup Members:

Allen Robinson, Carnegie Mellon University

Phil Hopke, Clarkson University

Jamie Schauer, University of Wisconsin-Madison

Jay Turner, Washington University at St. Louis

John Watson, Desert Research Institute (DRI)

Workgroup Facilitator:
George Allen, NESCAUM

Workgroup Process:

1. Identified PM measurement issues that might contribute to test method reproducibility (within and across labs)

    ==> Goal was to make a ***reproducible*** PM measurement (not to make the "right" PM measurement)

2. Held four conference calls between May and September to discuss potential recommendations.

3. Reached consensus on specific recommendations
    - with ranking (1 to 3) of priority – high (1) unless noted
    - March 30, 2017 memo to test labs

⇨ These are WG recommendations, not EPA recommendations

Summary of Recommendations:

1. Water/Humidity/Temperature control in dilution tunnel and the sample train/filter:

Liquid water should not be present anywhere in the sampling system for a valid sample.

Dilution tunnel T and RH (and calculated dew point T) should be measured and logged near the sample probe

1% RH, 0.5 deg. C accuracy

Filter T should be measured and logged (0.5 deg. C accuracy)

(better than thermocouples can do)

1. (Continued) Water/Humidity/Temperature control

Limits:

Filter T between 80 and 90 deg. F (26.7 to 32.2 C)

Tunnel T not to exceed 100 deg. F (37.8 C) - priority of 2

Tunnel RH not to exceed 90%*

Tunnel dew point T at least 2 deg. C less than filter T*

* If exceeded, the test report should explicitly note the results of the sampling train liquid water inspection.

2. Filter Media:

Pallflex® Emfab™ (TX40) is recommended

Teflon coated glass fiber media

Similar filtration efficiency as glass fiber filters

Minimize interferences
- adsorbed organic and acidic gases, water vapor

3. Filter equilibration / conditioning post-sample collection:

General recommendation: use conditions and times similar to those used for the ambient PM2.5.

Do not use desiccation; equilibrate at RH between 30 and 40% for at least 24 hours.

- saturated salt solution of magnesium chloride (33% RH)

When possible, front filter mass measurements should be made soon after end of sampling ("0-day") and the next day

- to document filter mass loss over time – priority of 2

(Final filter weights would still be made as described in EPA method 5G)

4. Size-cut Cyclone:

PM10 (10 μm) size selective inlet upstream of the filter
- exclude large ash particles
- improve test consistency and precision

5. Blanks:

(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;

(3) a dynamic tunnel blank, which is run as a sample but with a particle filter over the inlet of the dilution tunnel

A room blank PM sample should also be collected during every test run (as required by ASTM 2515-11).

6. Balance Resolution:

0.01 mg resolution or better (semi-micro)
- better precision for very clean burns

The front filter should have a minimum loading of 0.20 mg for a valid test (needed for precision).

7. Filter Sample Flow Measurement:

Recommend automated volumetric flow controller
- eliminate manual flow adjustments as the filter loads

Flow rates should match what is needed by the PM10 inlet to achieve a 10 ± 0.5 micron cut-point.

8. Probe Catch:

Report 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)

9. Filter Weighing Static Control:

Use an active ionizing air blower (or ^{210}Polonium 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.

^{210}Polonium alpha sources must be replaced annually or more often.

10. Weigh Room Environmental Conditions:

Temperature range of 68 to 78 deg. F

RH no higher than 45%

11. Sample Flow Corrections for Water Vapor:

Corrections should be done using the average of actual tunnel dew point measurements during a sample run, rather than an assumed dew point value.

Priority of 2

12. Proportionality:

Control relationship between sample and tunnel flows to not over or under-sample different parts of the burn

(Method 5G: 8.10, 8.11, 12.7, 16.2.3)

Current 5G limits are +/- 10%

WG recommendation: +/- 5%

Note:
Constant sample flow (# 7) and proportionality:

Implies constant tunnel flow

Better measurement and control of tunnel flow?

(See #13, tunnel flow data logging)

13. 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

*Filter temperature

*Tunnel temperature and RH (and dew point temperature)

Filter face velocity (flow rate divided by filter's actual particle loading area)

13. Additional operational parameters to be reported (cont.)

Front filter net mass loading (mg)

Average tunnel PM concentration (mg/m^3)

***Tunnel flow rate (See #12)**

Tunnel residence time (should be in the range of 1 to 3 seconds)

Estimate of stack flow

Estimated tunnel dilution ratio range

Related Issues and Future Work.

Hot sampling (European) vs. warm (US)

Filter mass lost during equilibration:
- water and/or SVOC?
- Seek PM Workgroup input on how to assess?

Continuous tunnel PM measurements (Teom?)