Appendix E

Laboratory Methods and Modifications

NIOSH Draft Method 9103 – Modified: Analysis of MCE Filter

1. Transferred each filter sample to pre-cleaned individual 250-mL HDPE bottles.
2. Added 10 mL of concentrated nitric acid to each sample and gently swirled until the filter was completely saturated.
3. Placed caps loosely on the bottles and then set samples in a water bath maintained at 90 to 92 °C. After 2 minutes in the water bath, removed samples and cooled bottles to room temperature.
4. Added 50 mL of ASTM Type II water to each sample and gently swirled to mix thoroughly.
5. Added 25 mL of 5% potassium permanganate to each sample and gently swirled to mix thoroughly.
6. Added 8 mL of 5% potassium persulfate to each sample and gently swirled to mix thoroughly.
7. Placed caps loosely on the bottles and then set samples in a water bath maintained at 90 to 92 °C. After 30 minutes in the water bath, removed samples and cooled bottles to room temperature.
8. Immediately prior to analysis, added 7 mL of 20% hydroxylamine hydrochloride to each sample, replaced and tightened caps, and shook the bottles to mix samples thoroughly. Allowed bottles to cool to room temperature and proceeded to analysis.

- All standards were prepared the same manner as the sample except no filter media was present.
- One set of QC samples (QB, LCS, and LCSD) were prepared using MOE filter at the rate of 1 set per 20 field samples. (LCS & LCSD samples were prepared using 0.5 mL of 1.0 μg/mL Hg standard, yielding a spike target at 0.5 μg/sample.)

NIOSH Method 6009 – Modified: Analysis of Carulite (Hydrar) Tube

1. Carefully broke the edge of the sampling tube adjacent to sorbent material, and carefully transferred only the sorbent material of each sample to pre-cleaned individual 50-mL volumetric flasks.
2. Added 2.5 mL of concentrated nitric acid to each sample and gently swirled until the sample was completely saturated.
3. Added 2.5 mL of concentrated hydrochloric acid to each sample and gently swirled until the sample became dark. Placed the sample in a hood at least for 1 hour and swirled occasionally.
4. Diluted each sample to 50 mL volume with ASTM Type II water and shook the flasks to mix thoroughly.
5. Allowed samples to settle and proceeded to analysis.

- All standards were prepared the same manner as the sample except no sorbent media was present and no 1 hour waiting time was needed.
- One set of QC samples (QB, LCS, and LCSD) was prepared using SKC Carulite (Hydrar) tubes at the rate of one set per 20 field samples. (LCS and LCSD samples were spiked using 0.5 mL of 1.0 μg/mL Hg standard, yielding spike targets at 0.5 μg/sample.)

NIOSH Draft Method 9103: Analysis of Wash'n Dri Wipe

1. Transferred each wipe sample to pre-cleaned individual 250-mL HDPE bottles.
2. Added 5 mL of concentrated nitric acid to each sample and gently swirled until the wipe was completely saturated.
3. Added 5 mL of concentrated sulfuric acid to each sample and gently swirled until the wipe was dissolved. Placed samples in a hood until all acid fumes were evolved and no further reaction was observed.
4. Added 50 mL of ASTM Type II water to each sample and gently swirled to mix thoroughly.
5. Added 10 mL of l0% potassium permanganate to each sample and gently swirled until purple color disappeared. Added another 10 mL of 10% potassium permanganate to each sample, gently swirling until the reaction subsided. Added an additional 30 mL of 10% potassium permanganate to each sample and gently swirled to mix thoroughly.
6. Added 8 mL of 5% potassium persulfate to each sample and gently swirled to mix thoroughly.
7. Placed caps loosely on the bottles and then set samples in a water bath maintained at 90 to 92 °C. After 30 minutes in the water bath, removed samples and cooled bottles to room temperature.
8. Immediately prior to analysis, added 7 mL of 20% hydroxylamine hydrochloride to each sample, replaced and tightened caps, and shook the bottles to mix samples thoroughly. Allowed bottles to cool to room temperature and proceeded to analysis.

- All standards were prepared the same manner as the sample except no wipe media was presented.
- One set of QC samples (QB = quality control blank = media blank, spiked LCS = laboratory control sample, and LCSD = duplicate spiked laboratory control sample) was prepared using Wash'n Dri wipes at the rate of one set per 20 field samples. (LCS and LCSD samples were spiked using 0.5 mL of 1 .0 μg/mL Hg standard, yielding a spike target at 0.5 μg/sample.)

EPA Method 7470 – Modified/Phillips Lab Procedure – Modified: Analysis of Unbroken, Spent Lamp

1. Each entire lamp was cooled with dry ice for 1 hour and one end of the lamp was carefully broken.

2. Inner contents of the lamp was washed out with 200 mL of concentrated nitric acid and mixed well.

3. 1 mL of the acid leached sample was transferred to pre-cleaned 250-mL HDPE bottles.

4. Added 99 mL of ASTM Type II water, 5 mL of concentrated sulfuric acid, 2.5 mL of nitric acid, 15 mL of 5% potassium permanganate, and 8 mL of potassium persulfate, then mixed well.

6. Placed caps loosely on the bottles and then set samples in a water bath maintained at 90 to 92 °C. After 2 hours in the water bath, removed samples and cooled bottles to room temperature.

7. Immediately prior to analysis, added 5 mL of 20% hydroxylamine hydrochloride to each sample, replaced and tightened caps, and shook the bottles to mix samples thoroughly. Allowed bottles to cool to room temperature and proceeded to analysis.

- All standards were prepared the same manner as the sample except no acid leaching was involved.

- One set of QC samples were prepared using ASTM Type II water at the rate of one set per 20 field samples. (LCS and LCSD samples were spiked using 0.5 mL of 1.0 µg/mL Hg standard, yielding spike targets at 5.0 µg/L.)

EPA Method 7470 – Modified/Phillips Lab Procedure – Modified: Analysis of Lamp Debris (including glass, metal endcaps, and fines)

1. The lamp debris samples were preserved in a cooler and each sample was weighed (total weight — bottle weight = sample weight).

2. Each sample was leached with 200 mL of concentrated nitric acid for 1.5 hours.

3. 2 mL of homogeneous representative aqueous sample was transferred into pre-cleaned 250-mL HDPE bottles.

4. Added 98 mL of ASTM Type I water, 5 mL of concentrated sulfuric acid, 2.5 mL of nitric acid, 15 mL of 5% potassium permanganate, and 8 mL of potassium persulfate, then mixed well.

5. Placed caps loosely on the bottles and then set samples in a water bath maintained at 90 to 92 °C. After 2 hours in the water bath, removed samples and cooled bottles to room temperature.

6. Immediately prior to analysis, added 5 mL of 20% hydroxylamine hydrochloride to each sample, replaced and tightened caps, and shook the bottles to mix samples thoroughly. Allowed bottles to cool to room temperature and proceeded to analysis.

• All standards were prepared the same manner as the sample except no acid leaching was involved.

• One set of QC samples (LCS, and LCSD) were prepared using EPA reference soil at the rate of one set per 20 field samples. LCS and LCSD samples were obtained by leaching 0.5 g of EPA reference soil (target concentration of 12.3 μg/g) in 20 mL of concentrated nitric acid. 2 mL of the leachate solution was used to prepare the QCs.

NIOSH Draft Method 9103 – Modified: Analysis of HEPA Filter

1. Each HEPA filter container was opened and a representative portion of the main filter membrane was cut by 5 cm x 5 cm (= 25 cm^2).
2. Transferred each filter sample to pre-cleaned individual 250-mL HDPE bottles.
3. Added 5 mL of ASTM Type II water to each sample and gently swirled until the filter was saturated.
4. Added 5 mL of aqua regia to each sample and gently swirled until the filter was saturated.
5. Added 50 mL of ASTM Type II water to each sample and gently swirled to mix thoroughly.
6. Added 30 mL of 5% potassium permanganate to each sample and gently swirled to mix thoroughly.
7. Added 8 mL of 5% potassium persulfate to each sample and gently swirled to mix thoroughly.
8. Placed caps loosely on the bottles and then set samples in a water bath maintained at 90 to 92 °C. After 30 minutes in the water bath, removed samples and cooled bottles to room temperature.
9. Immediately prior to analysis, added 7 mL of 20% hydroxylamine hydrochloride to each sample, replaced and tightened caps, and shook the bottles to mix samples thoroughly. Allowed bottles to cool to room temperature and proceeded to analysis.

- All standards were prepared the same manner as the sample except no filter media was present.
- One set of QC samples (QB, LCS, and LCSD) were prepared using Whatman filters at the rate of 1 set per 20 field samples. (LCS and LCSD samples were spiked using 0.5 mL of 1 .0 μg/mL Hg standard, yielding a target at 0.5 μg/sample.)

EPA Method 7470 – Modified: Analysis of Carbon Pellets, Fines from Lamp Debris Samples, and Pre-filter Samples

1. Weighed 0.5 g of each representative sample and transferred the sample to pre-cleaned individual 250-mL HDPE bottles.
2. Added 5 mL of ASTM Type II water to each sample and gently swirled until the sample was wetted.
3. Added 5 mL of aqua regia to each sample and gently swirled until the sample was fully wetted.
4. Placed caps loosely on the bottles and then set samples in a water bath maintained at 90 to 92 °C. After 2 minutes in the water bath, removed samples and cooled bottles to room temperature.
5. Added 50 mL of ASTM Type II water to each sample and gently swirled to mix thoroughly.
6. Added 15 mL of 5% potassium permanganate to each sample and gently swirled to mix thoroughly.
7. Placed caps loosely on the bottles and then set samples in a water bath maintained at 90 to 92 °C. After 30 minutes in the water bath, removed samples and cooled bottles to room temperature.
8. Immediately prior to analysis, added 50 mL of ASTM Type II water and 5 mL of 20% hydroxylamine hydrochloride to each sample, replaced and tightened caps, and shook the bottles to mix samples thoroughly. Allowed bottles to cool to room temperature and proceeded to analysis.

- All standards were prepared the same manner as the sample except no bulk or soil media was present.
- One set of QC samples (LCS and LCSD) were prepared using EPA reference soil at the rate of 1 set per 20 field samples. (LCS and LCSD samples were prepared using 0.5 g of EPA reference soil, which has a targeted mercury concentration at 12.3 μg/g.) Also, one matrix spike sample (MS) and one matrix spike duplicate sample (MSD) was prepared at the rate of one per 20 field samples by spiking 0.1 mL of 1.0 μg/mL Hg onto the field samples, yielding spike targets at 1 .0 μg/L.