



APPENDIX AVAILABLE ON REQUEST

Research Report 153

Improved Source Apportionment and Speciation of Low-Volume Particulate Matter Samples

Schauer et al.

Appendix C. Microwave Digestion of Aerosol Particulate Matter

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This document was reviewed by the HEI Health Review Committee but did not undergo the HEI scientific editing and production process.

ESS INO METHOD CLNRM-003

Revision 1

Effective Date:

Replaces:

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ESS INO METHOD (CLNRM-003)
Microwave Digestion of Aerosol Particulate Matter
Low-Volume Reference Method

University of Wisconsin-Madison
State Laboratory of Hygiene
Trace Element Laboratory

10. Procedure

Assembly of the Digestion Apparatus.

The inner carousel holds 3 vials. There is a notch on the inner carousel. Figure 1 is a top view of the carousel and shows the orientation of the 3 vials with respect to the notch. The three vials are designated A, B, and C. Each cap with a notch is placed on the inner carousel. There are 12 inner carousels labeled 0, 2, 3, . . . , 12.

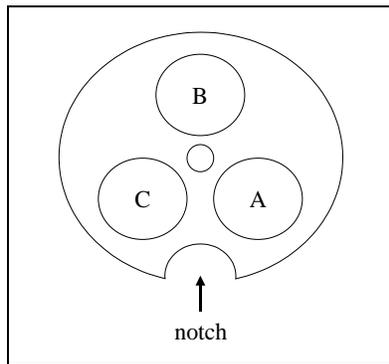


Figure 1. Top view of inner carousel.

- 10.1. Load 3 vials into the 3 slots on each inner carousel.
- 10.2. Place a carousel cap on each inner carousel. For carousel 0 the notch on its cap must be aligned with the notch of the carousel. (Carousel 0 receives the temperature probe.) The orientations of the notches on the other carousels do not matter. An assembled inner carousel is shown in Figure 2.

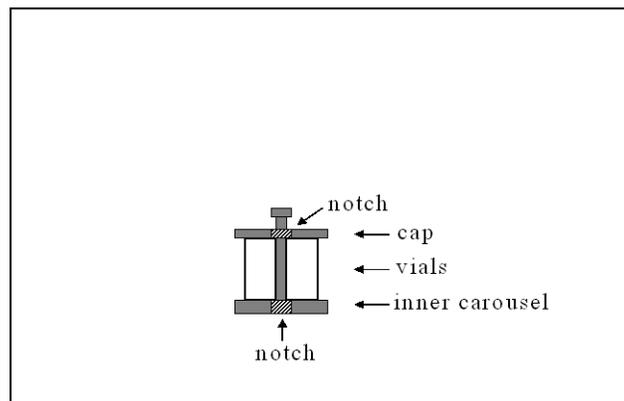


Figure 2. Side view of assembled inner carousel.

- 10.3. Place each inner carousel into an insert, and place the insert into a sleeve, as depicted in Figure 3. The insert has a lip on its rim so that it does not slide through the sleeve.

- 10.4. Place a plug assembly on each sleeve.

Note: Each sleeve is fitted with a cap assembly consisting of a ring, a plug, a spring seat, and a spring as illustrated in Figure 4. The ring and plug for carousel 0 are an integral unit and have a receiver for the temperature probe. Figure 5 depicts the plug assembly on a sleeve.

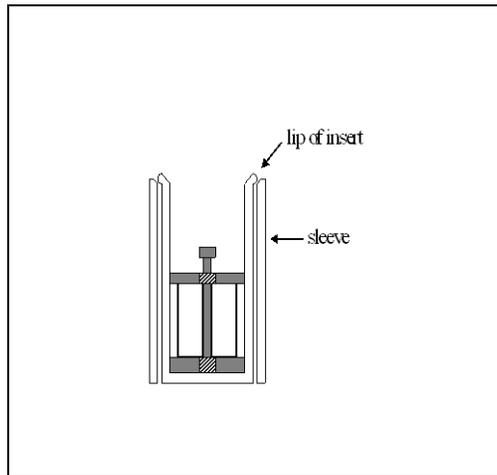


Figure 3. Assembled inner carousel, insert, and sleeve.

- 10.5. Place the sleeve and plug assembly into its sleeve housing.

Note: The plug assembly and sleeve are placed into one of the pie-shaped sleeve housings of the outer carousel. The sleeve housings are numbered 0, 2, 3, 4, . . . , 12. It is important that the plug assembly containing the receiver for the temperature probe be placed in sleeve housing 0. At the bottom of each sleeve housing is a circular rim. It is important that the bottom of the sleeve be seated within the rim; otherwise, when force is applied to the top of the spring, the bottom of the sleeve may slip.

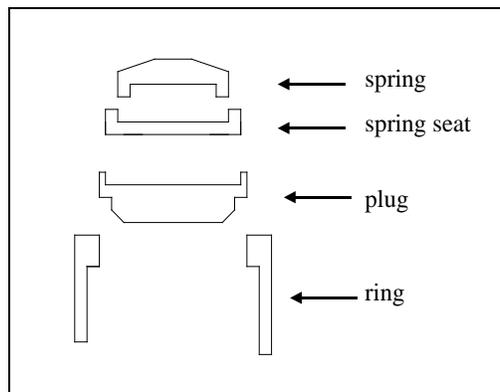


Figure 4. Exploded view of cap assembly.

10.6. Once a plug assembly and sleeve are in the sleeve housing, tighten the screw at the top of the housing until it is finger tight.

10.7. Place the sleeve housing into the sleeve-housing jig.

Note: The jig is constructed so that it holds the bottom of the sleeve housing firmly.

10.8. Once in the jig, use the torque wrench is used to tighten the screw of the sleeve housing until two clicks are heard. The remaining 11 sleeve housings are then assembled in the same way.

10.9. Place the cap on the outer carousel. At this point the outer carousel is ready to be loaded into the microwave.

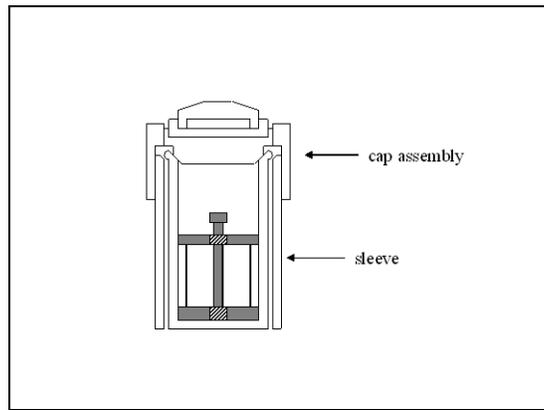


Figure 5. Cross section of plug assembly and sleeve.

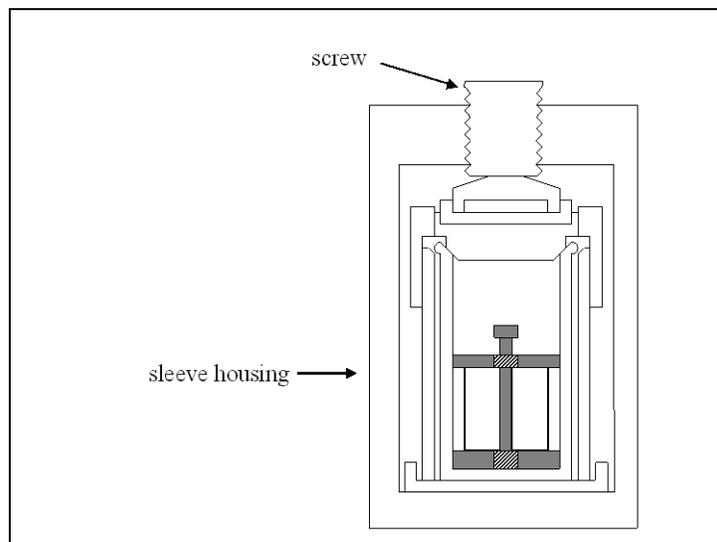


Figure 5. Sleeve assembly in pie-shaped-sleeve housing.

Cleaning of the Digestion Apparatus.

- 10.10. Rinse the vials, inner carousels, inner carousel caps, and inserts with MilliQ water from either the carboy or the gun.
- 10.11. Place 10 mL of trace metal grade nitric acid into each insert, and place 2 mL of trace metal grade nitric acid into each vial.
- 10.12. Assemble the entire digestion apparatus as discussed in Steps 10.1 through 10.9.
- 10.13. Open the door to the microwave cavity. (Pull down on the handle of the door.)
- 10.14. Place the outer carousel on the door.
- 10.15. Make a J shape with the cord to the temperature probe as illustrated in Figure 6. (When the cord to the temperature probe is in the J configuration, the cord will move smoothly with housing 0 as the outer carousel rotates in the microwave cavity.) Pull the temperature probe back far enough to place the probe into the receiver of housing 0.

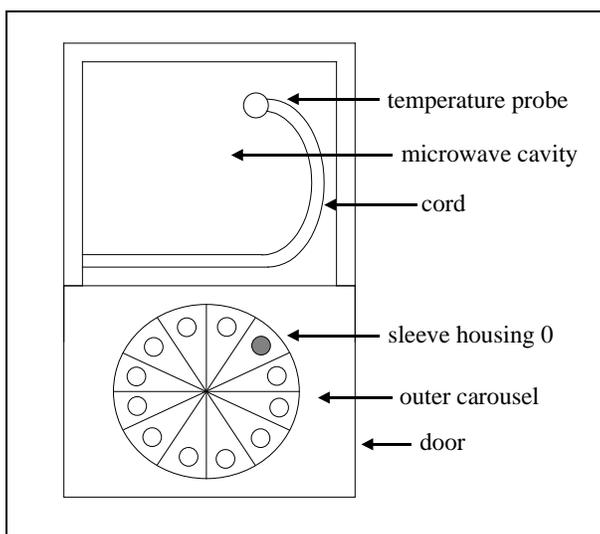


Figure 6. Placing outer carousel into microwave.

- 10.16. Slide the outer carousel into the microwave cavity. Place the outer carousel in the microwave cavity so that the depression on the bottom of the carousel is seated on the cone-shaped protrusion on the bottom of the microwave cavity. The outer carousel should now be centered in the microwave cavity.
- 10.17. Rotate the outer carousel once to make sure that the cord of the temperature probe moves smoothly with the rotating carousel.
- 10.18. The power to the microwave should have been turned on prior to loading the carousel. If not, the microwave circuits must be given an adequate period of time to warm up. If the

microwave had been shut down for 12 hours, a 24-hour warm-up period is required; if the microwave had been shut down for 24 hours or more, a 72-hour warm-up period is required.

- 10.19. Turn the power to the microwave controller on. If you are facing the controller, the power switch is located on the back of the controller on the top right side.
- 10.20. Place the “easy Wave Administrator” card into the slot on the front of the controller so that the logo of the printed circuit board is facing up and towards the controller.
- 10.21. With the mouse double click on the “easy wave” icon on the desk top.
- 10.22. Once the desktop appears, click on the “OK” button.
- 10.23. Click on the “Open” button on the right side of the controller screen. An “Open” window will appear that contains a number of files. Click on the file named “dan013101.mwp;” then click on the “Open” button of the “Open” window. This action will cause the digestion procedure to begin. To watch the temperature, as measured by the temperature probe, as the digestion proceeds, click on the “graphic” button.
- 10.24. Once the digestion is complete, allow the nitric acid in the vials and inserts to cool to room temperature. Loosening the screws from the sleeve housings prematurely may cause acid solution to spatter.
- 10.25. Empty the nitric acid into an appropriately labeled container.
- 10.26. Repeat the Steps 10.10 through 10.25 one more time.
- 10.27. Rinse the vials, inner carousels, inner carousel caps, and inserts with MilliQ water at least three times apiece. The digestion apparatus is now ready for the digestion of actual samples.

Cleaning of 60-mL LDPE (Low Density Polyethylene) Sample Bottles.

- 10.28. Fill each 60-mL sample bottle with 20% (v/v) nitric acid.
- 10.29. Allow the bottles to sit for two days.
- 10.30. Rinse the bottle with milliQ water.
- 10.31. Fill each 60-mL sample bottle with 2.0% (v/v) Trace Metal Grade nitric acid.
- 10.32. Allow the bottles to sit for two days.
- 10.33. Rinse the bottles with milliQ water.
- 10.34. Allow the bottles to dry.
- 10.35. Weigh each dry sample bottle and cap, and record the weight of the sample bottle on the outside of the bottle with a felt pen.
- 10.36. Place the bottles in resealable plastic bags labeled 0A, 0B, 0C, 2A, 2B, 2C, 3A, 3B, 3C, . . . , 12A, 12B, 12C. Two “extra” 60-mL bottles must always be prepared. These bottles are filled with the undigested acid matrix and used as acid matrix/bottle blanks.

Digestion and Preparation of Samples.

Preparation of the Standard Reference Materials and of the Blank Spikes.

The last eight sample positions (i.e., 10B, 10C, 11A, 11B, 11C, 12A, 12B, 12C) are for the standard reference materials (SRMs) and the blank spikes. The blank spikes are always in positions 12B and 12C. All of the SRMs are obtained from the National Institute of Standards and Technology (NIST). The SRMs typically include 2 samples of San Joaquin soil (NIST 2709), 2 samples of urban dust (NIST 1649a), and two samples of used auto catalyst (NIST 2556). NIST SRM 2702 (Inorganics in Marine Sediment) is also incorporated into selected batches.

- 10.37. Take the assembled inner carousels 10, 11, and 12 to room 206C.
- 10.38. Weigh out from 2 to 4 micrograms of each SRM into the appropriate vial. First placing the appropriate inner carousel with vials (but no inner carousel cap) on the balance and tare the balance. Using a clean 2.5-mL disposable plastic pipet tip, measure between 2 and 4 micrograms of the SRM into the appropriate vial. If somewhat more than 4 micrograms of the SRM is used, this is not a problem. Use a different pipet tip for each SRM.

Presently, the Mettler Toledo A6245 microbalance in room 206C is used to weigh out the SRMs. It is important that the balance display 5 digits past the decimal point. If not, press the "1/10 d" button of the balance until there are 5 decimal places. Use the using the "→Q/T←" button to tare the balance.

- 10.39. When done measuring out the SRMs, use a brush found in one of the drawers near the balance to clean the balance and the countertop around the balance
- 10.40. Preparation of the Blank Spikes (refer to separate SOP).

Preparation of the Air filters for Digestion.

The air sample filters are typically Teflon membranes, either pure Teflon laminates (e.g. Zefluor filters) or Teflon membranes mounted to a PMP plastic ring as shown in Figure 7 (Teflo filters). The air filters are usually stored in labeled Petri dishes.

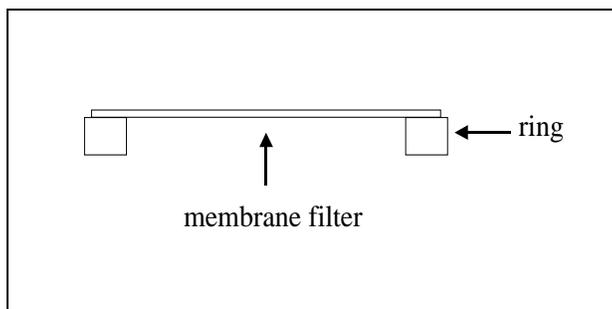


Figure 7. Air filter on plastic ring.

- 10.41. Remove a vial from an inner carousel.
- 10.42. Place air filter into the bottom of the vial. If the filter is of the Teflo variety, the PMP ring must be removed prior to digestion.

Note: To remove an air filter from its ring, it is best to use a Teflon tweezers to tease the membrane filter away from the plastic ring. Once enough of the edge of the filter has been removed from the ring, the tweezers can be used to pull the Teflon membrane filter away from the ring. Remove the proper vial from the inner carousel, and place the filter in the vial using the tweezers. The vial should be tapped on the counter until the filter settles to the bottom of the vial.

- 10.43. Put the vial back in the inner carousel.

Note: The tip of the tweezers should be rinsed with 20% trace metal grade nitric acid and dried with a clean room wiper between the removal of filters.

- 10.44. Add 0.1 mL of concentrated (48N) Optima grade hydrofluoric acid, 1.0 mL of concentrated (16N) Optima grade nitric acid, and 0.25 mL of concentrated (12N) trace metal grade hydrochloric acid to each vial (Total acid volume = 1.35 mL). After the addition of the hydrochloric acid, the solution in the vials should turn orange.
- 10.45. Assemble the digestion apparatus as discussed in steps 10.1 through 10.9.
- 10.46. Place the outer carousel in the microwave cavity as discussed in Steps 10.13 through 10.17.
- 10.47. Turn the power to the microwave controller on. If you are facing the controller, the power switch is located on the back of the controller on the top right side.
- 10.48. Place the “easyWave Administrator” card in the slot on the front of the controller so that the logo of the printed circuit board is facing up and towards the controller. With the mouse double click on the “easy wave” icon on the desktop. Click on the “OK” button.
- 10.49. Click on the “Open” button on the right side of the screen. An “Open” window will appear that contains a number of files. Click on the file named “EPA coal 111400.mwp;” then click on the “Open” button of the “Open” window. This action will cause the digestion procedure to begin. To watch the temperature, as measured by the temperature probe, as the digestion proceeds, click on the “graphic” button.
- 10.50. Fill a plastic tub with milliQ water. This will be used to soak the vials, inner carousels, inner carousel caps, and inserts after the digested samples have been removed.
- 10.51. Remove the insert containing the inner carousel from sleeve housing 0.

- 10.52. Remove inner carousel 0 from its insert.
- 10.53. Position the carousel over its insert so that any solution dripping from the carousel will fall into the insert.
- 10.54. Lift each of the 3 vials of the inner carousel out of its respective slot in the inner carousel, and allow the acid solution in each slot to drain into the insert.

Note: There is a hole in the center each slot that extends through the bottom of the carousel. When the vial is lifted from the slot, the digestion solution in the slot will drain into the insert.

- 10.55. Pour the acid solution from the insert into a properly labeled acid wasted container.
- 10.56. Put the insert into the plastic tub filled with milliQ water.
- 10.57. Remove one of the previously cleaned 60-mL polyethylene bottles from its plastic bag.
- 10.58. Remove a vial from its inner carousel.
- 10.59. Place the tip of the Teflon tweezers in the mouth of the vial to prevent the filter from falling into the bottle, when contents of the vial are transferred to the bottle.
- 10.60. Pour the contents of the vial into the 60-mL bottle.
- 10.61. Rinse the vial by squirting enough milliQ water into the vial to fill the vial ~half full, and, as before, pour the contents of the vial into the 60-mL bottle. Repeat this step twice.
- 10.62. Repeat Steps 10.57 through 10.61 for each of the vials.
- 10.63. Put all of the vials, inner carousels, carousel caps, and inserts into the plastic tub filled with milliQ water. These parts are ready to be cleaned using the procedure in Steps 10.10 through 10.27.
- 10.64. Add enough milliQ water to each 60-mL bottle so that the total weight of the contents of the bottle is 15.49 g.

Note: The weight of the bottle must be taken into account. Remember that the weight of the bottle had been recorded on the bottle in Step 10.35.