TITLE: Cleaning and Coating of Aluminum Honeycomb Denuders

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Prepared by: Katherine W. Barry
Laboratory Operations Manager

katherine.barry@amecfw.com
Digitally signed by katherine.barry@amecfw.com
DN: cn=katherine.barry@amecfw.com
Date: 2018.05.30 15:23:46 -04'00'

Reviewed by: Anne Glubis
Quality Assurance Manager

anne.glubis
Digitally signed by anne.glubis
Date: 2018.05.30 16:55:22 -04'00'

Annual Review			
Reviewed by:	Title:	Date:	Signature:

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1.0 Purpose

The purpose of this standard operating procedure (SOP) is to provide consistent guidance to Wood Environment & Infrastructure Solutions, Inc. (Wood) laboratory personnel for the cleaning and coating of aluminum honeycomb denuders used with the Met One Instruments, Inc. Speciation Air Sampling System (SASS) $PM_{2.5}$ samplers.

2.0 Scope

This SOP applies to all aluminum honeycomb denuders that are prepared for the Chemical Speciation Network (CSN) and CSN-like ambient air projects.

3.0 Summary of Method

Aluminum honeycomb denuders are cleaned, any previous coatings removed and then coated with magnesium oxide (MgO). Deviations from the analytical method described in this SOP are not permitted.

4.0 Materials

4.1 Apparatus

- Aluminum honeycomb denuders
- Glass tray, 1.5 L
- Glass beaker, tall, 125 mL
- Bottles, wide-mouth, 125 mL capacity or larger
- Nitrogen compressed gas cylinder
- Hand-held magnifying glass
- Tweezers, plastic
- Teflon-coated magnetic stir bar
- Teflon coated magnetic stirrer
- Kimwipes, large
- Resealable plastic bags
- Nitrile, powder-free gloves.
- Analytical balance capable of accurately weighing to the nearest 0.01 gram (g) (see GLO-3180-042 for balance check procedure).

4.2 Reagents

1. Reagent Water: Deionized (DI) water of resistivity of 15 mega Ohms (MΩ) or greater, derived from mixed bed ion exchangers, activated carbon filters and polishing exchangers. Water should contain particles no larger than 0.20 micrometers.
2. 2N HCl: prepared as follows, 83 mL of concentrated (12 N) ACS grade hydrochloric acid (HCl) is carefully added to approximately 300 mL of deionized water (DI) and after cool, brought to a final volume of 500 mL with DI. CAS# 7647-01-0
3. Reagent grade methanol. CAS# 67-56-1.
4. Magnesium Oxide, USP. CAS# 1309-48-4.
5. Ethanol, 200 proof, absolute. CAS# 64-17-5.

5.0 Safety

The analyst must be aware of the hazards associated with the chemicals used in this method. Reducing the possibility of accidental absorption or ingestion minimizes the hazards. Eating and drinking are not permitted in areas where chemicals are used or stored. Laboratory coats, gloves, and safety glasses must be worn at all times when handling these chemicals. Work in a laboratory hood when transferring HCl, alcohol, alcohol/MgO slurries and when removing MgO powder from the denuder. If the analyst is not familiar with the hazards associated with the chemicals being used, the Safety Data Sheets (SDS) must be consulted. The SDS by chemical and brand can be found in the Wood laboratory or at the SDS/MSDS search web site at https://www.msdsonline.com/msds-search/ or the Vermont Safety Information Resources, Inc. (SIRI) web site at http://www.hazard.com/msds/index.php using the CAS number.

6.0 Procedure

6.1 Cleaning Aluminum Honeycomb Denuder

1. Remove the denuder from its Met One aluminum sampler collar by gently pressing uniformly on one end to push it away from the collar. If the denuder is not clean, use a pair of tweezers to hold each denuder securely and dip it repeatedly into a methanol or ethanol bath to remove debris and oils. If the denuder is damaged, set it aside and return to a CSN technician so that it can be marked and taken out of service.
2. If the denuder was previously coated with magnesium oxide, the coating will be removed as follows:
   a. Drain any excess water from the denuder. Place enough 2N HCl in a glass tray to cover several denuders to at least half of their height.
   b. Use tweezers to carefully dip the denuders into the acid bath. To avoid damaging the aluminum denuders, exposure to the acid solution must not

exceed 2 minutes. It is recommended that a timer be used to account for the exposure time.

c. Using tweezers, move the denuder up and down and rotate in the solution until gas evolution ceases and the surface of the denuder is shiny. Turn the denuder over and repeat until all interior surfaces are cleaned and gas evolution again ceases.

d. Rinse the denuder interior and exterior walls with plenty of tap water and follow with multiple rinsing in a stream of deionized water, taking care to pass water through each honeycomb opening and the crevices on the outer surface.

e. Pass a stream of nitrogen gas through the denuder to remove most of the water.

f. Let each denuder air dry overnight before coating it.

g. Properly dispose of the acid bath solution.

6.2 Preparation of Magnesium Oxide Slurry

1. Working in a hood, add 120 mL of ethyl alcohol to a labeled wide-mouth glass bottle containing 37.5 g of MgO. Close the bottle tightly and swirl the contents until all of the powder is wet with alcohol. Insert a clean Teflon-coated magnetic stirring bar in the solution. Replace the cap and place bottle on a magnetic stirrer. Stir for 3-4 hours or overnight using the “low” setting on the magnetic stirrer. Shake the bottle from time to time to bring any MgO that may have settled out and accumulated on the walls of the bottle back into the slurry. If the slurry is too thick, a small amount of alcohol may be added to achieve the working milk-like consistency. The prepared slurry is enough to coat approximately 25 denuders.
2. During a coating session, the contents of the slurry must be continuously stirred to prevent the MgO from settling out.

6.3 Coating of Aluminum Honeycomb Denuders with Magnesium Oxide

1. Wearing gloves and appropriate eye protection, transfer 50-60 mL of the MgO slurry from the preparation bottle to a tall, 125 mL glass beaker. The beaker and the amount of slurry added must allow for the denuder to be completely covered and for unobstructed operation of the magnetic stir bar.
2. Insert tweezers into an outermost chamber of a honeycomb denuder and grasp firmly but gently. Submerge the denuder in the slurry and allow the device to remain covered for approximately 5 seconds. Lift the denuder out of the slurry, but continue to hold it over the mouth of the container.
3. Slowly dip the denuder in and out of the slurry five times. Ensure that the device is completely covered by the slurry each time. Submerge the device a sixth time and allow it to remain covered for 10 seconds.
4. Lift the denuder out of the slurry, but continue to hold it over the mouth of the container. Grasping the tweezers tightly, use a sharp downward thrust to remove as much of the slurry as possible from the chambers of the denuder. A significant amount of slurry may remain in the chambers. If so, the downward thrusting may

be repeated once more, but do not attempt to remove the slurry from the denuder's chambers by tapping against the side of the container. Such action could result in uneven distribution of the slurry in sections of the denuder or within individual chambers. Also, slurry splashed onto the sides of the container will dry quickly and could be released back into the solution, forming unwanted particles or lumps.

5. Care must be taken to limit the evaporation of alcohol from the slurry. Return the remaining MgO slurry to its container, seal tightly and resume stirring.
6. Grasp the denuder between the thumb and forefinger and look down into the device. The majority of the chambers should contain slurry and you should not be able to see through.
7. Rotate the denuder 180º from top-to-bottom-to-top five times to evenly coat the chambers. The slurry contained within the denuder chambers can be seen if the denuder is viewed from the bottom.
8. Crumple a large Kimwipe into a loose wad, creating channels that will promote draining the slurry from the denuder.
9. Place the denuder on the wadded Kimwipe and press it firmly into the paper. At the same time, bring the toweling up and around the sides of the device to absorb the excess slurry from its outside surfaces. Do not cover the top of the denuder with the Kimwipe.
    a. Lift the denuder and then firmly press it, bottom-side down, on a clean section of the toweling. Rotate the denuder bottom-side up and repeat the procedure, moving to a clean section of the toweling each time. If liquid can be seen in any of the chambers, it may be necessary to tamp the device lightly on the toweling to dislodge the excess slurry before it dries in place. Use a fresh Kimwipe for each denuder.
10. Tilt the denuder top-to-bottom, hold it up to a bright light, or look down into the chambers to verify that all of the chambers are clear of excess slurry.
11. Gently, but quickly wave the denuder up and down so that room air moves freely through the chambers. Do not sling or shake the denuder. As the alcohol evaporates, the exterior of the device should feel cool to the touch. Continue this process for approximately 15 to 30 seconds.
12. For continued evaporation of the alcohol, place the denuder in a hood on a wire rack, or some other structure that supports but does not block the bottom of the honeycomb denuder and allows unobstructed exposure to room air.
13. Prior to handling the next denuder, gloved hands should be rinsed under running water and dried completely.
14. Allow the coated denuder to dry for at least 1 hour. After drying, use a hand-held magnifying glass to view and ensure that none of the chambers are blocked by dried MgO coating. If necessary, use a thin wire to carefully scrape away or dislodge the plug. Apply a gentle stream of nitrogen to the opposite end of the previously blocked chamber to flush out MgO dust.

15. It is not critical for large amounts of dried slurry to be cleared from the openings on the extreme outer edge of the denuder. However, care should be taken that the amount of MgO coating present is not extensive or creates a dust or fine particle hazard for the other chambers. Too much MgO present on the outer edges of the denuder may prevent its correct placement in the mounting collar. Work in the hood to gently remove excess MgO powder with a stiff brush. Tap the denuder on a clean surface to remove adhering particles; complete the removal of the excess MgO dust by passing a stream of nitrogen or air over the surfaces.
16. Gently push the denuder into the aluminum collar and check that the O-rings are intact on either end of the collar. Place the dried, coated denuder in a plastic, zip-closure bag Label the bag with the date of preparation and return to a CSN technician.

7.0 References

U.S. Environmental Protection Agency (EPA). 2007. Guidance for the Preparation of Standard Operating Procedures, (SOPs) for Quality-Related Documents. EPA/600/B-07/001, April.

Research Triangle Institute, 2009. Standard Operating Procedures for Coating Aluminum Honeycomb Denuders with Magnesium Oxide. Rev. 4, February 17, 2009.

8.0 Attachments

This SOP does not contain attachments.