

Standard Operating Procedures for Coating Aluminum Honeycomb Denuders With Magnesium Oxide

Environmental and Industrial Sciences Division
RTI International*
Research Triangle Park, North Carolina

Prepared by: W. Cary Eaton

Date: 2-17-09

Reviewed by: Jan B. Hanayn

Date: 2/18/2009

Approved by: L. K. Gutknecht

Date: 2-19-09



* RTI International is a trade name of Research Triangle Institute.

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1.0 Purpose and Applicability

This document outlines procedures for cleaning and coating aluminum honeycomb denuders with magnesium oxide for the collection of gas-phase acidic species in the ambient air. This standard operating procedure (SOP) is applicable for coating aluminum honeycomb denuders used with the Met One Instruments, Inc. SASS line of PM_{2.5} Speciation samplers. Under most ambient sampling conditions, the denuder's performance is not expected to degrade significantly over three months of continuous field use.

2.0 Safety Precautions

- 2.1 Always wear latex or plastic gloves when handling solvents (ethyl alcohol or methyl alcohol) if there is a potential for spillage.
- 2.2 Always wear protective eye wear when conducting laboratory procedures specified in this SOP.
- 2.3 Read, understand, and follow the Material Safety Data Sheets (MSDSs) or Chemical Safety Cards for all chemicals involved in this procedure.
- 2.4 Always keep open chemical containers in fume hoods and wear adequate protective clothing according to the MSDSs for that chemical. Wear dust mask when transferring dry MgO powder during weighing operations, unless you work in a hood.
- 2.5 Always label secondary containers used in this procedure.
- 2.6 Work in a laboratory hood when transferring alcohol and alcohol/MgO slurries and when removing MgO powder from the denuder with laboratory tissue, cotton swabs, and/or a stream of air or nitrogen gas.

3.0 Equipment and Materials

- 3.1 Plastic or latex disposable gloves, sized to fit. RTI stockroom item.
- 3.2 Magnesium oxide, light powder, USP. 500 g. EM Science, product No. MX0064-1.
- 3.3 Ethanol (ethyl alcohol). 500 mL. RTI stockroom item. 200 proof, absolute.
- 3.4 Alternate slurry solvent. Methanol (methyl alcohol). J.T. Baker product 9076-03 or equivalent.
- 3.5 Hydrochloric acid solution. 2 normal (2N). Prepared by 1:3 dilution of 6M reagent grade concentrated acid. Work in a hood. Wear safety glasses and rubber gloves. Pour 2 parts of distilled water into a glass beaker. For example, pour 40 mL of water into a 100 mL beaker. Pour 20 mL of 6N hydrochloric acid into a glass graduated cylinder. Then slowly add the 20 mL of acid to the 40 mL of water to make 60 mL of 2N acid solution.

- 3.6 Wheaton bottles, 125 mL capacity with screw caps and plastic or Teflon cap liners (for preparation of smaller amounts). Wide-mouth bottles, 1L and 500 mL capacity, with screw caps and plastic or Teflon cap liners (for preparation of up to 400 mL of slurry). Larger glass bottles may be used when preparing greater volumes of slurry.
- 3.7 Funnel, glass, powder. 80-mm width, 60-degree slant. To transfer dry MgO powder and slurries of MgO.
- 3.8 Distilled water, laboratory grade. Available from in-house supply, RTI building No. 3 or 6.
- 3.9 Magnetic stirrer and Teflon-coated stir bar, 1-inch length.
- 3.10 Graduated cylinder, glass, 50 or 100 mL capacity. Graduated to nearest 1 mL. Various glassware (e.g., beakers, Erlenmeyer flasks)
- 3.11 Spatula, stainless steel. For transfer of magnesium oxide from the reagent bottle to the slurry bottle or beaker during weighing.
- 3.12 Top-loading electronic balance. 100 g or 400 g capacity. Readable to nearest 0.01 g.
- 3.13 Quality control test weights, appropriate for characterizing the operating range of the balance at 10%, 50%, and 75% of scale.
- 3.14 Source of clean, dry air or nitrogen. Flexible Tygon tubing for routing air or nitrogen to denuder tubes during drying stages. Air and nitrogen are available at hoods in Building No. 6 or 3.
- 3.15 Aluminum honeycomb denuder substrate, clean and dry. Denuder substrate material (i.e., the uncoated aluminum honeycomb denuder) is available from Met One Instruments, Inc.

4.0 Cleaning Aluminum Honeycomb Denuder

- 4.1 If the denuder is still in its Met One aluminum sampler collar, gently remove the denuder body by pressing uniformly on one end to push it from the collar using gentle pressure from your thumbs or the flat end of a wooden dowel of a diameter slightly less than the diameter of the denuder. Inspect each aluminum honeycomb denuder carefully to be sure it is not broken, bent, or compressed so the orifices are closed or a large gap exists. One technique for examining the denuder is to hold it up to a light and sight through the tubes while looking for signs of splitting or closure.
- 4.2 If the denuders are not clean (especially unused denuders received directly from the manufacturer), use a pair of tweezers to hold each denuder securely and dip it repeatedly into an acetone, methanol, or ethanol bath to remove debris and oils.
- 4.3 If the denuder is coated with magnesium oxide, 2N hydrochloric acid must be used to remove the coating. Proceed as follows, working in a hood and wearing safety glasses and protective apparel:
 - 4.3.1 Drain any excess water from the denuder. Place 25 mL of 2N acid in a glass beaker that will just accommodate the denuder when it is dipped into the solution.

To accommodate cleaning several denuders at once, a larger volume of acid may be placed in a glass Pyrex dish. The level of acid in the dish should only cover the denuder devices to a little more than half their height.

- 4.3.2 Thread a short length of polyethylene tubing through a honeycomb channel near the center of the denuder to serve as a means of inserting the denuder into the acid bath. Cautiously dip the denuder into the acid solution. For the larger volume of acid contained in the Pyrex dish, use stainless steel or plastic tweezers to place the denuder devices in the acid bath.

CAUTION: Gas will be evolved. Do not leave the denuder in the solution more than about 2 minutes. The object is to remove the spent MgO coating only and not to cause reaction of the aluminum denuder itself.

- 4.3.3 Move the denuder up and down in the solution until gas evolution ceases and the surface of the denuder is shiny. For the larger volume, use tweezers to continually rotate the denuders so that all surfaces are contacted by the acid. For the larger volume, turn the denuder over and repeat until all interior surfaces are cleaned and gas evolution is very slow. Remove the denuder from the acid bath using the polyethylene tubing or by use of the tweezers.
- 4.3.4 Rinse the denuder interior and exterior walls with plenty of tap water and follow this with multiple rinsing with a stream of distilled or deionized water from the laboratory source, taking care to pass water through each honeycomb opening and the crevices on the outer surface.
- 4.3.5 Finally, pass a stream of air or nitrogen through the denuder to remove most of the water. Let each denuder air dry overnight before coating it.

NOTE: If a denuder is needed sooner, continue passing a stream of laboratory air or nitrogen through the tubes until all surfaces are dry. Alternatively, ethanol or methanol can be added to the denuder to rinse out the water and then dry the denuder with a stream of air or nitrogen.

- 4.3.6 Properly dispose of the acid bath.

5.0 Preparation of Magnesium Oxide Slurry

- 5.1 Using the basic formulation, 12.5 g magnesium oxide per 40 mL ethyl alcohol, a powder funnel, and a top-loading balance, quantitatively transfer the appropriate amount of MgO into a labeled Wheaton bottle or wide-mouth glass bottle outfitted with a screw-type closure. Close the bottle tightly. Record information in laboratory notebook.

NOTE: The amount of slurry prepared will depend on the number of devices to be coated. One bottle of slurry prepared using the basic formulation is sufficient to apply a

single coating to upwards of a dozen Met One aluminum honeycomb denuders. Other types of bottles may be used to prepare larger amounts.

- 5.2 Working in the hood, use the graduated cylinder to add the appropriate amount of ethyl alcohol (e.g., 40 mL for each 12.5 g of MgO) to the Wheaton (or other) bottle containing the MgO powder. Close the bottle securely. Tilt the bottle and contents back and forth several times until all the powder is wet with alcohol.
- 5.3 Open the bottle and insert a Teflon-coated magnetic stirring bar. 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 consistency. Continue stirring the slurry container contents throughout the denuder coating session in order to prevent the MgO from settling out.

6.0 Coating of Aluminum Honeycomb Denuders with Magnesium Oxide

- 6.1 Wearing properly fitted laboratory gloves and appropriate eye protection, transfer enough MgO slurry from the preparation bottle to a tall, wide-mouth glass bottle or beaker. The height of the container and the amount of slurry added must allow for full submersion of the denuder into the slurry and for unobstructed operation of the magnetic stirrer bar.

NOTE: When coating less than 10 denuders, the slurry may be poured into a beaker and used with or without the magnetic stirrer. In either case, immediately after the denuder is removed from the slurry for the last time, the remaining liquid must be returned to the Wheaton (or other style) glass bottle, which should then be sealed and placed on the magnetic stirrer for continued stirring.

- 6.2 To apply a coating of slurry, insert tweezers into an outermost chamber of a honeycomb denuder and grasp firmly but gently.
- 6.3 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.

CAUTION: Do not allow the denuder to interfere with the operation of the stirrer.

- 6.4 Slowly dip the denuder in and out of the slurry five times. Ensure that the device is completely covered by the slurry each time it enters the slurry. Submerge the device a sixth time and allow it to remain covered for 10 seconds.
- 6.5 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 follow the cautions as noted below.

CAUTION: 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.

CAUTION: Care must be taken to limit the evaporation of alcohol from the slurry. Place a watch glass over the opening of the container immediately after the denuder is removed from the slurry.

- 6.6 Grasp the denuder between the thumb and forefinger and look down into the device. The majority of the chambers should contain slurry as evidenced by no light appearing (i.e., you cannot see through the chamber) at the bottom of the chamber.
- 6.7 To facilitate even distribution of the slurry within the chambers, rotate the device 180° from top-to-bottom-to-top five times. The slurry contained within the denuder chambers can be seen if the denuder is viewed from the bottom.
- 6.8 Crumple a large absorbent sheet of paper toweling into a loose wad, creating channels that will promote draining the slurry from the denuder. The toweling could be prepared prior to immersing the device at Step 6.4.

NOTE: Although either maybe used, toweling such as Kimwipes® appears to work better than ordinary 2-fold paper towels.

- 6.9 Place the denuder on the wadded toweling 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.

NOTE: Covering the top of the device with the toweling is not necessary or recommended.

- 6.10 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.

NOTE: Toweling used in Steps 6.10 and 6.11 must be discarded. Reuse with the next denuder is prohibited because particles sloughed from the dried slurry could block openings or adhere to chamber walls.

- 6.11 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.
- 6.12 To begin the drying process, 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.
- 6.13 For continued evaporation of the alcohol, place the denuder on an open-weave plastic mat, test tube holder, 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. Place the denuder in the laboratory fume hood.
- 6.14 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 (a paperclip wire works well) 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.

CAUTION: Particles from the dried MgO slurry are quite easily generated. Care must be taken when handling coated denuders. Prior to coating the next device, gloved hands should be rinsed under running water and dried completely.

- 6.15 Place the dried, coated denuder in a plastic, zip-closure bag . Label the bag with the date of preparation and store for later use. The coated denuder may be re-installed in its aluminum collar at this point. Gently push the denuder into the aluminum collar, check to see that the O-rings are intact on either end of the collar, and store the denuder units in the labeled zip-closure bag until they are ready to be placed in the Met One sampling module.

NOTE: 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 on the outer edges of the denuder may prevent its placement in the mounting collar. If this is so, 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.