

**PROCEDURES FOR COATING
R&P SPECIATION SAMPLER CHEMCOMB™
DENUDERS WITH SODIUM CARBONATE**

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PROCEDURES FOR COATING
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1.0 Purpose and Applicability

This document outlines procedures for cleaning and coating the R&P Chemcomb™ denuder with sodium carbonate for the removal of gas phase acidic species from the ambient air during collection of PM_{2.5} particulate matter. Refer to the Rupprecht & Patashnick Co., Inc. Operating Manual for the Chemcomb Model 3500 Speciation Sampling Cartridge, January 2000, Revision A, for details and photographs of the equipment.

2.0 Safety Precautions

- 2.1 Always wear latex or plastic gloves when handling solvents (methyl alcohol) if there is a potential for spillage. Also wear gloves when handling clean denuders.
- 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 (MSDS) 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 MSDS sheets for that chemical.
- 2.5 Always label secondary containers used in this procedure.
- 2.6 Work in a laboratory hood when transferring alcohol or solutions of alcohol, water, and sodium carbonate.

3.0 Equipment and Materials

- 3.1 Plastic or latex disposable gloves, sized to fit.
 - 3.2 Sodium carbonate, anhydrous powder, 500 g. EM Science, product No. SX00395-1.
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- 3.3 Glycerol, 500 mL. J. T. Baker, product No. M778-07.
 - 3.4 Methanol (methyl alcohol). 4 liter plastic bottle. J.T. Baker, product 9076-03.
 - 3.5 Funnel, glass, powder. 80 mm width, 60 degree slant, or similar. To transfer dry Na_2CO_3 powder to a volumetric flask.
 - 3.6 Laboratory tissues, for general use and for wiping exterior of denuders after coating.
 - 3.7 Distilled water, laboratory grade. Available from in-house supply equipped with Millipore Milli-Q Plus ultrapure water system, typical resistance, 18.2 megohm.
 - 3.8 Selection of sizes of glass graduated cylinders, up to 500 mL. Graduated to nearest 1 mL.
 - 3.9 Selection of sizes of glass volumetric flasks, up to 1000 mL.
 - 3.10 Spatula, stainless steel. For transfer of sodium carbonate from reagent bottle during weighing.
 - 3.11 Top-loading electronic balance. 200 g. capacity. Readable to nearest 0.1mg. Mettler AT400 or equivalent.
 - 3.12 Quality control test weights, 1, 5, 50, and 100 g. Balance readings should agree to within 0.0005 g of the true weight.
 - 3.13 Plastic caps for sealing denuder during application of coating solution and for protecting prepared denuders during storage. Caplug product EC-32.
 - 3.14 Source of clean, dry air or nitrogen. Flexible Tygon tubing and manifold (optional) for routing air or nitrogen to denuder tubes during drying stages.
 - 3.15 Chemcomb denuders, clean and dry, preweighed to the nearest 0.1 mg, equipped with caps. If the denuder is not clean, hold securely by its side and rinse from one end, then from the other end, with a stream of warm tap water for two minutes to dissolve the used Na_2CO_3 coating. Follow this by rinsing both ends and the exterior surfaces with a stream of laboratory deionized water. Denuders may be allowed to soak for several hours in deionized water before a final rinse with double deionized water. Finally, pass a stream of air or nitrogen through the denuder to remove most of the water. Cover the
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denuders with a laboratory tissue and let them air dry overnight before coating. If a clean 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 the denuder surfaces may then be dried quickly in a stream of air or nitrogen.

4.0 Preparation of Sodium Carbonate Coating Solution

- 4.1 This recipe is for preparation of one liter of 1 % (by weight) Na_2CO_3 , 1% (by weight) glycerol coating solution, dissolved in a 1:1 solution of water:methanol. Record all procedural and weighing information in a laboratory notebook. Other volumes may be used; adjust the weight accordingly. Density of water is 1.00 g/mL; density of methanol is 0.7914 g/mL. Thus, the mass of 500 mL of water plus 500 mL methanol is 896 g. Weigh out 8.96 g of anhydrous Na_2CO_3 . Then, using a powder funnel, quantitatively transfer the Na_2CO_3 into a labeled 1000 mL glass volumetric flask. Weigh out 8.96 g of glycerol into a small glass beaker and quantitatively transfer it to the volumetric flask as explained below.
 - 4.2 While working in a hood, add 500 mL of deionized water to the volumetric flask. A graduated cylinder may be used to deliver this volume. Pass the water through the powder funnel used to transfer the Na_2CO_3 and thus wash any remaining powder into the flask. Swirl the flask gently to dissolve the Na_2CO_3 . Next transfer the glycerol to the flask by pouring it from the beaker into the flask. Add small amounts of methanol to the beaker to dissolve the remaining glycerol and transfer the rinsate to the volumetric flask. Next, add sufficient methanol (approximately 500 mL) to the flask to bring the volume to the 1000 mL mark. Cap the flask, hold the cap in place, and turn the flask upside down several times to mix the contents thoroughly.
 - 4.3 Label the volumetric flask with the date of preparation and the contents. Set aside the capped flask for later use.
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5.0 Coating of Chemcomb™ Denuders with Sodium Carbonate Solution

- 5.1 Handle the clean denuders with plastic gloves. Rinse the outside of the gloves with deionized water and pat dry with laboratory tissues before beginning the following procedures. Inspect each clean Chemcomb denuder carefully to be sure it is not broken.
 - 5.2 Remove plastic caps and weigh the clean, dry denuder on a balance to the nearest 1.0 mg. An uncoated Chemcomb denuder will weigh between 75 and 100 g. Record information in a laboratory notebook. Return the denuder to the hood to start the coating process.
 - 5.3 Cap one end of the denuder with a solid polyethylene cap (Caplugs product EC-32). Be sure the cap is securely fastened, its rim is evenly placed around the circumference of the denuder, and it is not pressed fully against the denuder, but rather is slightly separated from the honeycomb surface.
 - 5.4 With one hand, hold the denuder by the side (capped end downward) and, with the other hand, dispense 10 mL of the coating solution directly into the center of the open end of the denuder. Use a graduated cylinder or dispensing pipette to measure the solution volume. The solution will flow through the denuder into the polyethylene cap.
 - 5.5 Close the open end of the denuder with another cap. This cap should have a small pin-hole in its center to allow air to escape. Affix the second cap securely and evenly.
 - 5.6 Hold the denuder in one hand by placing a thumb on the red cap that does not have a hole in it and use a finger to cover the hole in the opposite side's cap.
 - 5.7 Gently invert and reverse the denuder 10 times each way to mix the coating solution and apply it to the interior surfaces. Rotate the denuder lengthwise about 120 degrees and repeat the mixing. Rotate another 120 degrees and repeat. This process will ensure that all tubes in the denuder are completely coated. Do not shake the denuder during coating.
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- 5.8 Place the denuder on a laboratory tissue with the solid cap end down. Hold the sides of the denuder firmly with one hand, and with the other hand remove the cap with the hole in it. Set the cap aside for later cleaning with deionized water.
 - 5.9 Pour the excess coating solution into a waste beaker. Invert the denuder and gently tap the open end against a pad of laboratory tissues. This will remove some of the liquid coating solution that remains in the tubes.
 - 5.10 Remove the other cap, the one that has no hole, from the denuder. Set the cap aside for later cleaning. Gently tap this end of the denuder onto a stack of laboratory tissues to remove additional liquid from the honeycomb tubes.
 - 5.11 Wipe the outer solid surfaces of the denuder cylinder with a deionized water-moistened laboratory tissue to remove coating solution. Avoid touching or wiping the ends of the denuder. Wiping is important to prevent coating solution from later coming into contact with the interior of the aluminum cartridge cylinder.
 - 5.12 Attach a plastic cap, adapted to connect to a clean air or nitrogen source, to one end of the denuder. Allow air or nitrogen to gently flow through all channels of the denuder to dry the interior surfaces. A manifold may be constructed to allow several denuders to dry simultaneously. Refer to Figures 2-24 and 2-25 of the Operating Manual, ChemComb Model 3500 Speciation Sampling Cartridge.
 - 5.13 As soon as the denuder is dry (this will be determined by experience), remove the denuder from the drying assembly and reweigh it to the nearest 1.0 mg. Record the value and compare to the pre-coated weight to determine the approximate amount of coating material applied and the uniformity of application across several denuders.
 - 5.14 Promptly close both ends of the denuder with clean, dry Caplug EC-32 caps. Place the coated denuder in a plastic bag and zip the closure. Store in a cool, dry place until needed for assembling a sampling cartridge.
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