R&P Chemcomb Denuders **Revision** 4 Date: May 21, 2008 Page 1 of 6

Procedures for Coating R&P Speciation Sampler ChemcombTM Denuders with Sodium Carbonate

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Standard Operating Procedure for Coating R&P Speciation Sampler Chemcomb Denuders with Sodium Carbonate

1.0 Purpose and Applicability

This document outlines procedures for cleaning and coating the Rupprecht & Patashnick (R&P) Co., Inc., ChemcombTM 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 R&P 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) in case 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 number SX00395-1.
- 3.3 Glycerol, 500 mL; J.T. Baker, product number M778-07.
- 3.4 Methanol (methyl alcohol), 4 L plastic bottle; J.T. Baker, product number 9076-03.
- 3.5 Funnel, glass, powder; 80-mm width, 60-degree slant, or similar. Used to transfer dry Na₂CO₃ powder into a volumetric flask.
- 3.6 Laboratory tissues (for general use and for wiping exterior of denuders after coating).
- 3.7 Distilled laboratory-grade water. Available from in-house supply equipped with Millipore Milli-Q Plus ultra-pure water system, typical resistance, 18.2 MΩ.
- 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 1,000 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 0.1 mg. Mettler Toledo 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 number 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 are cleaned and dried and equipped with caps.

4.0 Cleaning R&P Denuders

- 4.1 Hold the denuder securely by its side and allow warm tap water to flow through the device from one end to the other.
- 4.2 Rotate the denuder, and then repeat the warm tap water rinse from other end.
- 4.3 Continue rinsing with warm tap water for approximately two minutes to dissolve the depleted Na₂CO₃ coating.
- 4.4 Rinse both the interior and exterior walls of the denuder with a stream of deionized water.

NOTE: If time permits, soak the denuders for several hours in deionized water before a final rinse with double deionized water.

4.5 Pass a stream of air or nitrogen through the denuder to remove most of the water. Cover the denuders with a laboratory tissue and let them air dry overnight before coating.

NOTE: If clean denuders are needed sooner, continue passing a stream of laboratory air or nitrogen through the channels until all surfaces are dry. Alternatively, ethanol or methanol can be poured through the channels to flush out the water and facilitate drying when using a stream of air or nitrogen.

5.0 Preparation of Sodium Carbonate Coating Solution

- 5.1 Record all procedural and weighing information in a laboratory notebook. Other volumes may be used; adjust the weight of sodium carbonate accordingly.
- 5.2 Prepare 1 L of 1% (by weight) Na₂CO₃, 1% (by weight) glycerol coating solution, dissolved in a 1:1 solution of water and methanol.

NOTE: The 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.

- 5.3 Weigh out 8.96 g of anhydrous Na₂CO₃.
- 5.4 Use a powder funnel to quantitatively transfer the Na₂CO₃ into a labeled 1,000 mL glass volumetric flask as described in Step 5.6.
- 5.5 Weigh out 8.96 g of glycerol into a small glass beaker and quantitatively transfer it to the volumetric flask as explained in Step 5.6.
- 5.6 While working in a laboratory 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 Na₂CO₃, and thus wash any remaining powder into the flask. Swirl the flask gently to dissolve the Na₂CO₃. Next, pour the glycerol from the beaker into the flask. Add small amounts of methanol into the beaker to dissolve the remaining glycerol and to transfer the rinsate to the volumetric flask. Next, add sufficient methanol (approximately 500 mL) into the flask to bring the volume to the 1,000 mL mark. Cap the flask, hold the cap in place, and turn the flask upside down several times to mix the contents thoroughly.
- 5.7 Label the volumetric flask with the date of preparation and the contents. Set aside the capped flask for later use.

6.0 Coating Chemcomb Denuders with Sodium Carbonate Solution

- 6.1 Handle the clean denuders with plastic gloves. Rinse the exterior 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.
- 6.2 Attach a red polyethylene cap (Caplugs product number EC-32) to one end of the denuder so that a void (space) is created between the cap and the bottom of the denuder. This void will contain the coating solution once it is poured through the channels in the denuder.
- 6.3 While holding the denuder by the side walls with one hand, pour 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.
- 6.4 While securely holding the denuder, close the open end of the denuder with another cap. The second (top) cap should have a small hole punched out of its center to allow air to escape. Make sure the cap is affixed securely and evenly.
- 6.5 Hold the denuder in one hand, placing a thumb on the bottom red cap and a finger over the hole in the top cap.
- 6.6 Slowly and gently rotate the denuder top to bottom 10 times so that the coating solution contacts the interior surfaces of the channels on one side of the denuder.

- 6.7 Rotate the denuder on its lengthwise axis one-quarter of a turn and then repeat the top to bottom rotation. Continue coating the interior channels by repeating the quarter turns until all interior channels have been contacted by the solution.
- 6.9 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. 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.
- 6.10 Remove the bottom cap 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.
- 6.11 While avoiding contact with the ends of the denuder, remove the coating solution by wiping the outer walls of the device with a laboratory wipe that is wetted with deionized water. Wiping is important to prevent coating solution from later coming into contact with the interior of the aluminum cartridge cylinder.
- 6.12 If the denuders are needed for immediate use, a very low flowing stream of nitrogen can be used 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. If covered with dry laboratory wipes, the devices can air dry for several hours, but they should not be allowed to sit in this manner for more than 24 hours.
- 6.13 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.