Standard Operating Procedure
Preparation, Evacuation, and Shipping of Gas Sample Vials V 1.0
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I. Purpose and Scope

This SOP describes the preparation, evacuation, and distribution of 12-mL gas sample vials for NEON headspace equilibration sampling.

II. Principle

Gas sample vials must be evacuated to < 0.2 Torr prior to sample collection. The evacuation chamber allows for septum-intact evacuation, reducing the risk of leaks and enabling more complete evacuation compared to traditional needle-based methods.

III. Equipment and Materials

- xyzTek ExeVacuatorr evacuation chamber
- Fisherbrand Maxima rotary vane vacuum pump, 2.8 CFM, maximum vacuum 1.5×10^{-3} Torr
- Chemglass Life Sciences vacuum gage, range 1×10^{-3} – 760 Torr
- Labco 12 mL Exetainer vials (part number 736W)
- Labco replacement double-wadded white caps (part number VC329)
- Centrifuge tubes (50 mL) and coolers for vial storage and shipping

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IV. Preparation of Vials

1. New vials:
    - Remove from packaging and inspect for cracks, debris, or manufacturing defects.
2. Reused vials:
    - Hold for 90 days after analysis before reuse.
    - Remove old labels, dirt, and debris.
    - Acid wash vials.
        - Rinse, submerge in 10% hydrochloric acid bath for ~24 hours, rinse three times with DI water, air dry.
    - Replace septa and cap with new double-wadded white cap (VC329).
3. Loosen caps and place vials upright in xyzTek ExeVacuatorr chamber.

V. Evacuation Procedure

1. Evacuate vials within two weeks of shipping to domains.
2. Connect evacuation chamber to vacuum pump.
3. Attach vacuum gauge inline to monitor chamber pressure.
4. Load up to seven vials per batch into the chamber.
5. Seal the chamber and start the vacuum pump.
6. Continue evacuation until gauge reads < 0.2 Torr.
    - This typically takes several minutes.
7. Once target vacuum is achieved, use the provided tool to cap each vial under vacuum before switching off the pump.
8. Slowly vent the chamber to atmospheric pressure.
9. Remove vials and check that each cap is tight.

VI. Quality Assurance / Quality Control

1. At least one vial per three evacuation batches (every ~21 vials) will be tested.
2. Fill a syringe with tap water and attach a 25-gauge needle.
   b. Pierce septum of evacuated vial and allow water to be drawn in by vacuum.
   c. After filling, observe bubble size inside the vial: a small or absent bubble indicates a good evacuation; large bubbles indicate inadequate vacuum.
3. Record QA/QC results in the vial preparation log.
4. If a test vial fails, test one vial from the previous two batches.
5. Re-evacuate the entire batch from which there is a failed test vial.

VII. Shipping

1. Ship vials to each NEON Domain Support Facility every 1–2 months according to the Battelle schedule. Vials will be evacuated within two weeks of shipment date.
2. Include 2–4 extra vials beyond the requested number for each shipment.
3. Store evacuated vials upside-down in 50-mL centrifuge tubes:
    - May–October: fill centrifuge tube with water to maintain seal. The vial cap will be submerged.
    - November–April: leave centrifuge tubes unfilled to prevent freezing during shipment.
4. Pack centrifuge tubes containing vials into coolers at room temperature:
    - Use reusable coolers from previous shipments whenever possible.
        - Note: never reuse coolers with hazardous or dry ice labels, even if labels have been removed or painted over.
    - If not available, use Uline Styrofoam coolers in cardboard boxes.

5. Ship via FedEx or UPS ground service.

VIII. Contingency Evacuation Method

If the xyzTek ExeVacuatorr is unavailable, vials can be evacuated using:

- Fisherbrand Maxima rotary vane vacuum pump
- Chemglass Life Sciences vacuum gauge
- Bev-A-Line manifold with three-way stopcocks
- 25-gauge needles for septum piercing

This method follows the same vacuum threshold (< 0.2 Torr) and QA/QC procedures.