

Standard Operating Procedure

Preparation, Evacuation, and Shipping of Gas Sample Vials V2

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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.

III. Equipment and Materials

- Fisherbrand Maxima rotary vane vacuum pump, 2.8 CFM, maximum vacuum 1.5×10^{-3} Torr
 - Inficon Pirani Gauge Enhanced 300 (PGE300) vacuum gage, range $1 \times 10^{-4} - 1000$ 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 and combust at 475°C for 5 hours
 - Replace septa and cap with new double-wadded white cap (VC329).
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V. Evacuation Procedure

1. Attach vacuum gauge inline to monitor vacuum pressure.

2. Attach 25-gauge needle to outlet via a two-way stopcock.
 3. Turn vacuum pump on (in the closed position) to warm up for ~5 minutes.
 4. Pierce septa with the needle and allow each vial to be evacuated for ≥ 1 minute.
 5. Confirm that the vacuum gauge is reading < 0.2 Torr (< 200 mTorr). Expect a much lower reading (< 10 mTorr).
 6. Remove vial from needle while vacuum pump is on.
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VI. Quality Assurance / Quality Control

1. Check at least every 25th vial.
 2. Check by filling the evacuated vial with 17 ml of ambient air to ensure it was properly evacuated.
 - Fill a 20-ml syringe with 17 ml of ambient air and attach a 25-gauge needle.
 - Pierce the septum of the evacuated check vial and allow air sample to be drawn in by vacuum. It should pull ≥ 11 ml of sample without any pressure placed on the plunger.
 - Use the plunger to push in the remaining air sample. Assess for any abnormal pressure or resistance.
 3. If a test vial fails, test every 5th vial working backwards to find when the issue emerged.
 4. Replace caps and septa and re-evacuate the entire batch from which there was a failed check vial.
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VII. Shipping

1. Ship vials to each NEON Domain Support Facility every 1–2 months according to the Battelle schedule.
 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.
 - November–April: leave tubes unfilled to prevent freezing during shipment.
 4. Pack centrifuge tubes containing vials into plastic bags labeled with the vial evacuation date.
 5. Pack the bags into coolers at room temperature:
 - Use reusable coolers from previous shipments whenever possible.
 - If not available, use Uline Styrofoam coolers in cardboard boxes.
 6. Ship via FedEx or UPS ground service.
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