XAD-2 TUBE SAMPLING IN AMBIENT AIR

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1.0 SCOPE AND APPLICATION

The purpose of the Standard Operating Procedure (SOP) is to define the procedure for XAD-2 tube sampling, which is utilized to identify specific contaminants in ambient air. The greatest selectivity of the XAD-2 sorbent is towards polyaromatic hydrocarbons (PAHs) and chlorinated pesticides.

A Quality Assurance Project Plan (QAPP) in Uniform Federal Policy (UFP) format describing the project objectives must be prepared prior to deploying for a sampling event. The sampler needs to ensure that the methods used are adequate to satisfy the data quality objectives listed in the QAPP for a particular site.

The procedures in this SOP may be varied or changed as required, dependent on site conditions, equipment limitations or other procedural limitations. In all instances, the procedures employed must be documented on a Field Change Form and attached to the QAPP. These changes must be documented in the final deliverable.

2.0 METHOD SUMMARY

XAD-2 tube sampling is performed by drawing a known volume of air using a sample pump calibrated to a specific flow rate through an opened XAD-2 tube that is comprised of a polystyrene/divinylbenzene-based polymer. As air is drawn through the tube during sampling, airborne chemicals adsorb onto the surface of the polymer. After sampling, the tubes are delivered to the laboratory for analysis.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

The polystyrene/divinylbenzene-based polymer that is used for sampling is housed in a glass-sealed tube. XAD-2 tubes most often used contain 150-milligram (mg) or 600-mg of this polymer. Both tubes are 8-millimeter (mm) outer diameter (OD) by 110-mm length containing two sections of sorbent separated by glass wool. For the 150-mg tube, the adsorbing section contains 100-mg of XAD-2 with a backup section of 50-mg of XAD-2. For the 600-mg tube, the adsorbing section contains 400-mg of XAD-2 with a backup section containing 200-mg of XAD-2. A greater volume of air can be drawn through the larger tube; thereby, providing a greater sensitivity. Sample stability is unknown; however, it is recommended that the samples be analyzed within 14 days of collection.

To preserve and store samples after sampling:

1. Place the supplied plastic caps on the XAD-2 tube ends.
2. Wrap the tube in aluminum foil and place the sample in a small bag. If samples have been collocated, place each tube in a separate bag.
3. Ship the tube(s) at less than or equal to (≤) 6 degrees Centigrade (°C) to the laboratory for analysis in accordance with the most current version of ERT-PROC-2004, *Sampling Packing and Shipping.*

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

High sampling flow rates, high temperature, and humidity may cause a decrease in the adsorption capacity of XAD-2 since it is considered to be hydrophobic. Keep the tube in a vertical position during sampling to prevent channeling of the absorbent. Ensure that the arrow on the tube, which indicates flow direction, is pointed to the pump. Contaminants from the front portion of the tube may migrate to the back portion of the tube, but refrigeration may minimize this migration.

Since compounds of interest may be lost to photodegradation by ultraviolet (UV) light, opaque tube holders

and cassettes should be used, and tubes and cassettes (if not opaque) should be wrapped in aluminum foil during sampling and shipment.

5.0 EQUIPMENT

- Personal Sampling Pump
- Sampling Stand
- Rotameter or Electronic Flow Meter
- XAD-2 Tubes (600-mg or 150-mg), SKC #226-30-04 or 226-30-06 or equivalent
- Pre-loaded 2 micron (µm), 37 mm polytetrafluoroethylene (PTFE) filter, SKC #225-1713 or equivalent (Optional pre-filter for particulate fraction)
- Tygon Tubing, for attaching the tube holder system to the suction side of the pump
- Sleeves or support tubes, to hold tubes in place
- Single or Dual Manifold Flow Controller (Optional)
- Tube Holder End, to support and seal the sampling tube within the plastic housing
- Filter Cassette Adapter (Gilian 800143 or equivalent Tygon Tubing) (Optional)
- Luer Adapter (Gilian 200156 or equivalent) (Optional)
- Cassette Opener (SKC 225-13-5A) or equivalent (Optional)
- Glass Tube Cracker
- Resealable Bags
- Small Bags, WHIRL-PAK or re-sealable plastic bag
- Plastic End Caps
- Air Sampling Worksheets and Sample Labels
- Chain of Custody Records
- Screwdriver Set
- SCRIBE Software and Printer

6.0 REAGENTS

This section is not applicable to this SOP; however, if equipment needs to be decontaminated, refer to the current version of ERT-PROC-2006, *Sampling Equipment Decontamination.*

7.0 PROCEDURES

7.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain and organize the necessary sampling and monitoring equipment.

3. Decontaminate or pre-clean equipment, and ensure that it is in working order. Pre-calibrate sampling pumps as described in Section 7.2.

4. Prepare scheduling and coordinate with staff, client, and regulatory agency, if appropriate.

5. Perform a general site survey prior to site entry, in accordance with the site-specific Health and Safety Plan (HASP).

6. Use stakes, flagging tape, Global Positioning System (GPS) or other appropriate means to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

7.2 Calibration Procedures

To save time in the field, sampling pumps can be pre-calibrated in the office prior to arriving at the site using a calibrated rotameter or an electronic flow meter. The calibration must be checked in the field prior to use, and upon completion of sampling. Ensure that the electronic flow meter used for calibration has been calibrated by the manufacturer within one year. Perform calibrations following directions established in the most current version of ERT-PROC-2118, *Rotameter Calibration.*

1. For flow rates **above** 750 cubic centimeters per minute (cc/min), assemble the calibration train as shown in Figure 1 (Appendix A), using the appropriate rotameter. If the desired flow rate is **below** 750 cc/min, assemble the calibration train as shown in Figure 2, using an electronic flow meter (use of a rotameter in the sampling range is also acceptable). As seen in the figures, the sampling train consists of (in order) the personal sampling pump, Tygon tubing connected to the protective housing holding the XAD-2 tube. The appropriate calibration device is affixed to the sampling inlet. Figure 3 depicts the set point for the rotameter based on the results of the last calibration.

   Refer to Figures 4, 5, and 6 respectively, for illustrations of sampling trains with a single manifold, without a manifold, and with a dual manifold for collocated low-flow samples. The calibration tube used is representative of the same lot of tubes that is used for sampling.

2. Turn on the pump and adjust the flow using the flow adjust mechanism on the manifold (if used) or on the pump itself, until the desired flow rate is achieved. This occurs when the center of the float ball on the rotameter is aligned with the rotameter's pre-calibrated flow rate value or until the electronic flow meter displays the desired flow rate value. A sticker on the rotameter should indicate this value as shown in Figure 3 (Appendix A).

3. If desired affix a sticker to the manifold (if used) and pump indicating the pre-calibrated flow rate and sampling media.

4. Remove the calibration tube from the sleeve. The pump and manifold (if used) are calibrated as a unit and should not be separated until the samples have been collected. If they are separated, a new calibration will need to be performed.

5. **DO NOT** use calibration tube for sampling.

6. If desired flow rate is not achieved, check the following:

   - Verify that the pump is operational
   - Ensure that all fittings on tube housing are secure
   - Check that the XAD-2 tube is not cracked beyond the tip
   - Verify that both ends of tube are open
   - Make sure that no other obstructions are present
   - Ensure that Tygon tube is free of cracks, holes or kinks.

7.3 Field Operation

1. Mobilize to a clean zone and calibrate the appropriate number of sampling pumps as described in Section 7.2. If the pumps were pre-calibrated, the calibration should be checked in the same manner. Fine tuning of the flow may be required.

2. Calibration of the sampling pumps prior to sampling may be conducted in a clean area near the sampling location or at the desired sampling location.

3. Crack the XAD-2 tube ends using a glass tube cracker.

4. Insert the XAD-2 tube in the sleeve that protects the tube from light with arrow pointing in the direction of air flow (the smaller media section of the tube is used for breakthrough and is positioned nearest the sampling pump).

5. Screw the tip onto the sleeve so the XAD-2 tube is held in place.

6. At low flow rates (<750 cc/min), attach the sleeve to a manifold for better flow adjustment. At high flow rates (>750 cc/min), XAD-2 tubes can sample without a manifold.

7. To set up the sampling train, attach one end of the Tygon tubing (approx. 2 foot) to the tip of the sleeve or manifold. Attach the other end of the tubing to the inlet of the sampling pump. Refer to Figures 4, 5, and 6 (Appendix A), respectively, for illustrations of sampling trains. For collocated samples, a second pump may be used, or for low flow rates a dual manifold may be used.

8. Adjust time on the sampling pump to the required sample time.

9. Place the XAD-2 tube in a position free from obstruction on a dowel rod or stand. Position sampling inlet in the downward facing position at a 45 degree angle in order to prevent intrusion of precipitation.

10. Verify calibration by connecting a rotameter or electronic flow meter with Tygon tubing and turning on the pump via the flow check button, adjust flow if needed. Record the start flow rate and start time on the Air Sampling Worksheet.

11. Record available weather data and observations (e.g., ambient temperature, barometric pressure, relative humidity, wind direction, precipitation) on the Air Sampling Worksheet or in a logbook.

12. Turn on the pump.

13. After the pump has run the full time, check the fault button to determine if the pump ran for the programmed time. The elapsed time should be displayed on the screen. The flow and battery check button can be used to post-check the flow rate.

14. Verify flow rate by connecting a rotameter or electronic flow meter with Tygon tubing and turning on the pump. Record the end flow rate and time on the Air Sampling Worksheet.

7.4 Post Operation Procedures

1. Record the sampling time (end and elapsed) on the Air Sampling Worksheet.
2. Record the end flow rate on the Air Sampling Worksheet.
3. Remove the XAD-2 tube from the sleeve.
4. Cap XAD-2 tubes with plastic caps immediately after sampling. Never use rubber caps.
5. Wrap the tube with aluminum foil and place the sample tube in a whirl-pak/re-sealable bag labeled with the sample ID #, total volume, and required analysis. If collocated samples have been collected, place each tube in a separate whirl-pak/re-sealable bag and assign a unique sample ID # to each tube. Initial and final flow rates and volumes should be measured/calculated for each tube, including dual-manifold.
6. Indicate all applicable information on the Air Sampling Worksheet (e.g., sample volume, ID #, location, date, and weather parameters).
7. Transfer data from Air Sampling Worksheets to SCRIBE.
8. Sample tubes must be stored in a refrigerated container or in a cooler with ice.
9. Prepare samples, including QC samples, for transport by packing them in a shipping container with ice; if needed, bubble wrap or Styrofoam pieces may be used to prevent breakage. Complete a Chain of Custody record.

Typically, XAD-2 tube sampling is utilized for PAHs. The appropriate analytical methodology should be determined prior to field activities.

8.0 CALCULATIONS

The total volume of a sample is calculated by multiplying the total sample time by the average flow rate of the start and end flow rates. The total volume for each sample must be indicated on the Chain of Custody record for the laboratory to calculate the data in micrograms per cubic meter ($\mu g/m^3$).

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The following general Quality Control (QC) procedures apply:

1. All data must be documented on Air Sampling Worksheets or within site logbooks and in SCRIBE.
2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the QAPP. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.
3. Records must be maintained, documenting the level of competency for the Contractor's personnel to operate the instrument.

The following specific QC activities apply:

1. Provide one field blank per sampling event or per 20 samples, whichever is greater, unless otherwise specified in the analytical method or project-specific QAPP. The field blank should be collected at the beginning of the sampling event and handled in the same manner as the sampling tube (break, seal, and transport) except that no air is drawn through it. Include pre-filters as part of the field blanks if they are used with the samples.

2. It is recommended to collect one collocated sample per sampling event or per 10 samples, whichever is greater. Collocated samples are two samples collected adjacent to each other during the same time period at the same flow rates. See the project-specific QAPP for final determination.

3. Submit a minimum of three XAD-2 tubes per manufacturer's lot of tube utilized per sampling event for the lab to use to calculate an average media blank concentration. In addition, field personnel need to supply the laboratory with a minimum of three additional XAD-2 tubes for the blank spike/blank spike duplicate analysis. Do not break the ends of these tubes.

10.0 DATA VALIDATION

Data verification (completeness checks) must be conducted to ensure that all data inputs are present for ensuring the availability of sufficient information. These data are essential to providing an accurate and complete final deliverable. The Contractor's Task Leader (TL) is responsible for completing the UFP-QAPP verification checklist for each project. The data generated will be reviewed and processed by the TL prior to distribution.

Results of the quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives. Analytical data will be evaluated by the Contractor's QA/QC Group.

11.0 HEALTH AND SAFETY

Based on Occupational Safety and Health Administration (OSHA) requirements, a site-specific health and safety plan (HASP) must be prepared for response operations under the Hazardous Waste Operations and Emergency Response (HAZWOPER) standard, 29 CFR 1910.120. Field personnel working for EPA's Environmental Response Team (ERT) should consult the Emergency Responder Health and Safety Manual currently located at https://response.epa.gov/_HealthSafetyManual/manual-index.htm for the development of the HASP, required personal protective equipment (PPE) and respiratory protection.

Specific hazards unique to XAD-2 tube sampling include:

1. Sharp edges associated with the tubes after they have been "cracked."

2. Walking and working surfaces, as well as possible heights, associated with access to sampling locations.

12.0 REFERENCES

(1) http://www.cdc.gov/niosh/docs/2003-154/ (accessed May 2016). NIOSH Method 5515, Polynuclear Aromatic Hydrocarbons by GC. In: *NIOSH Manual of Analytical Methods*, Fourth Edition, Third Supplement, Issue 2, 15 August 1994.

13.0 APPENDICES

A - Figures

APPENDIX A
Figures
SOP: ERT-PROC-2065-20
April 2020

FIGURE 1. Calibration Train (No Manifold)

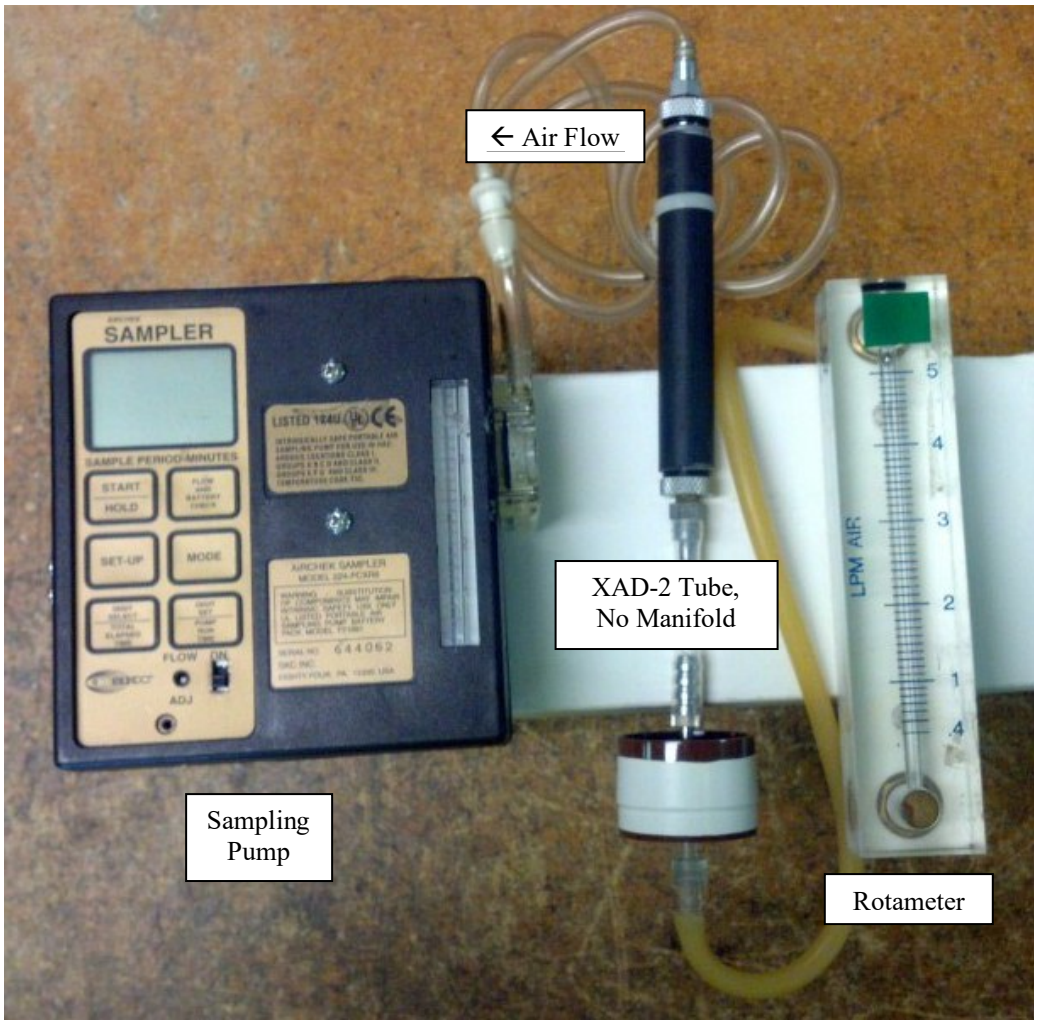


FIGURE 2. Calibration Train (Single Manifold)

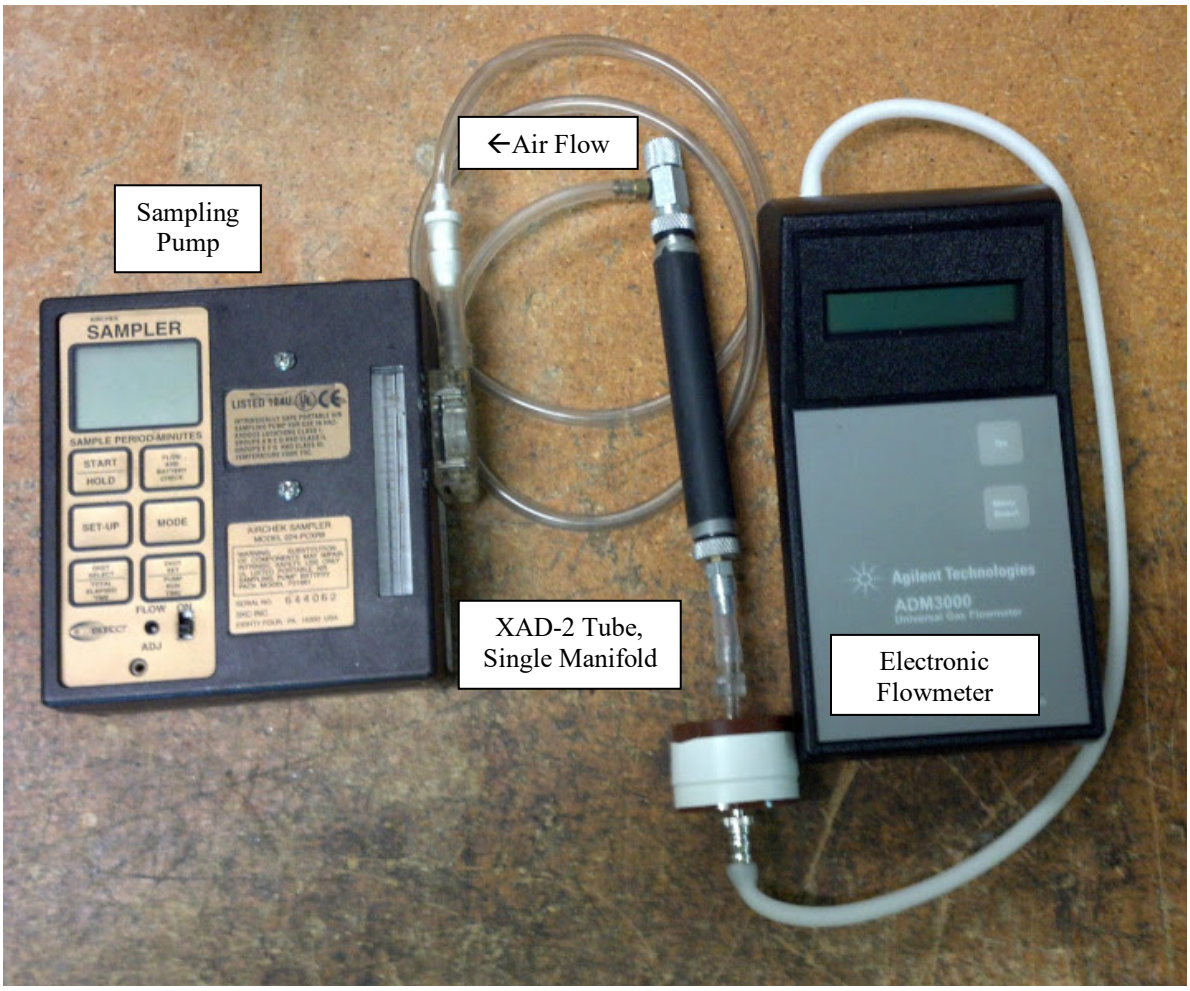


FIGURE 3. Rotameter Calibration Sticker

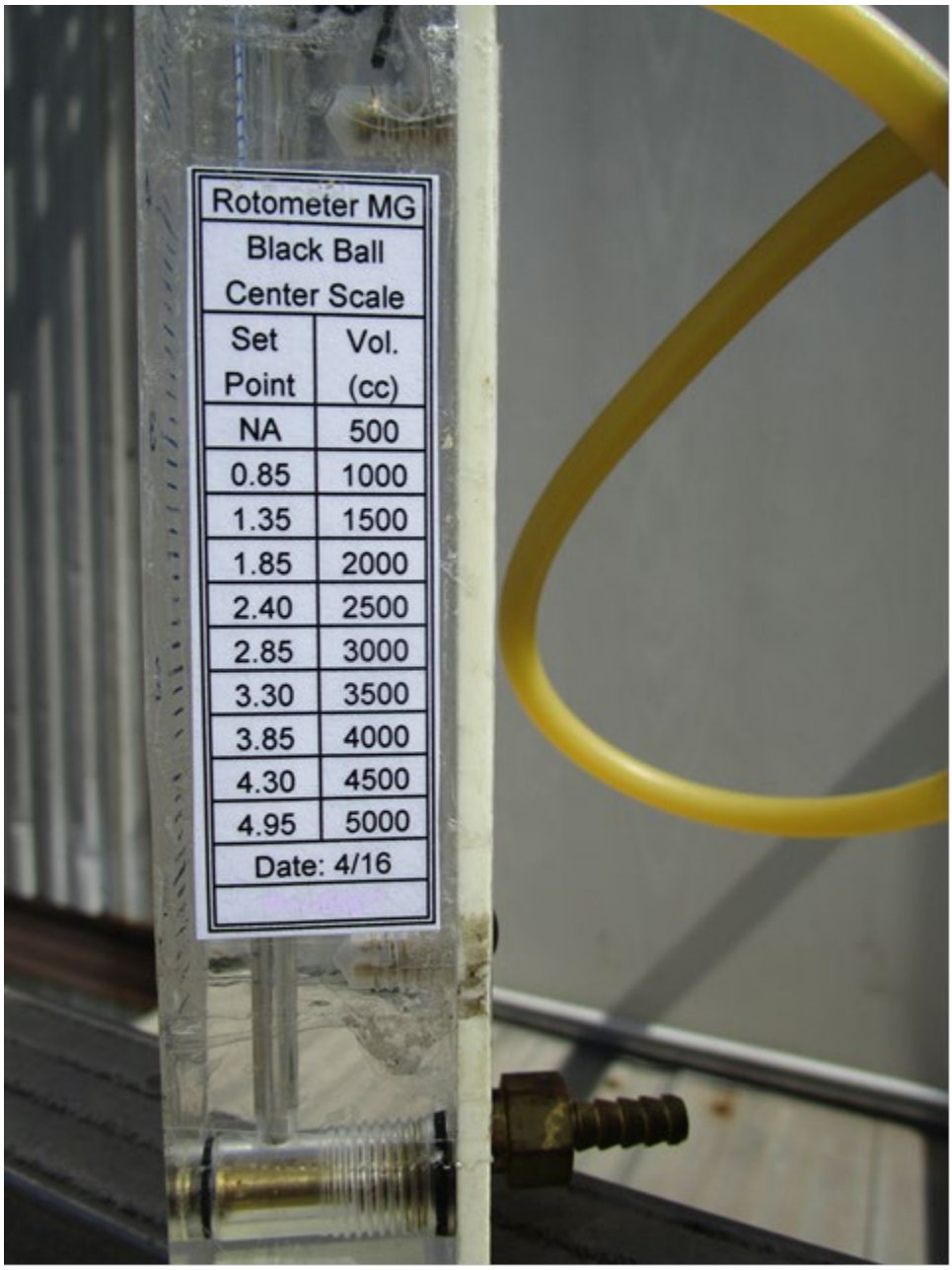
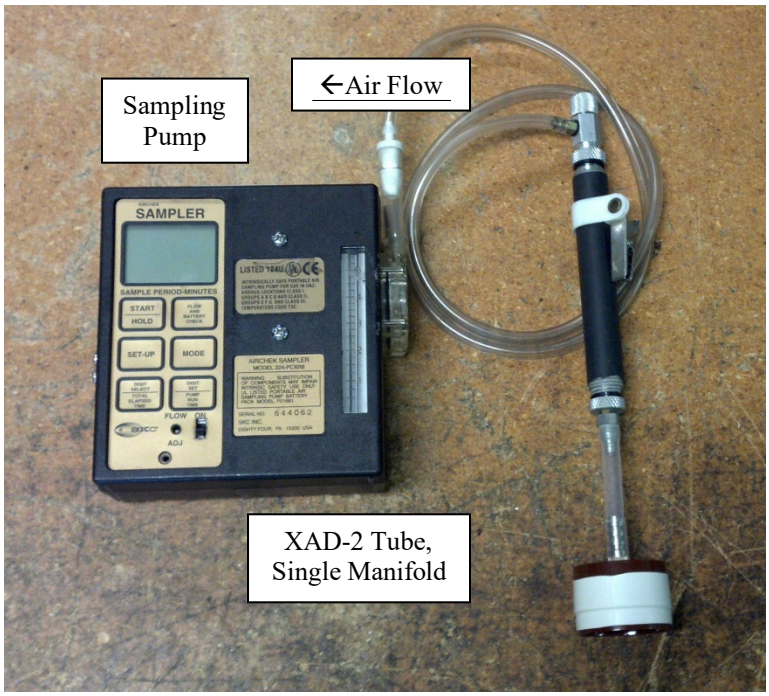


FIGURE 4. XAD-2 Tube Sampling Train (Single Manifold)
With Pre Filter



Without Pre-Filter

FIGURE 5. XAD-2 Tube Sampling Train (No Manifold) With Filter

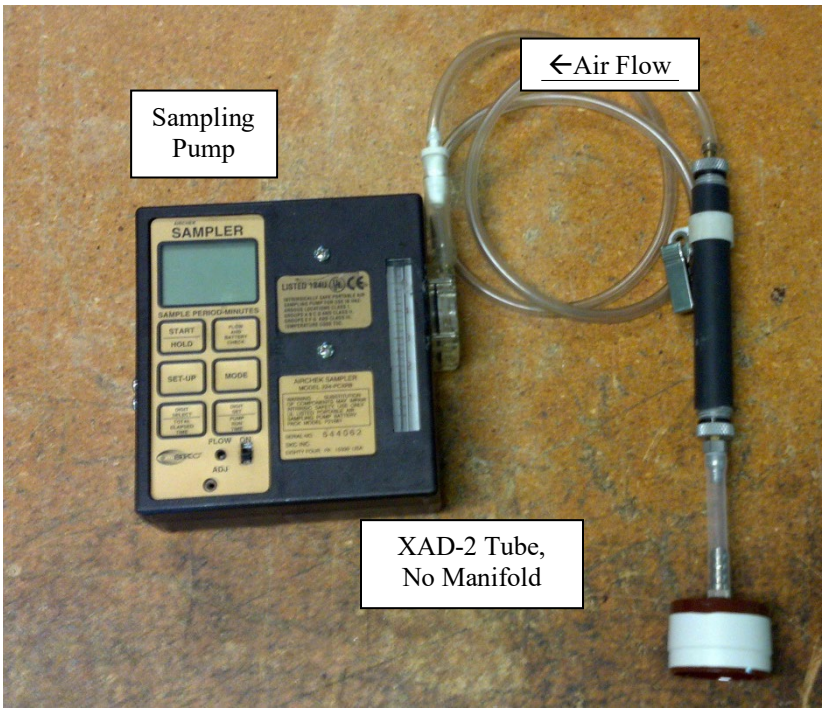


FIGURE 6. XAD-2 Sampling Train (Dual Manifold for Collocated Sampling)