



STANDARD OPERATING PROCEDURES

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LOW FLOW AIR SAMPLING FOR PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCBs) IN AIR

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1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to define the proper sample collection technique for air sampling of common pesticides and polychlorinated biphenyls (PCBs) in ambient air. This method is based on the sampling procedures outlined in EPA Method Toxic Organic (TO)-10A. Compounds typically covered by this method are listed in Table 1, Appendix A. This sampling method may be modified to include other compounds such as polychlorinated dibenzodioxins/furans, polybrominated biphenyls, polybrominated diphenyl ethers, polycyclic aromatic hydrocarbons (PAHs), and polychlorinated naphthalenes.

This sampling is designed to simultaneously collect suspended airborne particulates as well as trap airborne pesticide vapors. This type of collection can efficiently collect a number of organochlorine and organophosphate compounds. Polyurethane Foam (PUF) is typically used as a sample media, but sampling for these compounds is not limited to PUF only media.

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 UFP-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 that is attached to the UFP-QAPP. These changes must also be documented in the final deliverable.

2.0 METHOD SUMMARY

This is a low-volume sampling method, where 1- to 5-liters per minute (L/min, LPM) of sample is collected over a 4- to 24-hour period on a sorbent cartridge containing PUF or PUF in combination with another solid sorbent. The PUF media (plug) may come pre-loaded in sampling glassware or may be loaded into pre-cleaned glassware by the user. Whenever possible, the media should be provided by the analytical laboratory, spiked with surrogates required for the desired analysis.

The reporting limit (RL) will depend on the nature of the analyte and the collected sample volume.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

After sampling, the low-volume sample should be protected from light, wrapped with the foil in which it was originally packaged, labeled, and packaged for shipment. PUF media pre-loaded in glassware should be encased in aluminum foil, then wrapped in bubble wrap. The bubble-wrapped tube should then be stored in a glass jar (in which the media was received) and labeled as described herein. PUFs that were loaded by the user should be returned to the jars they were received in. Wrap the glass jar with aluminum foil (shiny side out) or use amber glassware and pack in a cooler with bubble wrap. When included, the sample filter should remain in the original cassette for pre-loaded filters, or folded and packaged with the corresponding PUF plug when loaded for sampling by the user. All samples should be labeled with the sample number, air volume, sample location, requested analysis and date and time sampled. Filters, when packaged separately, receive the same sample number as the PUF.

Samples must be maintained at less than or equal to (\leq) 4 degrees Centigrade ($^{\circ}\text{C}$). It is critical for the



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samples to be shipped to the laboratory as soon as possible since the extraction must be done within 7 days of collection.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The PUF media are purchased from the analytical laboratory or vendor and are pre-cleaned prior to use. If purchased from an analytical laboratory, the PUF media may also be spiked. PUF filters must not be re-used without cleaning. When sampling for light sensitive compounds, the PUF media must be in either an opaque holder or fully encased in aluminum foil to prevent loss of the compounds of interest.

5.0 EQUIPMENT/APPARATUS

The following equipment is required for low volume air sampling for Pesticides/PCBs:

- Personal Sampling Pump, SKC Universal XR Sampling Pump Model 224-PCXR8 or equivalent, capable of providing a flow rate of 1 to 5 L/min
- Sampling Stand, SKC Model Tripod Stand 228-506 or equivalent
- Rotameter SKC Rotameter 320-4A5, 320-4A20L or equivalent
- Re-Sealable Bags (Ziploc type or similar)
- Air Sampling Worksheets and Sample Labels
- Chain of Custody Records (SCRIBE preferred)
- Screwdriver Set, Universal Screwdriver Kit, SKC 224-11 or equivalent
- SCRIBE Software and Printer
- Particulate monitor, Thermo DataRAM or equivalent (Optional)
- Low volume PUF Tube Holder, which includes fitting with protective cover, 3 feet of tubing and collar clip SKC 224-29P or equivalent.
- Surgical gloves for handling the sampling media
- Personal Protective Equipment (Optional)
- Sampling media may include one or more of the following: low volume PUF tubes or their equivalents pictured in Figure 1, Appendix B: PUF/Tenax/PUF (30 millimeter [mm]/750 milligram [mg]/30 mm) SKC 226-124, PUF/glass fiber filter (76 mm) SKC 226-126, PUF (76 mm) SKC 226-92 (not pictured) and PUF/XAD-2/PUF (30 mm/1500mg/30mm) SKC 226-143 (not pictured).

Sampling media may also include PUF (76 mm) ORBO-1000 Sigma Aldrich 20557 with optional Filter Cartridge Assembly. The Filter Cartridge Assembly consists of an O-ring, stainless steel screen, quartz filter, filter ring and cartridge screw cap. Additional media not pictured is PUF/XAD-2/PUF (30mm/1500mg/30mm) ORBO-1500 Sigma Aldrich 21233U which is also available for use with the optional Filter Cartridge Assembly. The optional filter cartridge can be attached to the ORBO-1000 or ORBO-1500 cartridge to trap aerosol and particulate forms of semi-volatile compounds such as the compounds of interest referenced in Section 1.

While replacement uncleaned PUF filters SKC P22692 are available for repacking of SKC 226-92, they are not typically kept in stock by the ERT due to the limited ability to clean, pack and spike the PUFs to prepare them for sampling.



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6.0 REAGENTS

This section is not applicable to this SOP.

7.0 PROCEDURES

7.1 Field Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, the compounds of interest, flow rates, 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 equipment, if possible.
4. Prepare scheduling and coordinate with staff, client and regulatory agency, if appropriate.
5. If practical, 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, or other appropriate means to mark all sampling locations. If necessary, the proposed locations may be adjusted based on site access, property boundaries, surface obstructions and/or on-site activity.
7. If possible, make an estimate of the airborne concentrations of the elements of concern. It may be possible to extrapolate the concentration of particulates by assuming similar percentages of pesticides and PCBs present in the airborne particulates as in the soils. However, this is only a rough estimate; refer to the current version of ERT-PROC-2067, *Operation of the DataRAM4 Particulate Monitor*.
8. Arrange for pre-cleaned and spiked (if required) PUF media and subsequent sample analysis by an appropriately certified laboratory and check with the laboratory for any special requirements (e.g., additional lot blanks of PUFs). Laboratories generally require 3-5 business days lead time to obtain, clean, certify and spike PUF media due to the short shelf life after spiking. An additional one to two days shipping time, in addition to the 3-5 business day lead time, will be required depending on the location of field activities.

An example of a sampling train can be found in Figure 2, Appendix B.

7.2 Calibration

To save time in the field, the flow rate on the sampling pumps can be pre-calibrated prior to arriving at the site using a calibrated rotameter. The calibration of the flow rate must be checked in the field prior to use, and upon completion of sampling. Ensure that the (primary or secondary) calibration



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device has a valid calibration date. For ERT rotameters, perform calibrations following directions established in the current version of ERT-PROC-2118, *Rotameter Calibration*. Calibrate the sampling pumps in the following manner:

1. Assemble the calibration train as shown in Figure 3, Appendix B using a representative low volume PUF tube, Tygon tubing, low volume PUF tube holder, rotameter, and air sampling pump. Depending on the required flow rate, a low volume or a medium volume sampling pump may be required.
2. Turn on the pump and allow it to warm up for one minute. Adjust the flow 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. A sticker on the rotameter should indicate this value's set point as shown in Figure 4, Appendix B. **DO NOT** use this calibration PUF tube for sampling.
3. If desired flow rate is not achieved, check the following:
 - Verify that the pump is operational.
 - Ensure that all fittings on the PUF tube holder are secure.
 - Check that PUF tube holder is secure and not cracked (i.e. hairline cracks).
 - Make sure that no other obstructions are present.

Ensure that Tygon tubing is free of cracks, holes or kinks. As illustrated in Figure 3 Appendix B, the calibration train consists of (in order) the personal sampling pump, Tygon tubing connected to the protective housing holding the PUF tube, and the appropriate calibration device affixed to the sampling inlet. For PUF media that is not used with the protective housing, the PUF glassware should be wrapped in aluminum foil during sampling.

7.3 Sampling

1. Assemble the sampling trains with clean PUF tubes (Figure 2, Appendix B).
2. Deploy the sampling pumps as indicated in the UFP-QAPP, following site health and safety procedures. Ideally, the sampling pumps should be deployed in an area free from obstructions at a height of 3 to 5 feet. The cartridge should be positioned downward at a 45 degree angle.
3. Verify the pump calibration with the assembled sampling media. Ensure that all connections are tight, adjust flow if needed. Record the actual flow rate on the Air Sampling Worksheet (Appendix C). Record the serial number, barcode, or identification (ID) number of the calibration device used.
4. Program the sampling pump timer for the appropriate sampling time as determined by the data quality objectives (DQOs) for the project and as documented in the QAPP. Ensure that the Quality Control (QC) samples documented in the QAPP are set up and/or collected.



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5. Turn on the sampling pump and record the start time and date on the Air Sampling Worksheet.
6. After one minute, verify that the pump is running and that the timer on the face of the pump has incremented. Most faults occur during the first minute of operation.
7. After setting up multiple sampling locations, return to each location to verify pump sampling operation, if applicable and feasible.

7.4 Post Sampling

1. Verify the sampling period by reading the sample run time on the pump. Record the sampling time on the Air Sampling Worksheet (Appendix C) and turn off the pump.
2. Measure the pump flow rate by attaching a rotameter with Tygon tubing and turning on the sampling pump. Record the final flow rate on the Air Sampling Worksheet.
3. Complete the Air Sampling Worksheet and calculate the sample volume (refer to Section 8.0 for guidance on how to setup the calculation).
4. Remove the PUF media and any filters from the sampling train and return to a glass jar for labeling and shipment (refer to Section 3.0).
5. If collocated samples have been collected, place each sample in a separate jar and assign unique sample ID numbers to each sample.
6. Prepare the samples (including QC samples) for transport by packing them in a shipping container with bubble wrap or Styrofoam pieces in accordance with ERT-PROC-2004, *Sample Packing and Shipping*.
7. Document activities in accordance with ERT-PROC-2002, *Sample Documentation*. Enter all pertinent data into Scribe and print a Chain of Custody (COC) record from Scribe.
8. Sample custody must be maintained in accordance with ERT-PROC-4005, *Chain of Custody Procedures*.
9. When possible, do not immediately reuse PUF sampling glassware in the field because the glassware must be thoroughly decontaminated. To decontaminate, thoroughly rinse sampling glass ware with hexane and allow to air dry prior to reuse.

8.0 CALCULATIONS

The total volume (L) of a sample is calculated by multiplying the total sample time (total minutes) by the average flow rate (in LPM). The total volume for each sample must be indicated on the Chain of Custody Record.



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9.0 QUALITY ASSURANCE/QUALITY CONTROL

Specific quality assurance/quality control (QA/QC) activities that apply to the implementation of these procedures will be listed in the UFP-QAPP prepared for the applicable sampling event. The following general QA procedures will also apply:

1. All PUF sampling data, including the items listed in Section 10, must be documented in site logbooks or on field data sheets.
2. All equipment must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the UFP-QAPP. Equipment check-out and calibration is necessary prior to sampling and must be done according to the instruction manuals supplied by the manufacturer.
3. Records must be maintained, documenting the level of competency for the Contractor's personnel who will conduct the sampling.

The following specific QC activities apply:

1. Provide one field blank per sampling period or one for every 20 samples, whichever is greater, unless specified otherwise within the UFP-QAPP or the analytical method. The field blank media should be handled in the same manner as the sampling media with the exception that air is not drawn through the media.
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 UFP-QAPP for final determination.
3. Submit a minimum of three to five PUF cartridges per manufacturer's lot of tubes per sampling event for the lab to use as a media (lot) blank and for the blank spike/blank spike duplicate analysis. Consult with the laboratory prior to field mobilization.

10.0 DATA VALIDATION

Results of the QC samples (field and lot blanks) will be evaluated for contamination. This information will be utilized to qualify the sample results in accordance with the data quality objectives.

Data verification (completeness checks) must be conducted to ensure that all data inputs are present for ensuring the availability of sufficient information. This may include but is not limited to: Location, Sub-location, flow rate, flow meter ID number, and start and end times. 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.



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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.

Prior to initiating survey activities, a risk analysis is required to determine the hazards posed to sampling personnel. This will estimate any potential exposures to personnel, and define the extent of safety planning required to complete the task.

A safety plan is required prior to performing any site entry. In addition, real time monitoring may be necessary in order to verify ambient conditions and determine adequate respiratory protection.

12.0 REFERENCES

ASTM. 2005. ASTM Method D4861-11, Standard Practice for Sampling and Selection of Analytical Techniques for Pesticides and Polychlorinated Biphenyls in Air, ASTM International, West Conshohocken, Pennsylvania.

EPA. 1999. EPA Method TO-10A, Determination of Pesticides and Polychlorinated Biphenyls In Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detection (GC/MD), Center for Environmental Research Information, Office of Research and Development, U.S. EPA, Cincinnati, Ohio.

Sensidyne. 2008. Gilian Aircon-2 High Volume Air Sampler, Operation & Service Manual. Document No. F-PRO-3100, Rev. J, St. Petersburg, Florida.

SKC, Inc. 2012. Universal Sample Pump, Operating Instructions, Form #37711, Rev. 1302, Eighty-Four, Pennsylvania.

13.0 APPENDICES

- A – Table
- B – Figures
- C – Air Sampling Worksheet



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APPENDIX A

Tables

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TABLE 1 – Common Pesticide and PCB Compounds

PCBs in Ambient Air			
Analyte Description	CAS Number	RL -ug/sample	MDL ug/sample
PCB-1016	12674-11-2	1.00	0.200
PCB-1221	11104-28-2	1.00	0.313
PCB-1232	11141-16-5	1.00	0.353
PCB-1242	53469-21-9	1.00	0.116
PCB-1248	12672-29-6	1.00	0.235
PCB-1254	11097-69-1	1.00	0.309
PCB-1260	11096-82-5	1.00	0.289
Organophosphorous Pesticides in Ambient Air			
Analyte Description	CAS Number	RL -ug/sample	MDL ug/sample
Chlorpyrifos	2921-88-2	2.50	0.500
Demeton, Total	8065-48-3	5.00	1.00
Diazinon	333-41-5	2.50	0.500
Disulfoton	298-04-4	2.50	0.500
Ethion	563-12-2	2.50	0.500
Fenthion	55-38-9	2.50	0.500
Malathion	121-75-5	2.50	0.500
Methyl parathion	298-00-0	2.50	0.500
Ethyl Parathion	56-38-2	2.50	0.500
Pesticides (GC) in Ambient Air			
Analyte Description	CAS Number	RL -ug/sample	MDL ug/sample
Methoxychlor	72-43-5	0.0500	0.0184
Toxaphene	8001-35-2	0.0500	0.514
delta-BHC	319-86-8	0.0500	0.0210
Dieldrin	60-57-1	0.0500	0.0305
Endosulfan I	959-98-8	0.0500	0.0162
Endosulfan II	33213-65-9	0.0500	0.0249
Endosulfan sulfate	1031-07-8	0.0500	0.0238
Endrin	72-20-8	0.0500	0.0178
Endrin aldehyde	7421-93-4	0.0500	0.0242
gamma-BHC (Lindane)	58-89-9	0.0500	0.0261
Heptachlor	76-44-8	0.0500	0.0235
Heptachlor epoxide	1024-57-3	0.0500	0.0184
4,4'-DDD	72-54-8	0.0500	0.0169
4,4'-DDE	72-55-9	0.0500	0.0305
4,4'-DDT	50-29-3	0.0500	0.0196
Aldrin	309-00-2	0.0500	0.0167
alpha-BHC	319-84-6	0.0500	0.0312
beta-BHC	319-85-7	0.0500	0.0193
Chlordane (technical)	57-74-9	0.0500	0.162

ug/sample – micrograms per sample

RL – reporting limit

MDL – method detection limit



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APPENDIX B

Figures

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FIGURE 1. Examples of PUF Media



Other media (not pictured) include: SKC 226-143, SKC 226-92 and uncleaned PUF for repacking SKC 226-92.



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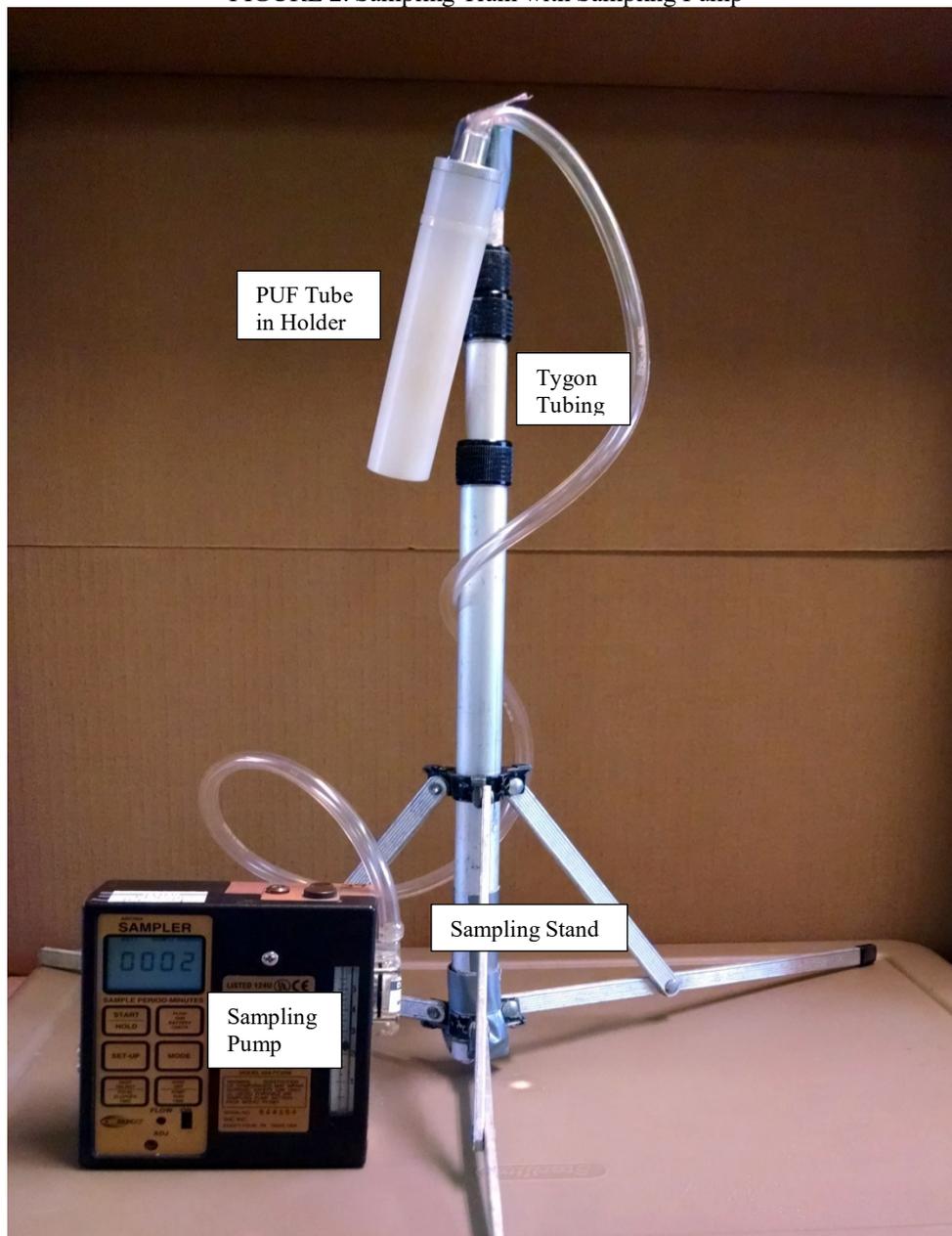
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FIGURE 2. Sampling Train with Sampling Pump





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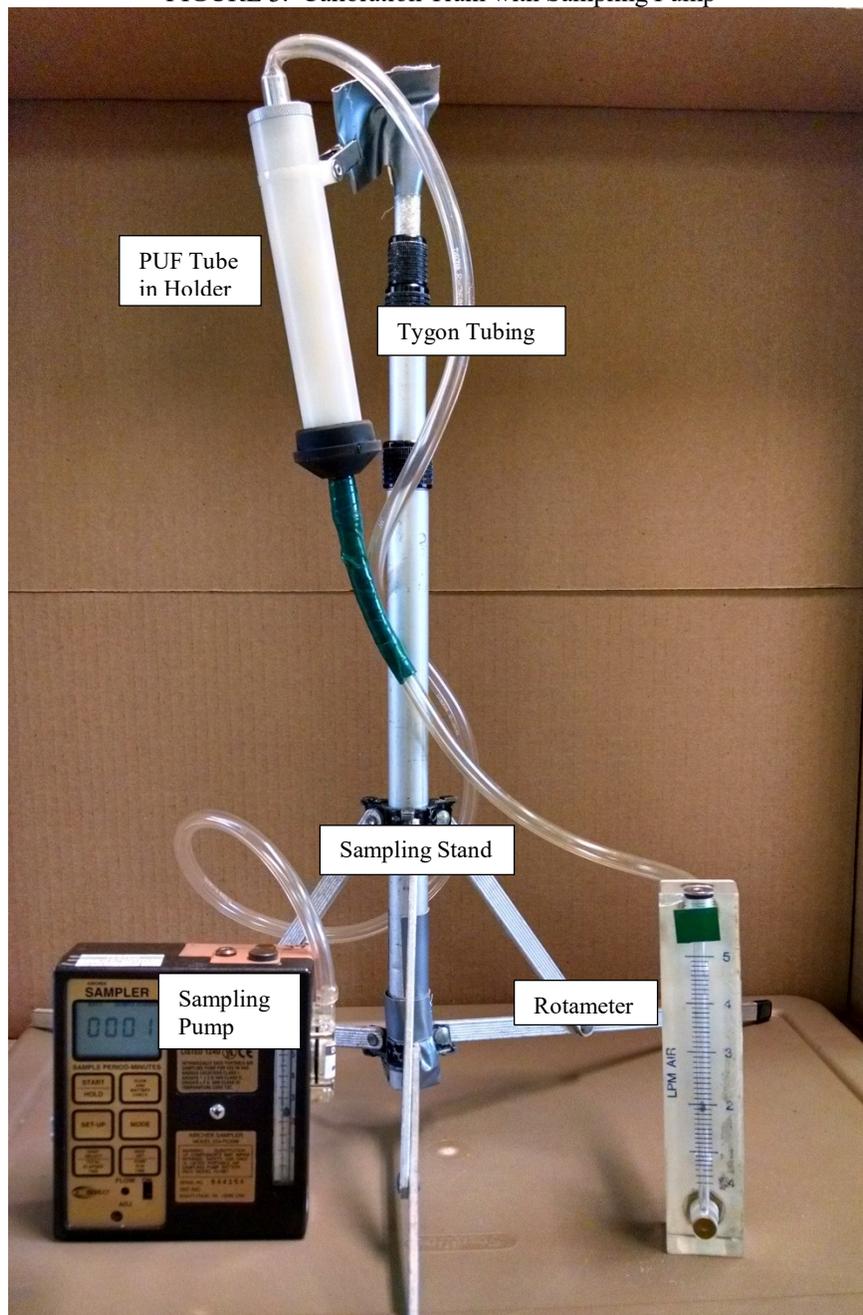
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FIGURE 3. Calibration Train with Sampling Pump





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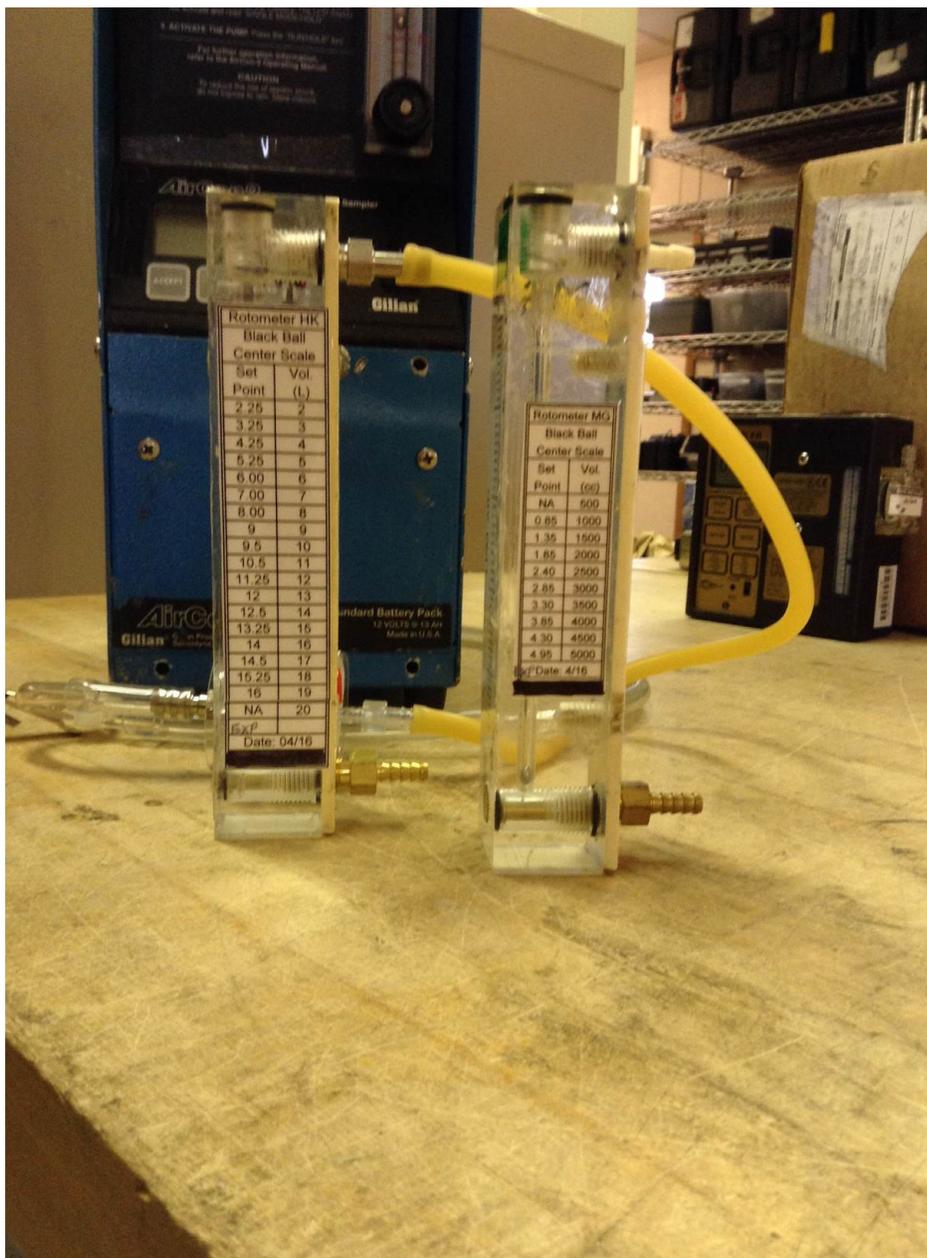
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FIGURE 4. Rotameter Flow Rate versus Set Point





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APPENDIX C
Air Sampling Worksheet
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Air Sampling Worksheet



EPA/Environmental Response Team
 Scientific Engineering Response and Analytical Services Contract
 Air Sampling Work Sheet
 Lockheed Martin Corp., Edison, NJ
 U.S. EPA Contract No. EP-W-09-031

Page ____ of



Site: _____

WA#: _____

Sampler: _____

U.S. EPA/ERT WAM: _____

Date: _____

SERAS Task Leader: _____

Sample #					
Location					
Pump #					
Media					
Analysis/Method					
Rotameter/ Calibration Device					
Time/Counter (Start)					
Time/Counter (Stop)					
Total Time					
Pump Fault	Y / N	Y / N	Y / N	Y / N	Y / N
Flow Rate (Start)					
Flow Rate (End)					
Flow Rate Average					
Sample Volume					
MET Station on Site?: Y / N					