MERCURY AIR SAMPLING

CONTENTS

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MERCURY AIR SAMPLING

1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to define the proper sample collection technique for sampling of mercury (Hg) in air. This method is based on National Institute for Occupational Safety and Health (NIOSH) Method 6009, *Mercury*. Refer to Table 1, Appendix A for sampling volumes.

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 (DQOs) listed in the QAPP for a particular site.

The procedures in this SOP may be modified, 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

Air samples of elemental Hg are collected on solid sorbent material (Hopcalite [or equivalent] in a single section, 200 milligram [mg]) contained in glass tubes according to NIOSH Method 6009. A pre-filter can be used to exclude particulate mercury species from the samples. The pre-filter can be analyzed by similar methodology. The particulate phase of the air, with a nominal size of greater than or equal to 0.8 microns (µm) is trapped in the filter. The sorbent material irreversibly collects elemental mercury.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

No preservatives or special storage conditions are required. However, the samples should be stored with the filter upright and transported at or near ambient conditions to prevent significant deterioration of the samples. When transporting and handling the samples, prevent impact and vibrations which would dislodge particulates from the filters. Ship sample tubes in accordance with the most current version of U.S. EPA Environmental Response Team (ERT) SOP, *Sample Packing and Shipping*.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Inorganic and organic mercury compounds may cause a positive interference.

5.0 EQUIPMENT/APPARATUS

The following equipment is required for air sampling for mercury:

- Personal Sampling Pump, SKC Universal XR Sampling Pump Model 224-PCXR8 or equivalent
- Sampling Stand, SKC Model Tripod Stand 228-506 or equivalent
- Electronic flow meter ADM3000, or equivalent
- Rotameter SKC Rotameter 320-4A1, or equivalent (Optional)
- 200 mg Hopcalite-equivalent tubes, Anasorb C300, SKC 226-17-1A
- Adjustable low-flow tube holder, SKC 224-16-01 or equivalent, or single manifold
- Mixed Cellulose Ester (MCE) Filters SKC 225-3-01 (0.8 µm) or equivalent (Optional)
- 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)

- Re-sealable Bags, Ziploc type
- Sample Bags, 18-ounce (oz) Whirl-Pak or Re-sealable plastic bag, Cole Parmer EW-06499-20
- Air Sampling Worksheets and Sample Labels
- Chain of Custody Records
- Screwdriver Set, Universal Screwdriver Kit, SKC 224-11 or equivalent
- SCRIBE Software and Printer
- Personal Protective Equipment (Optional)

6.0 REAGENTS

This section is not applicable to this SOP; however, if equipment needs to be decontaminated, refer to ERT SOP, *Sampling Equipment Decontamination.*

7.0 PROCEDURE

7.1 Field 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 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, global positioning system (GPS) coordinates 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. Arrange for sample analysis and check with the laboratory for any special requirements (e.g., additional lot blanks).

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 or an electronic flow meter. 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 device has a valid calibration date. For ERT rotameters, perform calibrations following directions established in the most current version of ERT SOP, *Rotameter Calibration.* Calibrate the sampling pumps in the following manner:

1. Assemble the calibration train as shown in Figures 1 and 2 (Appendix B) using a solid sorbent tube (Hopcalite in single section, 200 mg), Tygon tubing, adjustable low-flow tube holder or manifold, rotameter, and air sampling pump. If pre-filtering is used, include the pre-loaded 0.8-µm MCE filter cassette.

2. Turn on the pump and allow it to warm up for at least one minute. Adjust the flow using the flow adjust mechanism on the tube-holder or manifold until the desired flow rate is achieved (0.15 to 0.25 liters per minute [L/min], these are typical flow rates as specified in NIOSH 6009; flow rates may be adjusted to meet project objectives). This occurs when the center of the float ball on the rotameter is aligned with the rotameter's pre-calibrated flow rate value, or as a direct reading from the electronic flow meter. A sticker on the rotameter should indicate this value's set point as shown in Figure 3 (Appendix B). **DO NOT** use a calibration tube for sampling.

3. If multiple tubes and sampling pumps are utilized, affix a sticker to the pump indicating flow rate and media.

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

   - Verify that the pump is operational
   - Ensure that all fittings on tube holder are secure
   - Check that sample tube is secure and not cracked (i.e. hairline cracks)
   - Verify that both ends of tube are open
   - Verify that no glass shards have clogged manifold
   - Make sure that no other obstructions are present

   Ensure that Tygon tube is free of cracks, holes or kinks. As seen in the figures, the sampling train consists of (in order) the personal sampling pump, Tygon tubing connected to the manifold and tube holder and the appropriate calibration device is affixed to the sampling inlet.

7.3 Sampling

1. Assemble the sampling trains with sample tube (Figure 4, Appendix B) and (optional) with MCE cassette pre-filter, (Figure 5, Appendix B).

2. Low-flow sample rates can vary greatly from sorbent tube to sorbent tube, even within the same lot. Verify the pump calibration by attaching an electronic flow meter to the sorbent tube holder with the sampling pump running. Ensure that all connections are tight. Record the actual flow rate on the Air Sampling Worksheet (Attachment A, Appendix C).

3. Program the sampling pump timer for the appropriate sampling time as determined by the 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.

4. Deploy the sampling pumps as indicated in the QAPP, following site health and safety procedures. Verify calibration by connecting a rotameter or electronic flow meter and turning on the pump via the flow check button; adjust flow if needed. Record the start flow rate on the Air Sampling Worksheet. Record the serial number, barcode or ID number of the calibration device used.

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 and turn off the pump.

2. Verify the pump calibration by attaching an electronic flow meter or rotameter with Tygon tubing and turning on the sampling pump. Record the final flow rate on the Air Sampling Worksheet.

3. Remove the sorbent tube from the sampling train and cap both ends. If using a pre-filter, insert plugs to the inlet and outlet.

4. Complete the Air Sampling Worksheet and calculate the sample volume (see Section 8.0 for calculations.)

5. Place the solid sorbent tube in a Whirl-Pak bag labeled with sample ID #, total volume, and required analysis.

6. Prepare the samples (including QC samples) for transport by packing them in a shipping container, protect samples from breakage in accordance with the most current version of ERT SOP, *Sample Packing and Shipping*.

7. QC samples include a field blank. Prepare the field blank by breaking both sides of the tube and carry it through the field activities. Submit additional unopened tubes, which will be analyzed with the samples and used for the media blanks and QC samples (see Section 9.0).

7. Document activities in accordance with the most current version of ERT SOP, *Sample Documentation.* Enter all pertinent data into Scribe and print a COC record from Scribe.

8. Sample custody must be maintained in accordance with the most current version of ERT SOP, *Chain of Custody Procedures.*

8.0 CALCULATIONS

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

9.0 QUALITY ASSURANCE/QUALITY CONTROL

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

1. All sample collection data, including sample number, sample location, start and end times, start and end flow rates, pump number, media used and analysis/method must be documented on site logbooks or Field Sampling Worksheets.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer or instrument-specific SOPs, unless otherwise specified in the 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 training of the operators that use instrumentation and equipment for the collection of environmental information.

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 with 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 Hopcalite 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 Hopcalite 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. This may include but is not limited to location information, start and end times, sampling method and total volume sampled. These data are essential to providing an accurate and complete final deliverable. The ERT contractor’s Task Leader is responsible for completing the UFP-QAPP verification checklist for each project.

Results of the QA/QC samples will be evaluated for contamination during the data validation process. This information will be utilized to qualify the environmental sample results accordingly with the data quality objectives of the project.

11.0 HEALTH AND SAFETY

Based on Occupational Safety and Health Administration (OSHA) requirements, a site-specific 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 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.

12.0 REFERENCES

NIOSH Method 6009, Mercury. In: *NIOSH Manual of Analytical Methods*, Fourth Edition, Third Supplement, Issue 2, 15 August 1994. http://www.cdc.gov/niosh/docs/2003-154/ (accessed May 2016).

United States Environmental Protection Agency. 1995. *Superfund Program Representative Sampling Guidance. Volume 2: Air (Short-Term Monitoring).* EPA 540-R-95/140. Interim Final.

SKC, Inc. Universal Sample Pump, Operating Instructions. Form #37711. Rev. 1302.

13.0 APPENDICES

A Table
B Figures
C Attachment

APPENDIX A
Table
SOP: ERT-PROC-2107-21
January 2021

TABLE 1. Sampling Volumes

Element (Symbol)	Minimum Air Volume - Liters (@ 0.5 mg/m^3)	Maximum Air Volume – Liters* (@ 0.5 mg/m^3)	Validation Status
Mercury	2L	10L	Valid

* - higher volumes may be collected in order to demonstrate lower reporting limits based on individual project needs. Especially when trying to determine the cleanliness of an area.

APPENDIX B
Figures
SOP: ERT-PROC-2107-21
January 2021

FIGURE 1. Calibration Train with Personal Sampling Pump

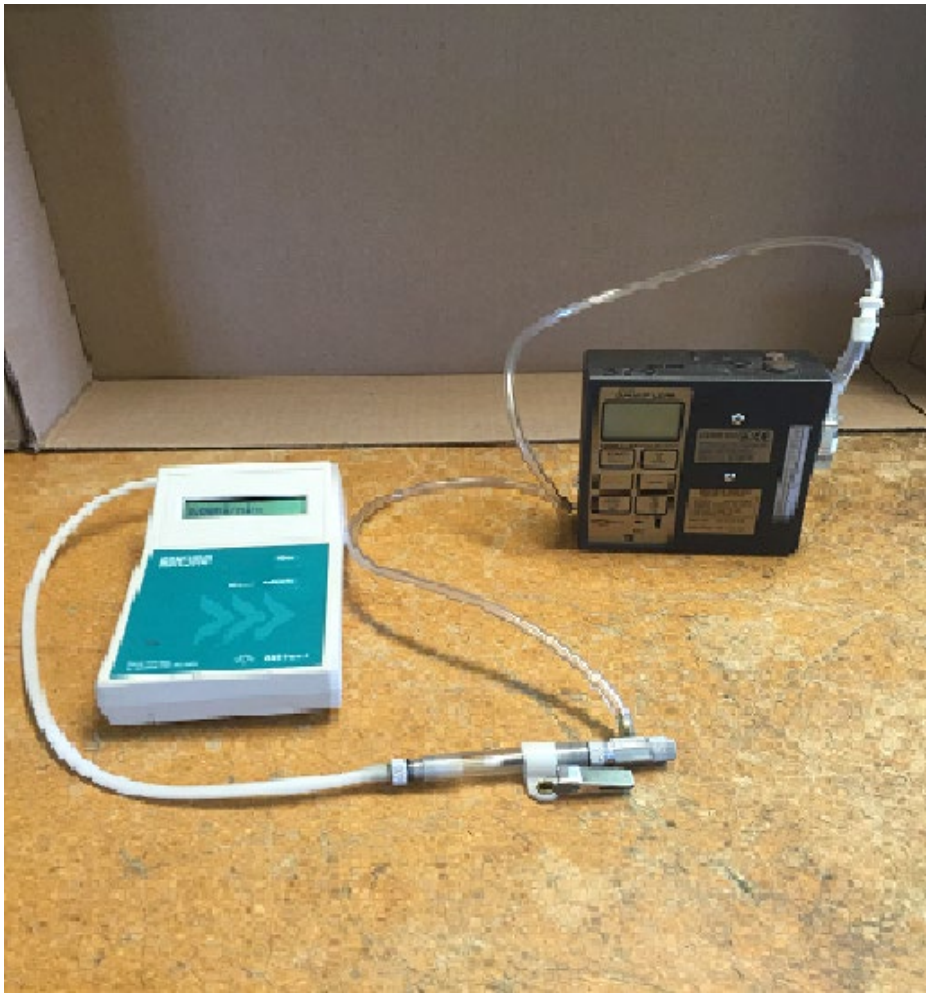


FIGURE 2. Calibration Train with Personal Sampling Pump and Filter

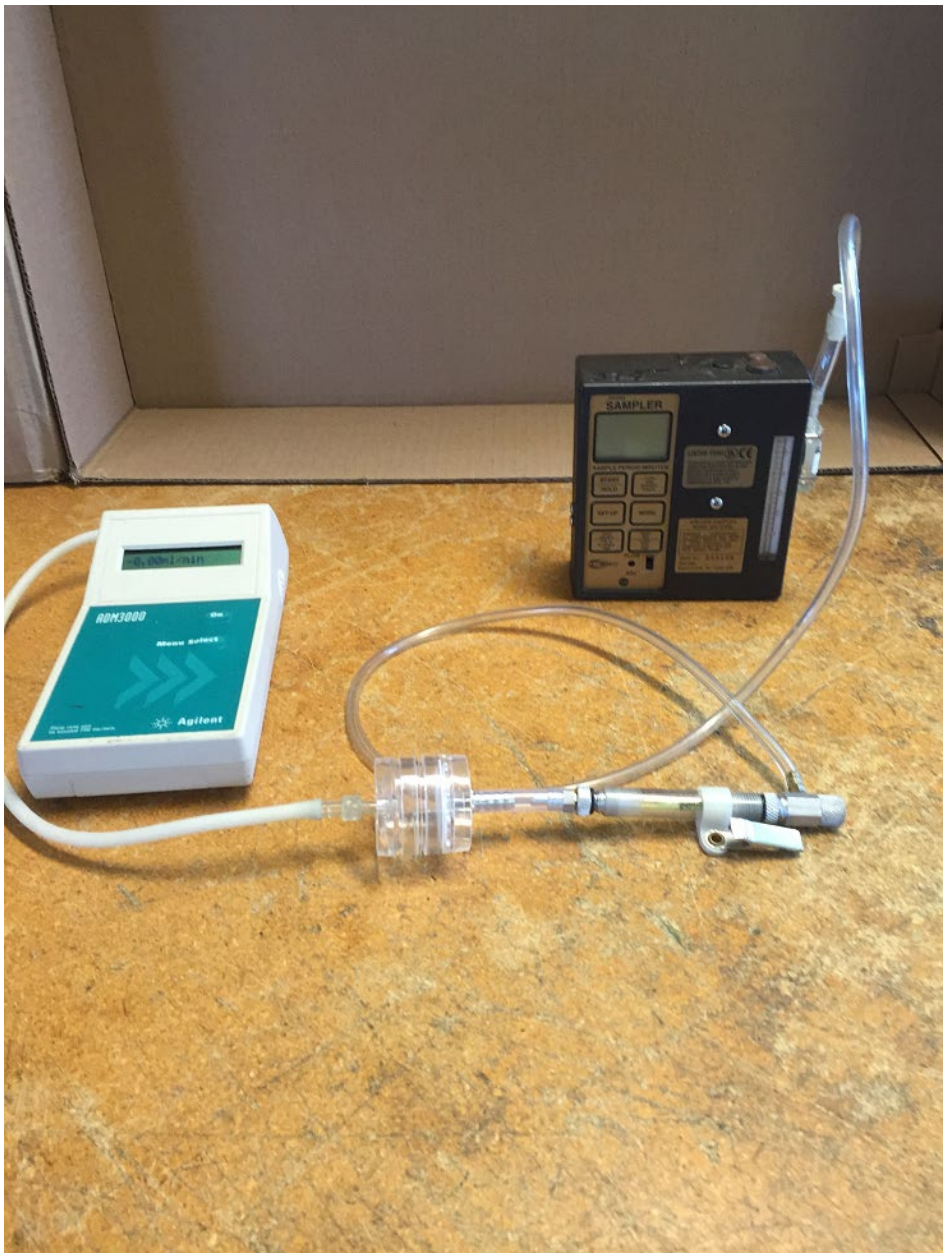


FIGURE 3. Rotameter Flow Rate versus Set Point

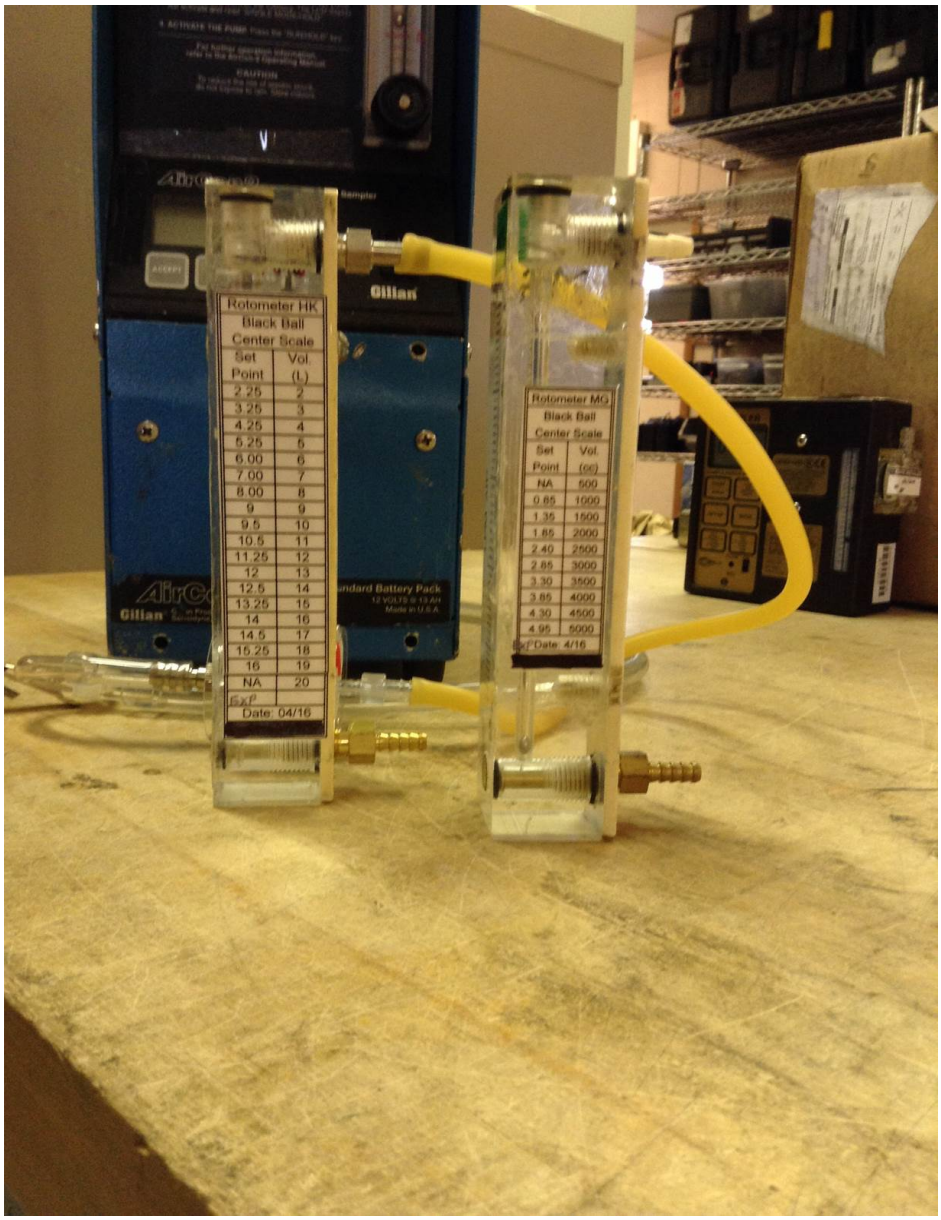


FIGURE 4. Sampling Train with Personal Sampling Pump

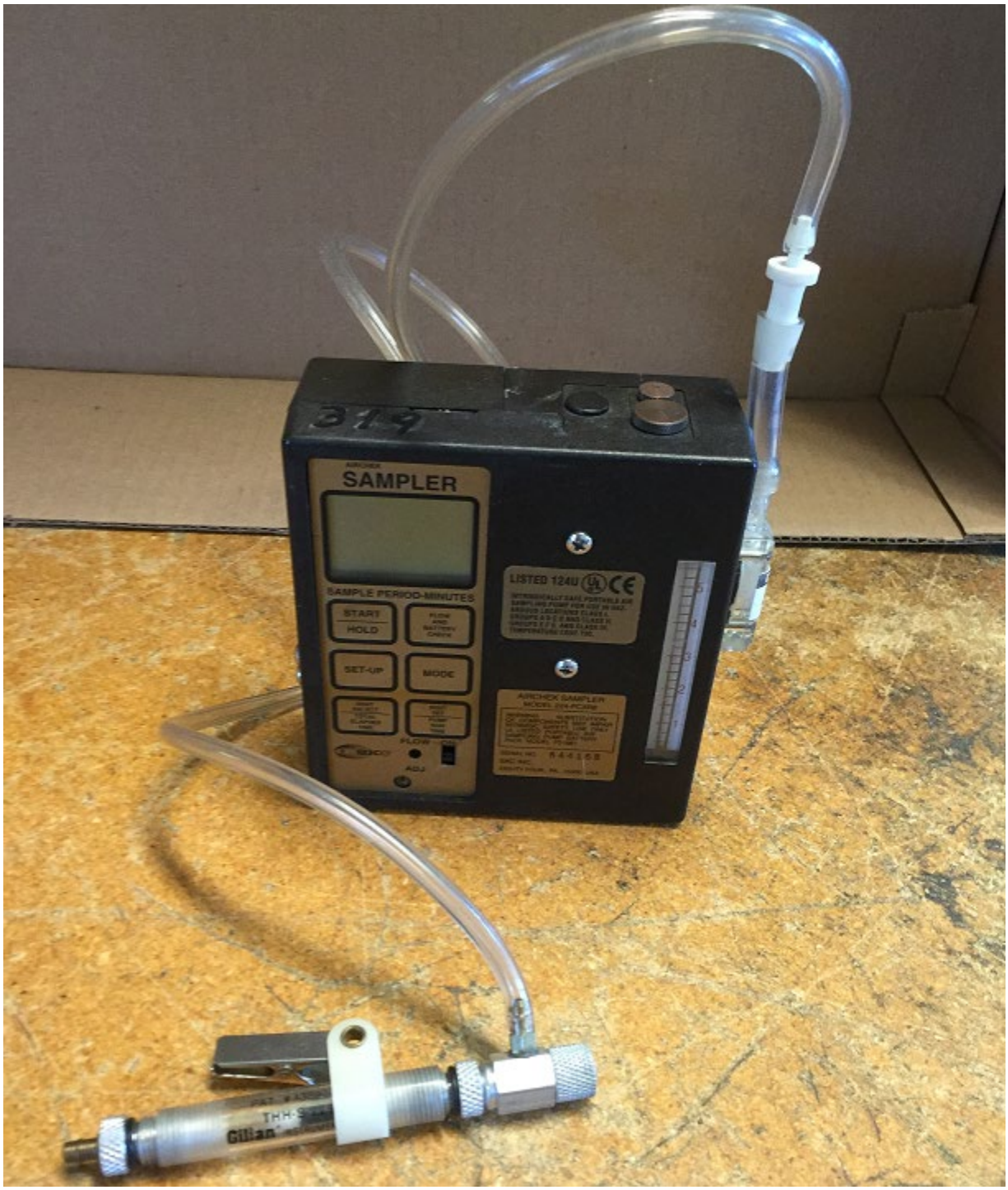
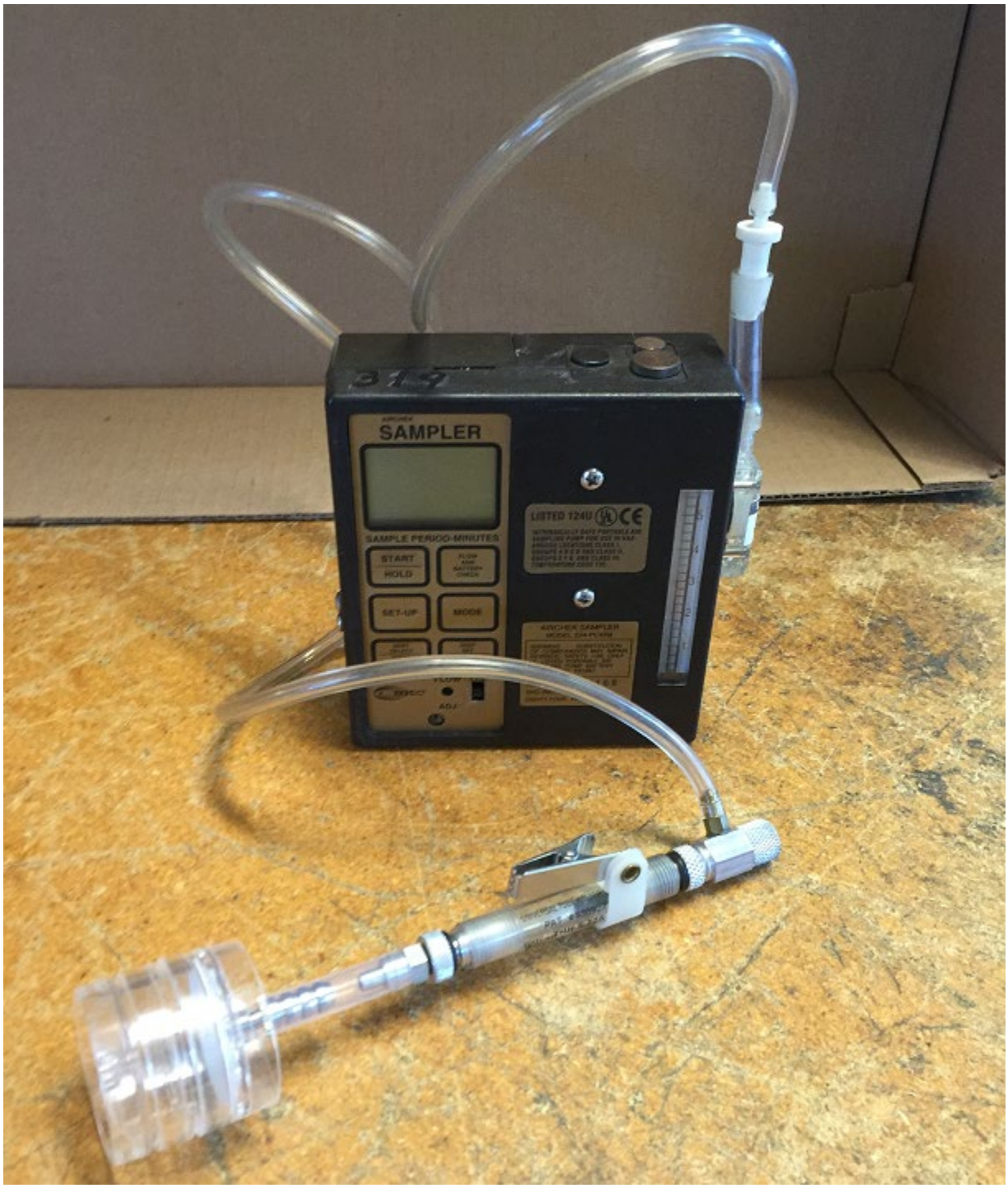


FIGURE 5. Sampling Train for an Area Sampling Pump



APPENDIX C
Attachment
SOP: ERT-PROC-2107-21
January 2021

Attachment1. Air Sampling Worksheet

Page _____ of

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

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					