AIR SAMPLING FOR METALS
[Based on NIOSH Method 7303, Elements by ICP (Hot Block/HCl/HNO_3 Digestion)]

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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 elemental metals, as well as delineate the typical working range of the method and indicate potential interferences. Elements covered by this method are listed in Table 1 (Appendix A).

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 sampling for elemental metals involves passing a known quantity of air across a mixed cellulose ester (MCE) filter. 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.

This method requires air sampling using 37-millimeter (mm), 3-stage cassettes loaded with 0.8 µm MCE filters and support pads. The approximate minimum and maximum sample volumes required for detection of the metals of interest are listed in Table 1 (Appendix A).

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.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

A potential problem with the sampling method is particulate overloading of the filter. This can disrupt air flow, consequently producing analytical results that may be biased low. Periodic checking of the filter and pump during sampling can reduce this error and sample cassettes can be changed during the sampling period In the event of heavy sample loading, multiple filters would be submitted and analyzed as a single sample. The total air volume must be indicated on the Chain of Custody (COC) record.

5.0 EQUIPMENT/APPARATUS

The following equipment is required for air sampling for elements:

- Personal Sampling Pump, SKC Universal XR Sampling Pump Model 224-PCXR8 or equivalent
- (Optional) Medium Sampling Pump capable of 10 liters per minute (LPM; Sensidyne AirCon-2 PN 801012 or equivalent)
- Sampling Stand, SKC Model Tripod Stand 228-506 or equivalent

- Rotameter SKC Rotameter 320-4A5, 320-4A20L or equivalent
- Mixed Cellulose Ester Filters SKC 225-3-01 (0.8 µm) or equivalent
- Filter Cassette Adapter (Gilian 800143 or equivalent Tygon Tubing)
- Luer Adapter (Gilian 200156 or equivalent)
- (Optional) Cassette Opener (SKC 225-13-5A) or equivalent
- Re-sealable Bags, Ziploc type
- Sample Bags (18-ounceWhirl-Pak or Re-sealable plastic bag), Cole Parmer EW-06499-20
- Plastic Cassette Plugs, SKC is part of SKC 225-3-01
- Air Sampling Worksheets and Sample Labels
- Chain of Custody Records
- Screwdriver Set, Universal Screwdriver Kit, SKC 224-11 or equivalent
- SCRIBE Software and Printer
- (Optional) Particulate monitor (Thermo DataRAM or equivalent)
- (Optional) Personal Protective Equipment

6.0 REAGENTS

This section is not applicable to this SOP.

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.

6. Use stakes, flagging tape or other appropriate means to mark all sampling locations. If necessary, the proposed locations may be adjusted based onsite access, property boundaries, surface obstructions and/or on-site activity.

7. 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 metals are present in the airborne particulates as in the soils. However, this is only a rough estimate. If estimation of the airborne concentration of metals is not possible, then sample volumes should remain within the limits recommended in Table 1 (Appendix A).

8. Arrange for sample analysis by an appropriately certified laboratory and check with the laboratory for any special requirements (e.g., additional lot blanks of cassettes).

7.2 Calibration

In order 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 U.S. EPA Environmental Response Team (ERT) rotameters, perform calibrations following directions established in ERT SOP, *Rotameter Calibration.* Calibrate the sampling pumps in the following manner:

1. Assemble the calibration train as shown in Figure 1 and 2 (Appendix B) using a representative 37-mm, 3-stage pre-banded cassette pre-loaded with a 0.8 µm MCE filter and support pad (outlet plug removed), Tygon tubing, hose-barb filter adapter, 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 five minutes. 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. A sticker on the rotameter should indicate this value's set point as shown in Figure 3 (Appendix B). **DO NOT** use this calibration filter cassette for sampling.

3. If multiple filters and sampling pumps are utilized, affix a sticker to the pump and media 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 cassette housing are secure
   - Check that cassette housing is secure and not cracked (i.e. hairline cracks)
   - Verify that both ends of cassette are open
   - 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 protective housing holding the cassette housing and the appropriate calibration device is affixed to the sampling inlet.

7.3 Sampling

1. Assemble the sampling trains with clean filter cassettes (Figures 4 and 5, Appendix B).

2. Verify the pump calibration by removing the inlet plug from the cassette, attaching a rotameter with Tygon tubing and turning on the sampling pump. Ensure that all connections are tight. Record the actual flow rate on the Air Sampling Worksheet (Attachment A, Appendix C). Replace the inlet plug until ready to sample.

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 sampling plan, following site health and safety procedures. 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 on the Air Sampling Worksheet. Record the serial number, barcode or ID number of the calibration device used.

5. Remove the cassette cap or inlet plug from the cassette. Sampling for elemental metals can be conducted with the cassettes open-faced (cassette cap removed) or closed-faced (only inlet port plug removed). Open-faced is preferred as it permits an even loading of the filter cassette and should be used whenever high particulate concentrations are expected. This allows greater particulate loading of the filter. Area and perimeter samples should be collected open-faced. Personnel samples should be collected with the closed-face set-up. Closed-faced sampling is typically performed when there is a possibility that the sample may be shaken and particulates may be lost. Either method is acceptable, since the entire filter is used during the sample analysis.

6. Turn on the sampling pump and record the start time and date on the Air Sampling Worksheet.

7. 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.

8. 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 a rotameter with Tygon tubing and turning on the sampling pump. Record the final flow rate on the Air Sampling Worksheet. Insert the inlet plug.

3. Remove the sampling cassette from the sampling train and insert the outlet plug.

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

5. Place the cassette in a Whirl-Pak bag labeled with sample ID #, total volume, and required analysis. If collocated samples have been collected, place each cassette in a separate Whirl-Pak bag and assign unique sample ID #s to each cassette.

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 SOP, *Sample*

Packing and Shipping.

7. Document activities in accordance with 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 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 will 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.

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. The collection of field blanks is recommended to evaluate the potential for contamination from media. The field blank (MCE cassette) should be handled in the same manner as the sampling cassette (remove/replace cap and plug, and transport) except that no air is drawn through it. Field blanks are collected with the frequency of one per sampling event.

4. Depending on the DQOs for the site, collect one collocated sample at the frequency of 5% or as documented in the site-specific QAPP. Collocated samples are two samples collected from two adjacent pumps during the same time period at the same flow rates.

5. A minimum of three lot blanks per lot of sampling cassettes utilized for a sampling event is typically sent to the laboratory for use as a method (media) blank and a blank spike (BS)/blank spike duplicate (BSD). Consult with the analytical laboratory to determine if additional lot blanks are required.

6. Records must be maintained, documenting the training of the operators that use instrumentation and equipment for the collection of environmental information.

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 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 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

National Institute for Occupational Safety and Health. 2003. *NIOSH Manual of Analytical Methods*. Method 7303. 4th Ed.

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.

Sensidyne, Gilian Aircon-2 Air Sampler, Operation and Service Manual. Document No. F-PRO-3100, Rev. J.

13.0 APPENDICES

A Table
B Figures
C Attachment

APPENDIX A
Table
SOP: ERT-PROC-2119-20
October 2020

AIR SAMPLING FOR METALS
[Based on NIOSH Method 7303, Elements by ICP (Hot Block/HCl/HNO_3 Digestion)]

TABLE 1. Sampling Volumes

Element (Symbol)	Minimum Air Volume - Liters	Maximum Air Volume – Liters	Validation Status
Aluminum (Al)	2(1)	10,000(1)	Valid
Arsenic (As)	8	5,000,000	Valid
Gold (Au)	1	3,300	Valid
Boron (B)	1	3,300	Valid
Barium (Ba)	1	100,000	Pending
Beryllium (Be)	35	25,000,000	Valid
Bismuth (Bi)	1	10,000	Partially Valid (2)
Calcium (Ca)	2	10,000	Valid
Cadmium (Cd)	3	500,000	Valid
Cobalt (Co)	3	500,000	Valid
Chromium (Cr)	8	500,000	Valid
Copper (Cu)	15	500,000	Valid
Iron (Fe)	1	5,000	Valid
Gallium (Ga)	1	3,300	Valid
Indium (In)	15	500,000	Valid
Magnesium (Mg)	1	10,000	Valid
Manganese (Mn)	0.05	10,000	Valid
Molybdenum (Mo)	0.5	10,000	Valid
Neodymium (Nd)	0.1	3,300	Valid
Nickel (Ni)	1	50,000	Valid
Phosphorus (P)	250(1)	500,000(1)	Valid
Lead (Pb)	35	100,000	Partially Valid (3)
Palladium (Pd)	0.1	3,300	Valid
Platinum (Pt)	200	25,000,000	Valid
Antimony (Sb)	3	100,000	Partially Valid (4)
Selenium (Se)	8	250,000	Valid
Tin (Sn)	1	25,000	Partially Valid (5)
Strontium (Sr)	300	100,000,000	Valid
Tellurium (Te)	125	500,000	Valid
Titanium (Ti)	0.1	10,000	Valid
Thallium (Tl)	35	500,000	Valid
Vanadium (V)	2.5	500,000	Valid
Yttrium (Y)	0.1	50,000	Valid
Zinc (Zn)	0.5	10,000	Valid

NOTE: Do not exceed a filter loading of approximately 2mg total dust.
(1)Larger volumes may be required if the anticipated concentration is less than the ACGIH Threshold Limit Value (TLV).
Valid: The method is suitable for samples up to at least 0.0500 g bulk material with recoveries of between 90 and 110 percent. This weight exceeds most expected levels encountered in work environments.
Partially Valid: The method is suitable with bulk-material recoveries of between 90 and 110 percent under certain conditions (as footnoted)
(2) Valid up to 10,000 µg/sample and within 7 days of sample digestion.
(3) Valid up to 50,000 µg/sample and at least 24 hours after sample digestion; Valid up to 15,000 µg/sample within 24 hours of sample digestion.
(4) Valid up to 25,000 µg/sample and within 7 days of sample digestion.
(5) Valid up to 30,000 µg/sample and within 7 days of sample digestion.

APPENDIX B
Figures
SOP: ERT-PROC-2119-20
October 2020

FIGURE 1. Calibration Train with Personal Sampling Pump

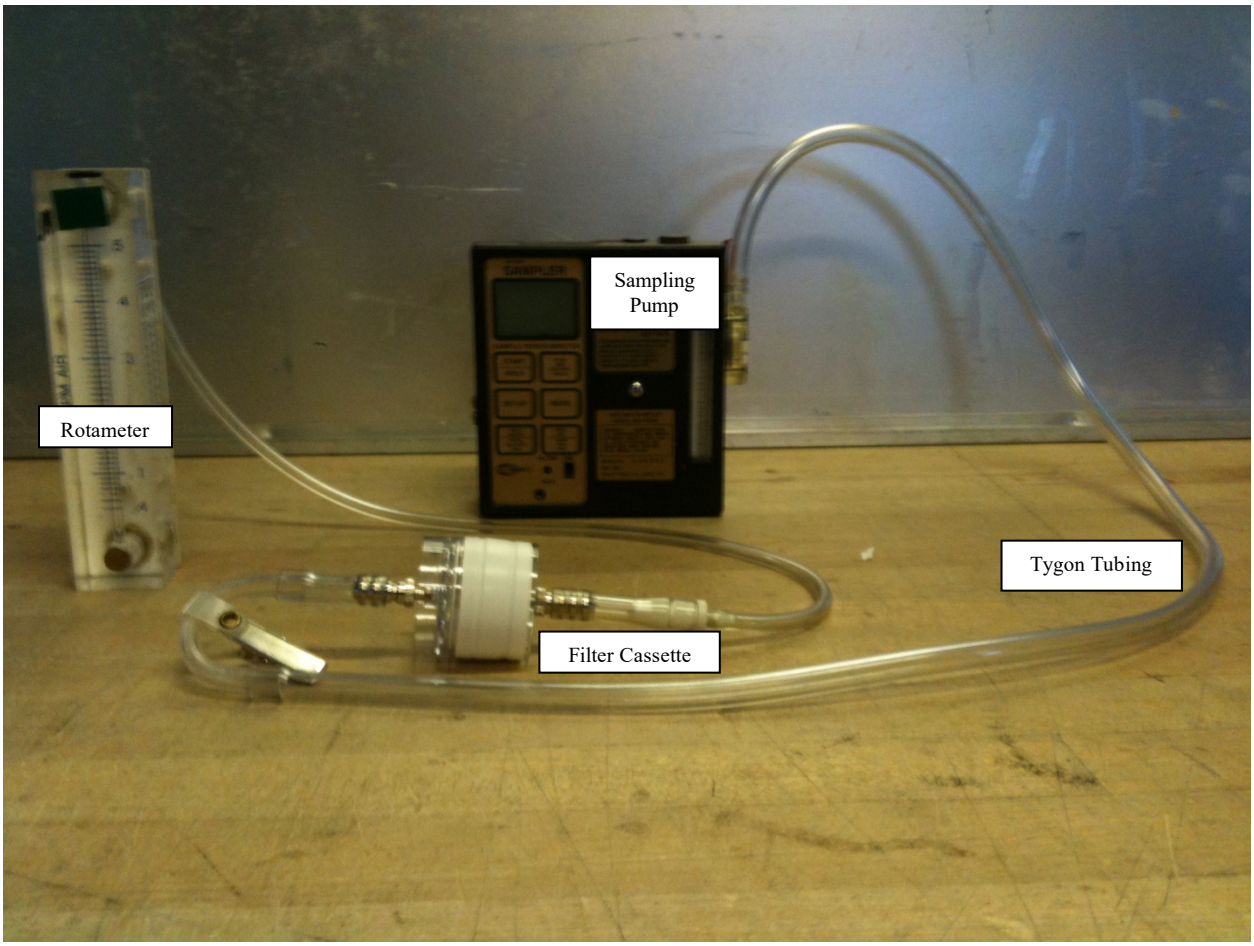


FIGURE 2. Calibration Train for an Area Sampling Pump

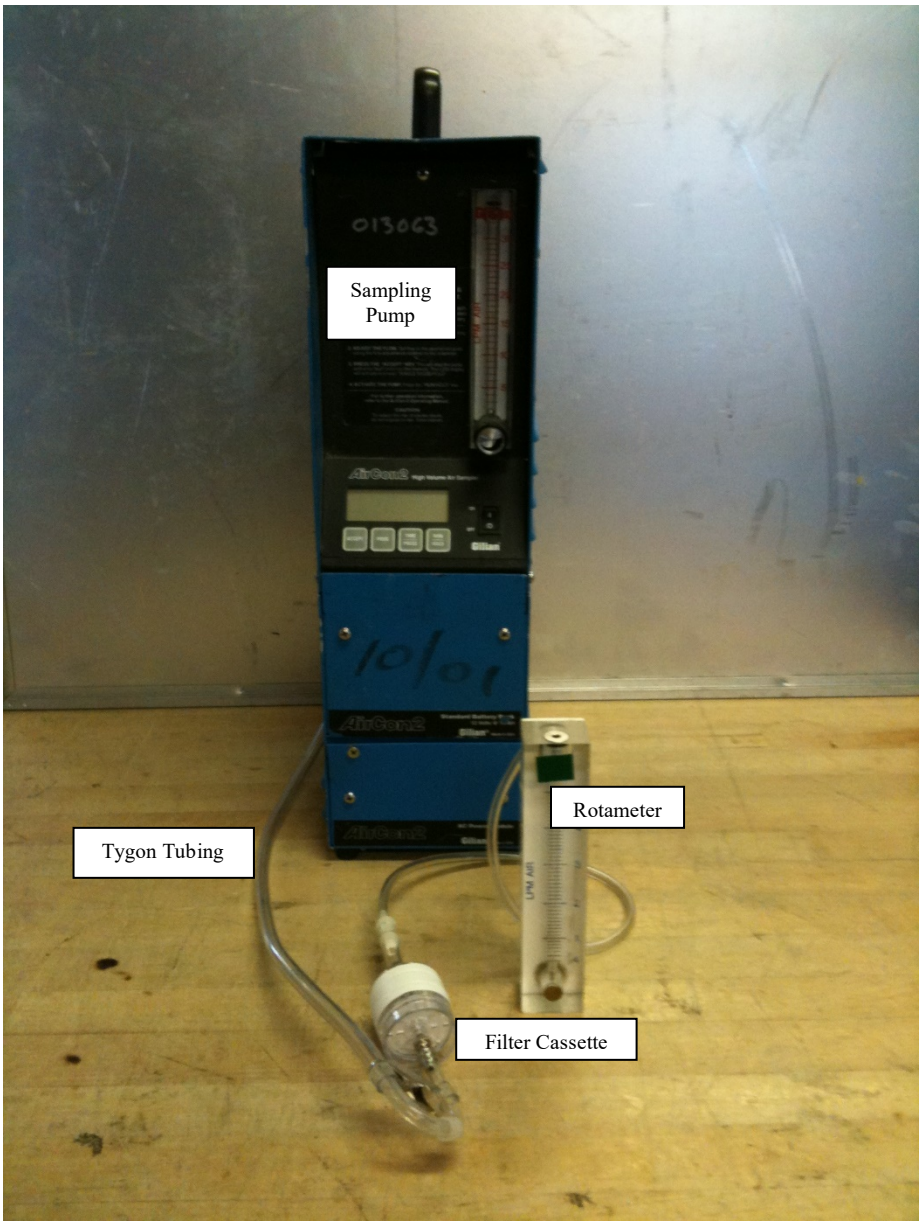


FIGURE 3. Rotameter Flow Rate versus Set Point

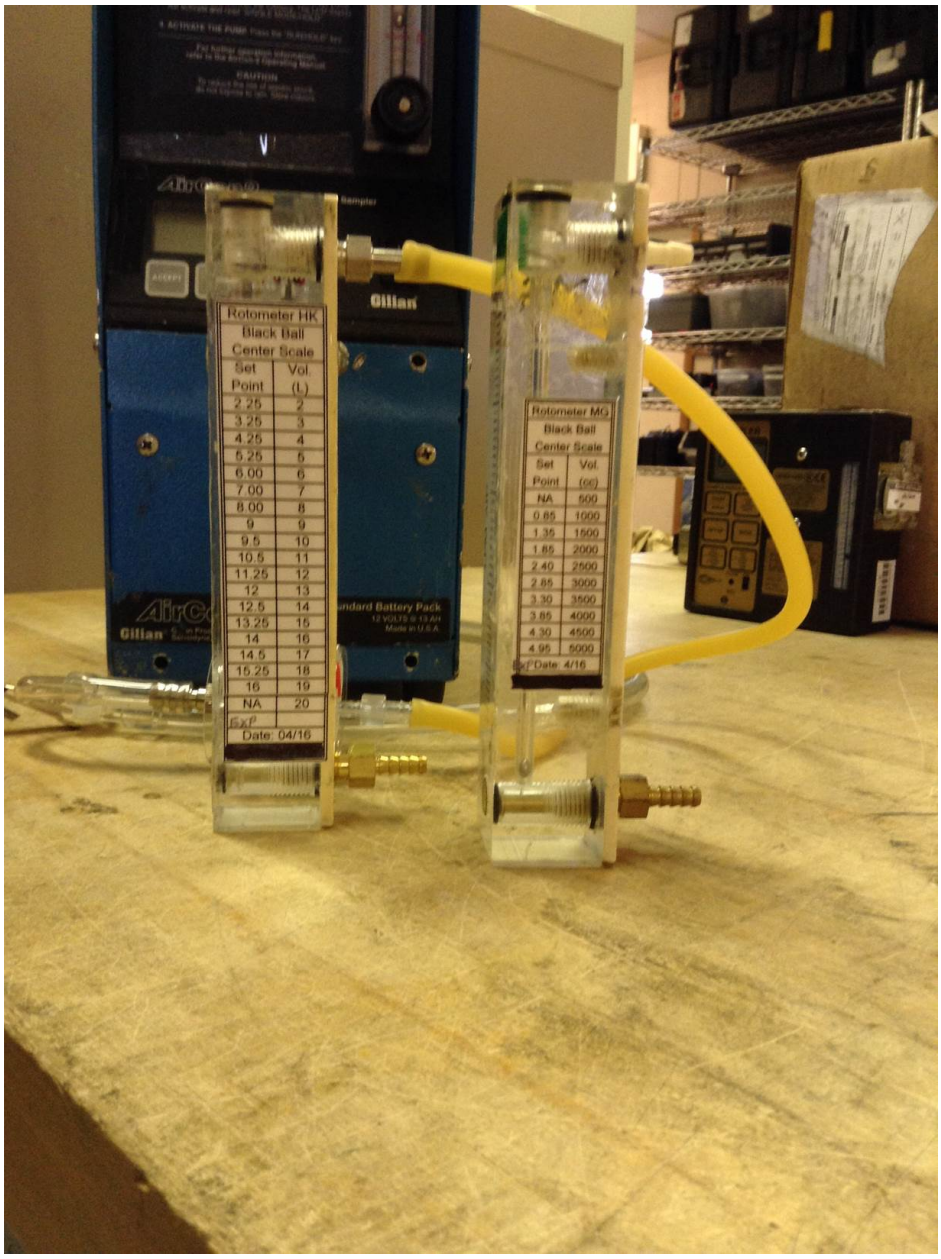


FIGURE 4. Sampling Train with Personal Sampling Pump

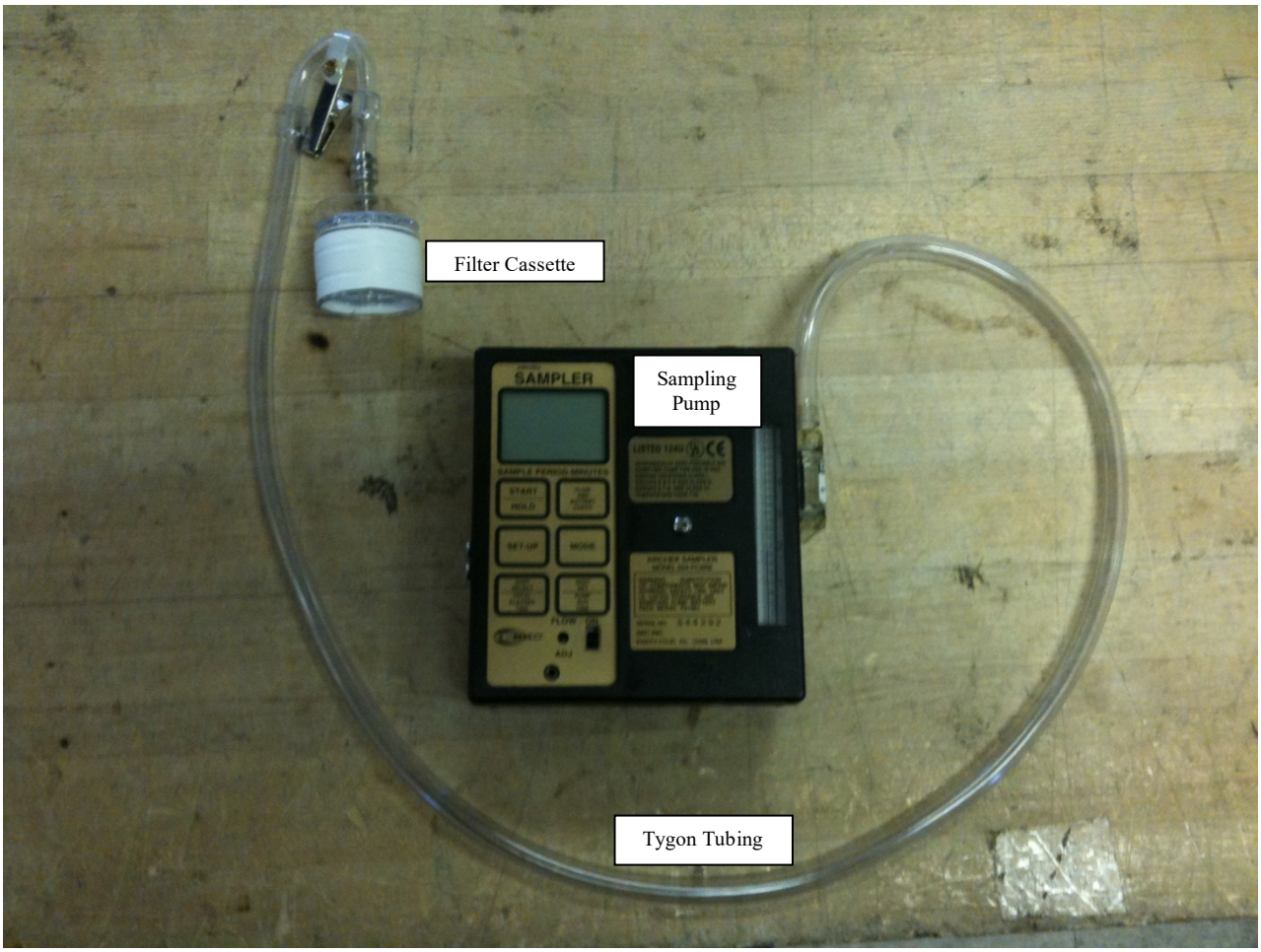
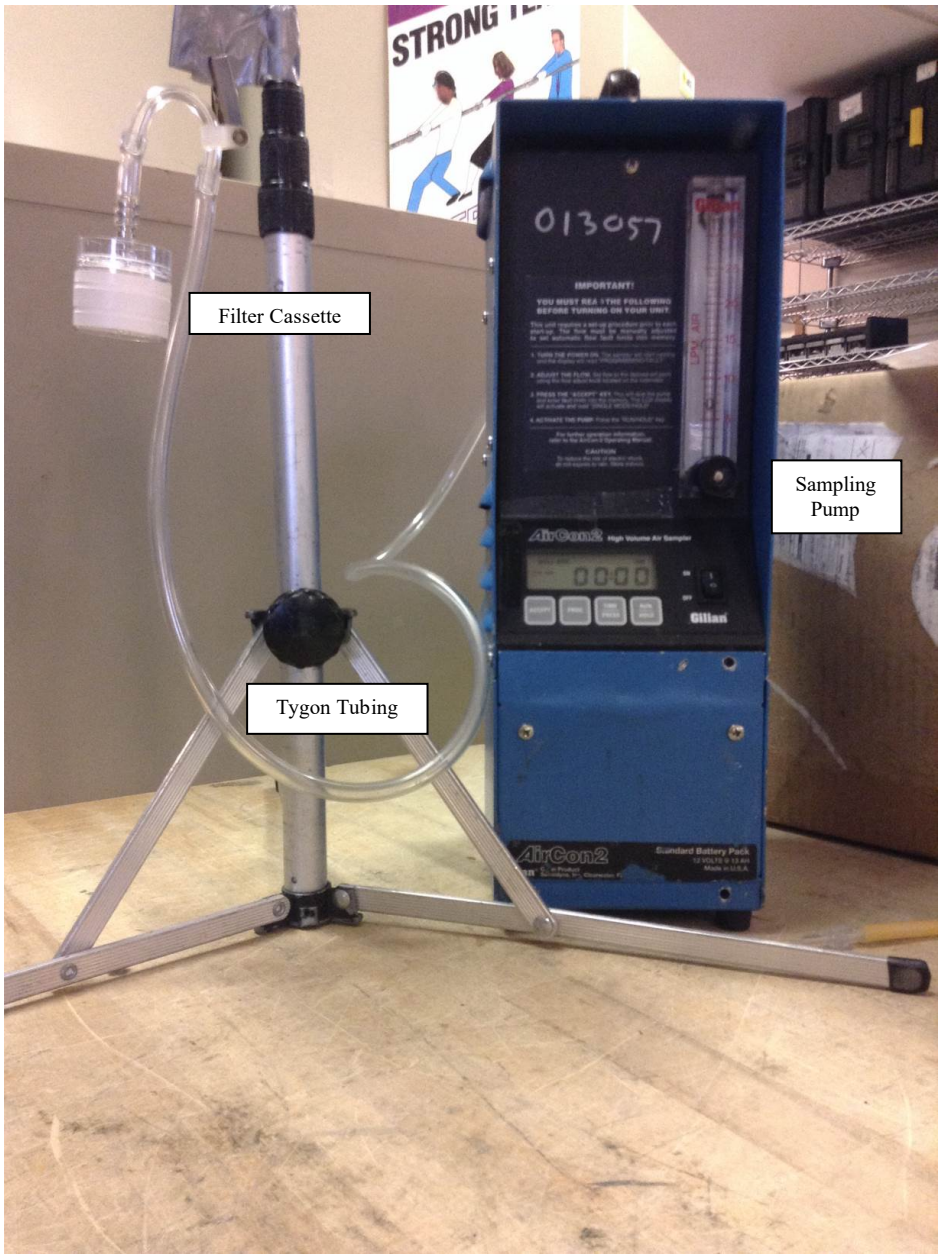


FIGURE 5. Sampling Train for an Area Sampling Pump



APPENDIX C
Attachment
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AIR SAMPLING FOR METALS

[Based on NIOSH Method 7303, Elements by ICP (Hot Block/HCl/HNO_3 Digestion)]

Air Sampling Worksheet

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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					