STANDARD OPERATING PROCEDURE

Eleven

General Air Sampling Guidelines

Modified from

U.S. Environmental Protection Agency Environmental Response Team SOP's 2102, 2103, and 2104

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1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to define the use of several air sampling techniques that could be used for indoor air monitoring/remediation system monitoring (i.e. soil vapor extraction systems) of petroleum constituents. The sampling methods that will be discussed include Tedlar bag sampling, charcoal tube sampling, and Tenax/Carbon Molecular Sieve (CMS) tube sampling.

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site situations, equipment limitations, or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute South Dakota Department of Environment and Natural Resources (Department) endorsement or recommendation for use.

2.0 METHOD SUMMARY

2.1 Charcoal Tube Sampling

Charcoal tube sampling is performed by drawing a known volume of air through a charcoal adsorption tube. As air is drawn through the tube during sampling, gases and vapor adsorb onto the surface of the charcoal. After sampling, the tubes are delivered to the laboratory for analysis.

2.2 Tenax/CMS Tube Sampling

Tenax/CMS tube sampling is performed by drawing a known volume of air through a Tenax adsorbent followed by a CMS adsorbent. Volatile organic compounds are captured on the adsorbent while major inorganic atmospheric constituents pass through or are partially retained. After sampling, the tubes are delivered to the laboratory for analysis.

2.3 Tedlar Bag Sampling

Tedlar bag sampling allows for the collection of a representative grab sample of a gaseous media for analysis. The Tedlar bag collection system allows for this and consists of the following items:

- The Tedlar bag complete with necessary fittings
- A box in which a vacuum is created
- A sampling pump to create the necessary vacuum
- An appropriate Teflon and Tygon tubing

The Tedlar bag is placed into a vacuum box (See Figure 1 in Appendix) and the fitting is inserted into the Teflon tubing. The Teflon tubing is the path through which the gaseous media will travel. The pump is attached to the Tygon tubing, which is part of the vacuum fitting on the vacuum box. The pump evacuates the air in the vacuum box, creating a pressure differential causing the sample to be drawn into the bag. The sample drawn into

the Tedlar bag never flows through the pump. The flow rate for the pump must be defined prior to sampling. The usual flow rate for bag sampling is 3 liters/minute (l/min).

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Charcoal Tube Sampling

Charcoal used for sampling is housed in a glass tube that has been flame sealed. The most commonly used charcoal tubes contain 150 milligrams (mg) or 600 mg of carbon. The smaller 150-mg tube is 7 centimeters (cm) long with a 4 millimeter (mm) inside diameter (ID) and a 6 mm outside diameter (OD) containing two sections of 20/40 mesh activated carbon separated by urethane foam. The adsorbing section contains 100-mg of charcoal, with the backup section contains 50-mg of charcoal. The larger 600-mg tube is 11-cm long with a 6-mm ID and 8-mm OD containing two sections of 20/40 mesh activated charcoal separated by urethane foam. The adsorbing section contains 400-mg of charcoal, with the backup section containing 200-mg of charcoal. A greater volume of air can be drawn through the larger tube thereby providing a greater sensitivity.

To preserve and store samples:

- Place plastic caps on the charcoal tube ends.
- Place the sample in a whirl bag. If collocated samples have been collected, place both tubes in one whirl bag.
- If the sample tube must be stored for more than a week, refrigeration is recommended. **Maximum recommended holding time is two weeks.**

3.2 Tenax/CMS Tube Sampling

Tenax/CMS tubes contain a granular inert chemical compound with adsorbent properties. A flame-sealed outer glass tube protects the inner tube from contamination. This outer glass tube must be broken and the Tenax/CMS tube must be removed prior to sampling. The Tenax/CMS tube has a 6 mm OD and a 4 mm ID containing one section of 150-mg Tenax, 35/60 mesh and one section of 150-mg CMS 60/80 mesh.

After sampling is complete, the Tenax/CMS tubes are stored in culture tubes. Therefore, prior to site work, the culture tubes should be cleaned and prepared using the following procedure:

- A plug of pre-cleaned glass wool (methanol rinsed, baked in an oven at 120°C) is placed in the bottom of each tube.
- The culture tubes are placed in an oven for at least two hours at 120⁰ C. The Teflon lined caps are not baked.
- The culture tubes are removed from the oven and allowed to cool.
- The culture tubes are placed in a resealable bag or whirl bag.

Refrigerate the samples and keep out of sunlight. **Storage for more than four weeks is not recommended.**

3.3 Tedlar Bag Sampling

The Tedlar bags most commonly used for sampling have a 1-liter volume. When the sampling procedure is concluded, the Tedlar bags are stored in either a clean cooler or a trash bag to prevent photodegradation. Note: It is essential that sample analysis be conducted within 48 hours, after this time compounds may escape or become altered.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 Charcoal Tube Sampling

Low sampling flow rates, and high temperature and humidity may cause a decrease in the adsorption capacity of activated carbon. Contaminants from the front portion of the tube may migrate to the back portion of the tube. Refrigeration may minimize this migration.

4.2 Tenax/CMS Tube Sampling

Contamination of the Tenax/CMS tubes with the compound(s) of interest is a common problem. To minimize this problem, the user must be extremely careful in the preparation, storage, and handling of the tube throughout the sampling analysis process. To avoid contamination from skin oils, a lint free glove must be used when handling Tenax/CMS tubes.

4.3 Tedlar Bag Sampling

Contamination is a major concern since many of the compounds in question will be present in the parts per billion range. In order to minimize the risk of cross contamination, the following factors should be considered:

- Proximity of the bags to source(s) of potential contamination during transportation and storage. The farther away from source(s) the bags are the less likely the chances of external contamination.
- Bags must be attached only to clean Teflon tubing.
- Once the bag has been collected, affix the sample label to the edge of the bag. Adhesives found in the label may permeate the bag if placed on the body of the bag. Fill out labels with a ballpoint pen as permanent markers contain volatile compounds that may contaminate the sample.
- Due to the chemical structure of Tedlar, highly polar compounds will adhere to the inner surface of the bag. Also, low molecular weight compounds may permeate the bag. Real-time monitors such as the organic vapor analyzer, photoionization detector, and combustible gas indicator should be used as screening devices prior to sampling. The information gathered is written on the sample label to inform the individuals performing the sample analysis.

The Tedlar bag sampling system is straightforward and easy to use. However, there are several things to be aware of when sampling.

• The seal between the top and bottom half of the vacuum box must be air tight in order for the system to work.

- Check the O-ring gasket to see if it is in place with the proper fit. O-rings that have been stretched out will not remain in place, thus requiring constant realignment.
- Check that all the fittings associated with the vacuum joints are securely in place. The fittings can be pushed loose when inserting the valve stem into the Teflon tubing.
- Occasionally, a corner of the Tedlar bag will stick out between the two halves of the
 vacuum box causing a poor seal. Since the bags will hold only a given volume, overinflation will cause the bags to burst.

5.0 EQUIPMENT

5.1 Charcoal Tube Sampling

Equipment List:

- Personal sampling pump
- Dowel rods
- Single or dual rotameter (with stand and desired pre-calibrated flow rate)
- Charcoal tubes (150 or 600 mg)
- 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
- Tube holder end (to support and seal the sampling tube within the plastic housing)
- Glass tube cracker
- Resealable bags
- Whirl bags
- Plastic end caps
- Air sampling worksheets and sample labels
- Chain of custody records
- Screwdriver set

5.2 Tenax/CMS Tube Sampling

Equipment List:

- Calibrated personal sampling pump
- Dual rotameter with stand and pre-calibrated flow rate
- Tenax/CMS tubes, preferably of the same lot number
- Flexible Tygon tubing (for attaching the tube holder system to the suction side of the pump)
- Universal tube holder system which includes, dual variable manifold flow controller, tube holder end with rubber boot adapter, sleeves, and clear plastic housings
- Glass cracker
- Lint-free gloves
- Glass wool
- Teflon tape
- Culture tubes
- Screwdrivers
- Resealable bags

- Whirl bags
- Air sampling worksheets
- Chain of custody records

5.3 Tedlar Bag Sampling

The following items must be operational to perform Tedlar bag sampling:

- Vacuum box must be clean, Teflon tubing replaced, and equipped with extra O-rings
- Pump(s) must be charged, in good working order, and set with the appropriate flow rate of 3 liters per minute
- Tedlar bags must be free of visible contamination and preferably new
- Chain of custody records, custody seals
- Sample labels
- Air sampling worksheets
- Opaque trash bags

6.0 REAGENTS

6.1 Charcoal Tube Sampling

This section is not applicable to charcoal tube sampling.

6.2 Tenax/CMS Tube Sampling

Methanol is used in the laboratory to clean the culture tubes, which hold the Tenax/CMS samples. This is performed prior to site work.

6.3 Tedlar Bag Sampling

This section is not applicable to Tedlar bag sampling.

7.0 PROCEDURES

7.1 Charcoal Tube Sampling

7.1.1 Preparation

- Determine the extent of the sampling effort, the sampling methods to be employed and the types and amounts of equipment and supplies needed.
- Obtain and organize the necessary sampling and monitoring equipment.
- Decontaminate or pre-clean equipment, and ensure that it is in working order. Pre-calibrate sampling pumps as described in Section 7.1.2.
- Prepare scheduling and coordinate with staff, client, and the department, if appropriate.
- Perform a general site survey prior to entry, in accordance with the site specific Health and Safety Plan.

7.1.2 Calibration Procedures

To save time in the field, sampling pumps can be pre-calibrated in the office prior to arriving on-site. The calibration must be checked in the field prior to, and upon completion of sampling.

- Assemble the calibration train as shown in Figure 2 of Appendix, using a rotameter, sampling pump, manifold (only if the desired flow rate is below 750 cc/min), a tube holder system (sleeve and tube end holder), and a calibration tube. The calibration tube is a representative tube from the same lot of tubes that is used for sampling.
- 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 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.
- Affix a sticker to the manifold (if used) and pump indicating the pre-calibrated flow rate and sampling media.
- 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.

7.1.3 Field Operation

- Mobilize to a clean zone and calibrate the appropriate number of sampling pumps as described in Section 7.1.2. If the pumps were pre-calibrated, the calibration should be checked in the same manner. Fine-tuning of the flow may be required.
- Mobilize to the sampling location.
- Crack the charcoal tube ends using a glass tube cracker.
- Screw the tip onto the sleeve so the charcoal tube is held in place.
- Attach the sleeve(s) to a single or double manifold. At higher flow rates (>750 cc/min), charcoal tubes can run straight without a manifold.
- To set up the sampling train, attach one end of the Tygon tubing (approximately 2 feet) to the tip of the sleeve or manifold. Attach the other end of the tubing to the inlet plug on the pump, Figure 3 in Appendix. Refer to Figures 4 and 5 in the appendix, respectively, for illustrations of sampling trains without a manifold and with a dual manifold (for collocated samples).
- Adjust time on the pump to the required sample time.
- Place the charcoal tube in a position free from obstruction on a dowel rod or stand.
- Record weather data (e.g., ambient temperature, barometric pressure, and relative humidity) on the air sampling worksheet or in the logbook.
- Turn on the pump.
- After the pump has run the full time, check the fault button to determine if the pump ran for the scheduled time.
- Verify calibration by connecting a rotameter with Tygon tubing and turning on the pump. Record the final flow rate on the air sampling worksheet.

7.1.4 Post Operation Procedures

- Record the sampling time on the air sampling worksheet.
- Remove the charcoal tube from the sleeve.
- Cap charcoal tubes with plastic caps immediately after sampling. Never use rubber caps.
- Place the sample in a whirl bag labeled with sample ID No., total volume, and required analysis. If collocated samples were collected, place each tube in a separate whirl bag and assign a unique sample ID No. to each tube.
- Indicate all applicable information on the air sampling worksheet (e.g., sample volume, ID No., location, date, and weather parameters).
- If the sample tube must be stored for more than a week, refrigeration is recommended.
- Prepare samples, including QC samples, for transport by packing them in a shipping container with bubble wrap or styrofoam. Complete a chain of custody record in accordance with the appropriate chain of custody procedures.

The following methods are typically used for the analysis of charcoal tubes: NIOSH Methods 1501-aromatic hydrocarbons; 1500-hydrocarbons boiling point 36⁰ to 126⁰ C; and 103-halogenated hydrocarbons. If other analytical parameters are required, the appropriate methodology should be determined prior to field activities.

7.2 Tenax/CMS Tube Sampling

7.2.1 Preparation

- Determine the extent of the sampling effort, the sampling methods to be employed and the types and amounts of equipment and supplies needed.
- Obtain and organize the necessary sampling and monitoring equipment.
- Decontaminate or pre-clean equipment, and ensure that it is in working order. Pre-calibrate sampling pumps as described in Section 7.1.2.
- Prepare scheduling and coordinate with staff, client, and the department, if appropriate.
- Perform a general site survey prior to entry, in accordance with the site specific Health and Safety Plan.

7.2.2 Calibration Procedures

Assemble the calibration train as shown in Figure 6, in Appendix, using a dual rotameter, sampling pump, dual manifold, and representative Tenax/CMS tubes. The same lot number of Tenax/CMS tubes is used for both sampling and calibration.

• Adjust the sampling pump to the low flow mode.

- Remove the end caps on the flow controller manifold. To adjust flow, turn the needle valve with a small screwdriver (counter-clockwise to increase, clockwise to decrease).
- Turn the flow adjustment screw on each manifold until the float ball on the rotameter is lined up with the pre-calibrated flow rate value. A sticker on the rotameter should indicate this value.
- Affix a sticker to the manifold and pump indicating the calibrated flow rate and media.
- Remove the representative Tenax/CMS tubes from the sleeves.

The pump and manifold (including boots) are calibrated as a unit and should not be separated until the samples have been collected. See Table 1 below for recommended flow rates and sample volumes.

TABLE 1. RECOMMENDED FLOW RATES AND SAMPLE VOLUMES Flow Rates		
Maximum	50 cc/min	
Optimal	30-40 cc/min	
Minimum	10 cc/min	
Volumes		
Maximum	5 liters	
Optimal	2 liters	
Minimum	0.5 liters	

7.2.3 Field Operation

- Calibrate the appropriate number of pumps with manifolds as described in Section 7.2.2. If the pumps were pre-calibrated, the calibration should be checked in the same manner. Fine-tuning of the flow may be required.
- Crack the outer glass tubes using a glass cracker.
- Use clean, lint-free gloves or cloth to remove the Tenax/CMS tubes from the outer glass housings.
- Insert the Tenax/CMS tubes into a boot, with the CMS sections closet to the manifold.
- Attach protective sleeves over the tubes. Do not enclose the Tenax/CMS tube ends
- Set up the sampling train, by attaching one end of the Tygon tubing (approximately two feet) to the manifold; and the other end to the inlet plug on the pump (Figure 7 in Appendix).
- Place the sampling tubes in the breathing zone. The pump and tubes can be placed on any solid stationary surface.
- Place the tubes in either a vertical or horizontal position.
- Adjust the pump time and turn it on.
- Record weather data (e.g., ambient temperature, barometric pressure, and relative humidity) on the air sampling worksheet or in the logbook.

• Check the pump at the midpoint of the sampling period if longer than four hours.

7.2.4 Post Operational Procedures

- At the end of the sampling period, record the run in a logbook or on a worksheet.
- Check the flow rate by attaching a rotameter with Tygon tubing and turning on the pump.
- Remove the Tenax/CMS tubes from the sleeves using a lint-free cloth or gloves.
- Place the Tenax/CMS tubes in a culture tube. Pack with glass wool.
 Tenax/CMS tubes from the same manifold and identical flow rates must be placed in the same culture tube.
- Place a sample sticker indicating sample ID No. on the culture tube. Do not put a sample sticker on the Tenax/CMS tubes itself, as this will contaminate the tube.
- Attach the culture tube lid and wrap the lid/tube interface with Teflon tape.
- Place the culture tubes into a resealable bag or a whirl bag.
- Keep the samples refrigerated and out of the sunlight. **Storage for more than four weeks is not recommended.**
- Prepare the samples, including QC samples, for transport by packing them in shipping containers with bubble wrap or styrofoam. Complete a chain of custody record in accordance with the appropriate chain of custody procedures.

7.3 Tedlar Bag Sampling

7.3.1 Preparation

- Determine the extent of the sampling effort, the sampling methods to be employed and the types and amounts of equipment and supplies needed.
- Obtain and organize the necessary sampling and monitoring equipment.
- Decontaminate or pre-clean equipment, and ensure that it is in working order. Pre-calibrate sampling pumps as described in Section 7.1.2.
- Prepare scheduling and coordinate with staff, client, and the department, if appropriate.
- Perform a general site survey prior to entry, in accordance with the site specific Health and Safety Plan.

7.3.2 Field Operation

Tedlar bags are stored in boxes of ten. The valve is in the open position when stored. Occasionally, a piece of debris will clog the valve, making it necessary to close the valve stem to clear the debris. Pulling it out closes the valve stem. If the valve stem is difficult to pull, it helps to spin the valve stem while pulling it.

• Remove the Tedlar bag from the carton.

- Insert the valve stem into the Teflon tube which runs through the vacuum box (See Figure 1 of Appendix).
- Place the Tedlar bag in the vacuum box. Seal the vacuum box by applying pressure to the top and bottom (ensure that the O-ring is in place and unobstructed).
- Connect the sampling pump to the evacuation tube.
- Connect the intake tube to the desired source or place the intake tube into the media of concern.
- Turn on the sampling pump.
- Allow the bag to fill (visual observation and sound of laboring pump).
- Turn off the sampling pump and remove the evacuation tube from the pump.
- Remove bag and pull the valve stem out.
- Lock the valve stem.
- Label the bag using either a tag or sticker placed on the edge of the bag. Do not write on the bag itself.
- Place Tedlar bag in a clean cooler or opaque trash bag to prevent photodegradation.

7.3.3 Post Operation

- Once the samples are collected, transfer them to the laboratory for analysis. It is essential that sample analysis be conducted within <u>48 hours</u>, after this time compounds may escape or become degraded.
- When transferring Tedlar bags, a chain of custody form must accompany the samples. Note: Personnel should be aware that some of the chemicals of concern will degrade within a few hours of sampling. A study conducted by Posner and Woodfin (1986) showed that over a 4 to 6 hour period benzene losses approached 5%.
- For the time prior to analysis, samples may be stored in a clean cooler or opaque trash bag with a trip blank (a Tedlar bag filled with "zero air") and the chain of custody form.

8.0 CALCULATIONS

8.1 Charcoal Tube Sampling & Tenax/CMS Tube Sampling

The total volume of a sample is calculated by multiplying the total sampling time by the flow rate. The total volume for each sample should be indicated on the chain of custody form.

8.2 Tedlar Bag Sampling

This section is not applicable to Tedlar bag sampling.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

9.1 Charcoal Tube Sampling

The following general QA procedures apply:

- All data must be documented on a worksheet or in a logbook.
- All instruments must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan.
 Equipment checkout and calibration activities must occur prior to sampling and operation and they must be documented.

The following specific QC activities apply:

- Provide one field blank per sampling event or per 20 samples which ever is greater. The field blank should be handled in the same manner as the sampling tube (break, seal, and transport) except that no air is drawn through the tube.
- 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.)
- Include a minimum of one lot blank tube per manufacture's lot of tubes used per sampling event. These tubes are taken directly from the charcoal tube box. Do not break the ends.

9.2 Tenax /CMS Tube Sampling

The following general QA procedures apply:

- All data must be documented on a worksheet or in a logbook.
- All instruments must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling and operation and they must be documented.

The following specific QC activities apply:

- Provide one field blank per sampling event or per 20 samples which ever is greater. The field blank should be handled in the same manner as the sampling tube (break, seal, and transport) except that no air is drawn through the tube.
- Provide a minimum of one lot blank tube per sampling episode. These tubes are taken directly from the Tenax/CMS tube box. Do not break the outer glass housing. Place in a resealable bag and keep with other samples
- All sample stations should have duplicated sample tubes.

9.3 Tedlar Bag Sampling

The following general QA procedures apply:

- All data must be documented on a worksheet or in a logbook.
- All instruments must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. Equipment checkout and calibration activities must occur prior to sampling and operation and they must be documented.

Depending upon the quality assurance work plan requirements, a background sample consisting of upgradient/downgradient, beginning/ending of day or a combination, may be collected. It may also be desirable to change sample train tubing between locations.

Tedlar bag standards must be filled on-site to identify the contaminants' degradation from the time the sample is collected until its analysis. Trip blanks, Tedlar bags filled with "zero air", must accompany sample bags at a minimum rate of one per day to identify possible contamination during handling. For each lot of Tedlar bags, a minimum of one bag must be filled with "zero air" and then analyzed for the parameter(s) of interest to detect contamination due to the Tedlar bag which may itself cause false positives. Duplicate Tedlar bags should be collected at a minimum rate of 5% of the total number of samples per sampling event.

10.0 DATA VALIDATION

Results of the quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results according to the project's data quality objectives.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures.

12.0 SUMMARY

This SOP discusses three types of sampling methods that are acceptable for the collection of indoor air/remediation system monitoring (i.e. soil vapor extraction systems) that may contain petroleum constituents. The collection of indoor air samples is not limited to these three methods. However, the department **must** be contacted prior to using an alternative air sampling method.

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STANDARD OPERATING PROCEDURE

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Appendix of Figures

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APPENDIX A

FIGURE 1 - Tedlar Bag Sampling Apparatus

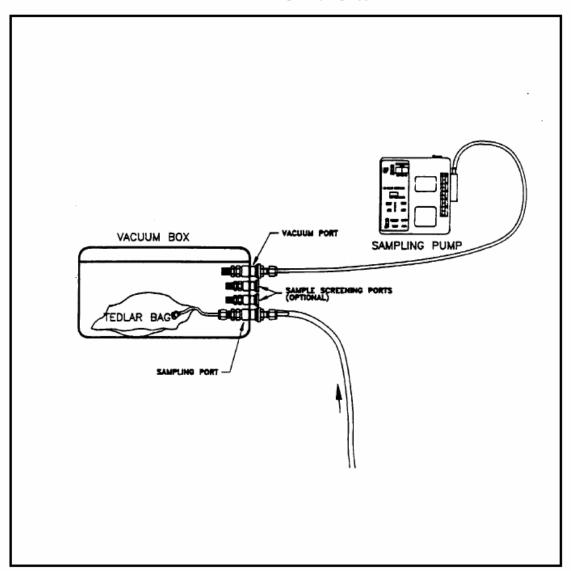


Figure 1. Tedlar Bag Sampling Apparatus

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FIGURE 1. Calibration Train

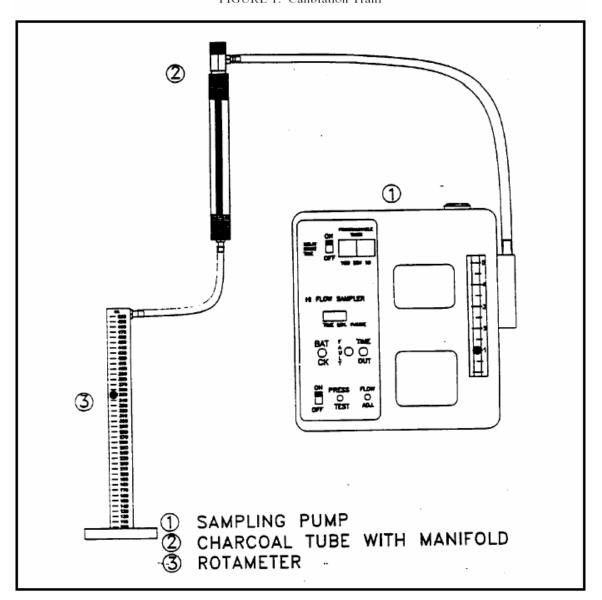


Figure 2. Calibration Train

APPENDIX A (Cont'd)

FIGURE 2. Charcoal Tube Sampling Train (Single Manifold)

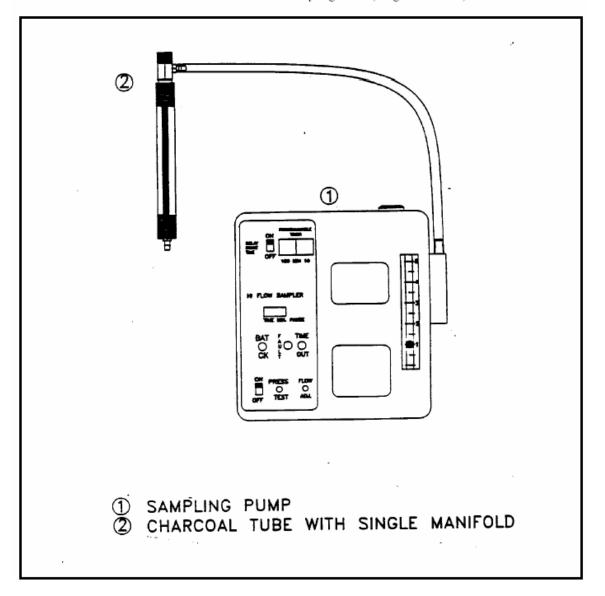


Figure 3. Charcoal Tube Sampling Train (Single Manifold)

APPENDIX A (Cont'd)

FIGURE 3. Charcoal Tube Sampling Train (No Manifold)

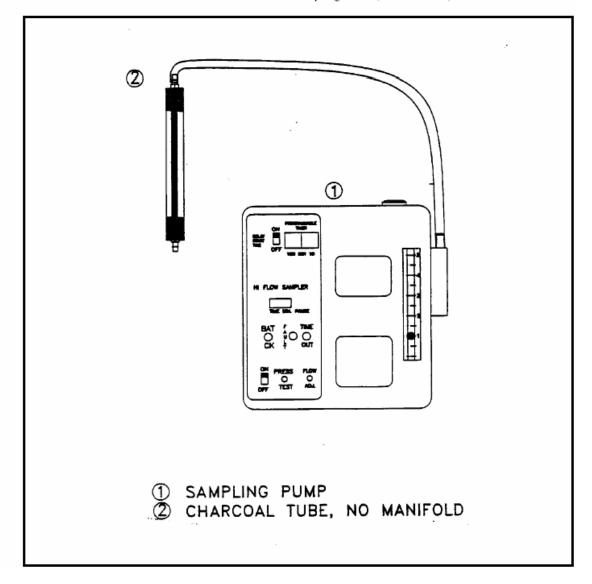


Figure 4. Charcoal Tube Sampling Train (No Manifold)

APPENDIX A (Cont'd)

FIGURE 4. Charcoal Tube Sampling Train (Dual Manifold for Collocated Sampling)

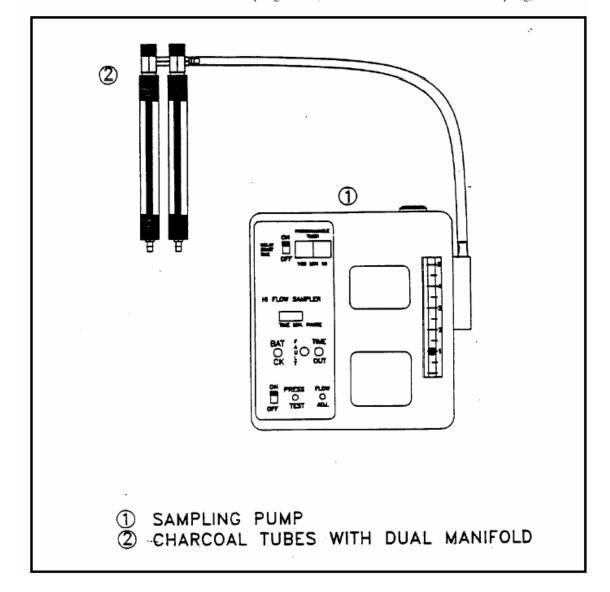


Figure 5. Charcoal Tube Sampling Train (Dual Manifold for Collocated Sampling)

APPENDIX B

FIGURE 1. Tenax/CMS Calibration Train

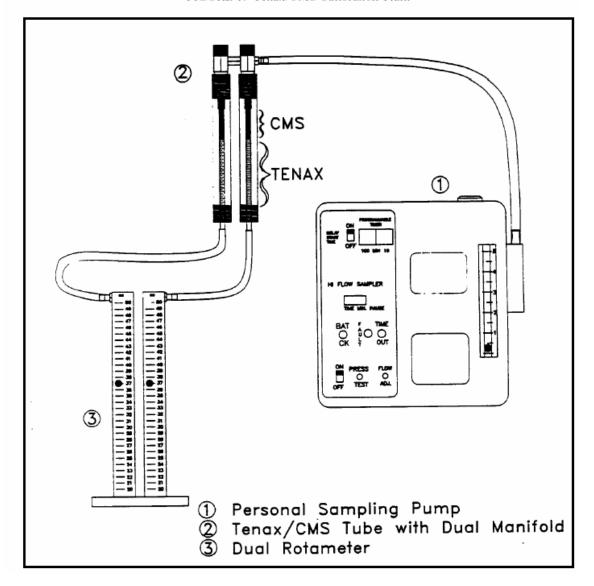


Figure 6. Tenax/CMS Calibration Train

APPENDIX B (Cont'd)

Figures

FIGURE 2. Tenax/CMS Sampling Train

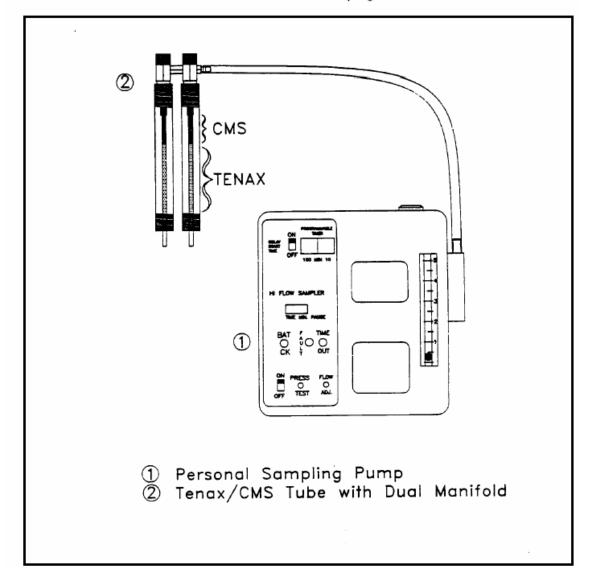


Figure 7. Tenax/CMS Sampling Train

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