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U. S. Environmental Protection Agency
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Athens, Georgia

OPERATING PROCEDURE

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1 GENERAL INFORMATION

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting ambient air samples for field screening or laboratory analysis. Air toxics, as used in this procedure, means air pollutants for which National Ambient Air Quality Standards have not been established. Criteria pollutants mean air pollutants for which National Ambient Air Quality Standards have been established.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling ambient air samples in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain an ambient air sample, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Quality Control

Quality control for sampling associated with ambient air monitoring is method specific. The sampling methods described in the sections that follow contain the quality control procedures appropriate for each method.

1.5 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-002. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005.

1.6 General Precautions

1.6.1 Safety

Proper safety precautions must be observed when collecting ambient air monitoring samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.6.2 Procedural Precautions

The following precautions should be considered when collecting ambient air monitoring samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.

- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.

- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.

- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.

- Documentation of field sampling is done in a bound logbook.

- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.

- All shipping documents, such as bills of lading, etc., shall be retained by the project leader and stored in a secure place.

1.7 References

Ambient Monitoring Technology Information Center (AMTIC) Internet Bulletin Board: $PM_{2.5}$ QA area (http://www.epa.gov/ttn/amtic/pmqa.html)

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Method TO-4A (pesticides and PCBs) and TO-13A (polynuclear aromatic compounds)

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Method TO-9A

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Method TO-11A

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Method TO-15A

International Air Transport Authority (IATA) Dangerous Goods Regulations, Most Recent Version

Neutronics Model RI-2002PA Portable Automotive Refrigerant Identifier Operation Instruction Manual

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

Title 40 Code of Federal Regulations (CFR), Part 50, Appendix A - Reference Method for the Determination of Sulfur Dioxide in the Atmosphere (Pararosaniline Method), Most Recent Version

Title 40 CFR, Part 50, Appendix B - Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High Volume Method), Most Recent Version

Title 40 CFR, Part 50, Appendix C - Measurement Principle and Calibration Procedure for the Measurement of Carbon Monoxide in the Atmosphere (Non-Dispersive Infrared Photometry), Most Recent Version.

Title 40 CFR, Part 50, Appendix D-Measurement Principle and Calibration Procedure for the Measurement of Ozone in the Atmosphere, Most Recent Version

Title 40 CFR, Part 50, Appendix F - Measurement Principle and Calibration Procedure for the Measurement of Nitrogen Dioxide in the Atmosphere (Gas Phase Chemiluminescence), Most Recent Version

Title 40 CFR, Part 50, Appendix G - Reference Method for the Determination of Lead in Suspended Particulate Matter Collected From Ambient Air, Most Recent Version

Title 40 CFR Part 50, Appendix J - Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere, Most Recent Version

Title 40 CFR Part 50, Appendix L - Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere, Most Recent Version

Title 40CFR Part 53 – Ambient Air Monitoring Reference and Equivalent Methods, Most Recent Version

Title 40 CFR, Part 58 – Ambient Air Quality Surveillance, Most Recent Version

Title 40 CFR, Part 58, Appendix A -Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS), Most Recent Version

Title 40 CFR, Part 58, Appendix C - Ambient Air Quality Monitoring Methodology, Most Recent Version

Title 40 CFR, Part 58, Appendix D - Network Design for State and Local Air Monitoring Stations (SLAMS), National Air Monitoring Stations (NAMS), and Photochemical Assessment Monitoring Stations (PAMS), Most Recent Version

Title 40 CFR, Part 58, Appendix E - Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring, Most Recent Version

Title 49 CFR, Parts 171 to 179, Most Recent Version

US Environmental Protection Agency (US EPA) 1994. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I, Principles, EPA/600/R-94/038a, April1994.

US EPA. 1995. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV: Meteorological Measurements, (revised edition) EPA/600/R-94/038d, March, 1995.

US EPA. 1998. Quality Assurance Handbook for Air Pollution Measurement System, Volume II: Part 1, Ambient Air Quality Monitoring Program, Quality System Development, EPA-454/R-98-004, August 1998 (commonly referred to as the "Red Book.").

US EPA. 1998. EPA Quality Assurance Document: Method Compendium, $PM_{2.5}$ Mass Weighing Laboratory Standard Operating Procedures for the Performance Evaluation Program, United States Environmental Protection Agency Office of Air Quality Planning and Standards, October 1998.

US EPA. 2006. Standard Operating Procedure for Through-the-Probe Performance Evaluations of Ambient Air Quality Monitoring of Criteria Air Pollutants, January 2006.

US EPA. 1998. Quality Assurance handbook for Air Pollution Measurement Systems, Volume II, Ambient Air Specific Methods, Section 2.12, EPA/600/R-94/038b, November, 1998

US EPA. 2002. EPA Quality Assurance Guidance Document: Method Compendium, Field Standard Operating Procedures for the $PM_{2.5}$ Performance Evaluation Program, United States Environmental Protection Agency Office of Air Quality Planning and Standards, Revision No. 2, March 2002.

US EPA. 2005. Standard Operating Procedures for Through-The-Probe (TTP) Performance Evaluations of Ambient Air Quality Monitoring of Criteria Air Pollutants, Office of Air Quality Planning and Standards, May 2005.

US EPA Quality Assurance Document: Quality Assurance Project Plan for the Performance Evaluation Program

US EPA Implementation Plan: $PM_{2.5}$ Federal Reference Method Performance Evaluation Program, EPA Quality Assurance Guidance Document 2.12

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

2 AUDITING OF OZONE AND CARBON MONOXIDE MONITORING SITES IN THE NATIONAL PERFORMANCE AUDIT PROGRAM (NPAP)

2.1 Auditing Ozone Analyzers

The following is a synopsis of procedures which should be strictly adhered to for the auditing of ozone at ambient monitoring sites.

- The photometer (audit instrument) will meet 40 CFR Part 50, Appendix D specifications for a Primary Standard Calibration Photometer. This instrument will be re-plumbed to act as an analyzer to measure test gas concentrations.

- The photometer will be verified quarterly against a National Institute of Standards and Technology (NIST) Standard Reference Photometer before use.

- The mobile laboratory will meet the specifications of monitor reference/ equivalent designation for temperature control which is between 20-30 degrees Celsius when the analyzer is in operation.

- The probe line must meet the requirements stated in 40 CFR Part 58 for materials and sample residence time.

- Flows will be calibrated annually, and all flow calibrations will be traceable to a primary standard. Flows will be corrected to EPA standard temperature and pressure (25^oC and 760 mm Hg).

- The ozone generator/calibrators flow and ozone generation output will be calibrated annually.

- The ozone generator and photometer will operate using zero air supplied by a zero air generator. The zero air scrubber components will be replaced annually or if anomalies in the zero measurement are observed. The desiccant will be regenerated when two thirds (2/3) of the blue indicating silica gel has turned pink.

- During the audit, stability of each audit point will be demonstrated for at least five minutes.

- All auditing will be performed at approximately ambient pressure (utilizing a vent for excess during the audit procedure). There should be excess air flow at the probe at all times.

- All audits will be conducted "through the probe" where the probe is safely accessible.

- Chain-of-custody must be maintained at all times (monitor will be locked in the mobile laboratory when the operator is not present to prevent tampering).

2.1.1 Audit Procedure

Auditing will be conducted using the procedures as summarized below, and in accordance with the EPA Standard Operating Procedure for Through-the-Probe Performance Evaluations of Ambient Air Quality Monitoring of Criteria Air Pollutants, and 40 CFR Part 50, APPENDIX A:

- Procedures detailed in the approved instrument manual will be used for installation, calibration, QA checks, maintenance, and repairs.

- The photometer will be verified quarterly against a National Institute of Standards and Technology (NIST) Standard Reference Photometer after major maintenance or when a quality assurance (QA) check shows an out-of-control condition exists.

- A data telemetry system, with electronic strip charting capability will be operated to record the audit progress and each audit activity will be annotated on the chart. This will serve as a permanent record and diagnostic tool.

- Prior to the audit, the audit equipment, including the photometer and calibrator, will be allowed a warm up period of at least 3 hours. During this time, the system should be conditioned with at least 200 ppb of ozone. The concentration of ozone used to condition the system must always be higher than the highest audit point that will be audited.

- The audit will consist of at least three consecutive levels where at least 80% of ozone measurements are expected to be at the monitoring site and a zero measurement. The prescribed audit levels are recorded in 40 CFR Part 58, Appendix A, 3.2.2.1.

- Audit data from the mobile laboratory and monitoring site will be recorded electronically on the NPAP Audit Worksheet. This worksheet, along with the electronic strip chart trace will be printed at the conclusion of each audit event. In addition, the NPAP Audit Worksheet will be copied and placed on the network drive for archival. A hard copy of the preliminary audit report and electronic strip chart will be provided to the site operator at the conclusion of the audit.

- After completion of the study, the mobile laboratory will be returned for inspection, maintenance, and repair. All field documentation will be retained by the Air Monitoring Staff.

2.2 Auditing Carbon Monoxide Analyzers

The following is a synopsis of procedures which should be strictly adhered to for the auditing of carbon monoxide in air.

- Calibration systems will meet 40 CFR Part 50, Appendix C specifications.

- The mobile laboratory will meet the specifications of monitor reference/ equivalent designation for temperature control which is between 20-30 degrees Celsius when the analyzer is in operation.

- The audit analyzer will be calibrated using certified concentrations of standard gases before and after use.

- The probe line must meet the requirements stated in 40 CFR Part 58 for materials and sample residence time.

- Calibrator flows will be calibrated annually, and all flow calibrations will be traceable to a primary standard. Flows will be corrected to EPA standard temperature and pressure (25^oC and 760 mm Hg).

- The gas calibrator will operate using zero air supplied by a zero air generator. The zero air scrubber components will be replaced annually or if anomalies in the zero measurement are observed. The desiccant will be regenerated when two thirds (2/3) of the blue indicating silica gel has turned pink.

- During the audit, stability of each audit point must be demonstrated for at least five minutes.

- All auditing will be performed at approximately ambient pressure (utilizing a vent for excess during the audit procedure). There should be excess air flow at the probe at all times.

- All audits will be conducted “through the probe” where the probe is safely accessible.

- Chain-of-custody must be maintained at all times (monitor will be located in a locked enclosure when the operator is not present to prevent tampering).

2.2.1 Audit Procedure

Auditing will be conducted using the procedures summarized below, and in accordance with the EPA Standard Operating Procedure for Through-the-Probe Performance Evaluations of Ambient Air Quality Monitoring of Criteria Air Pollutants, and 40 CFR Part 50, APPENDIX C:

- Procedures detailed in the approved instrument manual will be used for installation, calibration, QA checks, maintenance, and repairs.

- The carbon monoxide analyzer will be verified using G1 or G2 certified tank gases before and after each audit or when a quality assurance (QA) check shows an out-of-control condition exists.

- A data telemetry system, with electronic strip charting capability will be operated to record the audit progress and each audit activity will be annotated on the chart. This will serve as a permanent record and diagnostic tool.

- Prior to the audit, the audit equipment, including the carbon monoxide analyzer and calibrator, will be allowed a warm up period of at least 6 hours. During this time, the system should be conditioned with approximately 200 ppb of ozone.

- The audit will consist of at least three consecutive levels where at least 80% of carbon monoxide measurements are expected to be at the monitoring site and a zero measurement. The prescribed audit levels are recorded in 40 CFR Part 58, Appendix A, 3.2.2.1.

- Audit data from the mobile laboratory and monitoring site will be recorded electronically on the NPAP Audit Worksheet. This worksheet, along with the electronic strip chart trace will be printed at the conclusion of each audit event. In addition, the NPAP Audit Worksheet will be copied and placed on the network drive for archival. A hard copy of the preliminary audit report and electronic strip chart will be provided to the site operator at the conclusion of the audit.

- After completion of the study, the mobile laboratory and equipment will be returned for inspection, maintenance, and repair. All field documentation will be retained by the Air Monitoring Staff.

2.3 Sampling of Particulate Matter in Ambient Air as $PM_{2.5}$ in the $PM_{2.5}$ Performance Evaluation Program

The following is a synopsis of procedures which should be strictly adhered to for filter based sampling of PM2.5 in air. The procedures below are a summarized version of the procedure described in the September 2006 Revised Quality Assurance Guidance Document, Method Compendium "Field Standard Operating Procedures for the $PM_{2.5}$ Performance Evaluation Program" which should be referenced for all low volume $PM_{2.5}$ sampling:

- Field personnel will take the filters, field data sheets, and COC to the monitoring site and setup the portable samplers.
- Air flow rate, barometric pressure, and two temperature measuring devices will be verified prior to each sampling event. All flow, barometric pressure and temperature standard devices are annually calibrated and traceable to a primary standard.
- An external leak check will be performed.
- The sampler will be programmed to operate and the sampling unit will be locked to prevent tampering.
- Within 8 to 16 hours following the sampling event the exposed filters will be collected, and transported refrigerated (≤ 4°C) to the SESD Filter Weighing Laboratory.
- Field personnel will return the exposed filter, data downloads, field data sheets, and COC to SESD keeping a set of the data records in two separate locations.
- After sample collection, all filters will be stored in petri-slides, boxed, and archived for one (1) year at ≤ 4°C and at least two (2) years at ambient room temperature ≤ 25°C.

2.4 Sampling of Particulate Matter in Ambient Air as PM_{10}

The following is a synopsis of procedures which should be strictly adhered to for the sampling of particulate matter as PM_{10} in air.

- All filters used will be supplied by the EPA National Filter Distribution Program, and of the same quality as supplied to the State and Local Agency Air Monitoring Stations.
- Prior to use, all filters will be checked for pinholes, and desiccated at 15°C - 30°C ± 3°C, and less than 50 ± 5 percent relative humidity, for at least 24 hours.

- Initial and final (exposed) filter weights will be determined by air monitoring personnel. One of ten filters will be re-weighed as a quality assurance check. For batches less than ten, one filter will be re-weighed. Weights of clean filters should be within 2.8mg of original values, and exposed filters should be within 5 mg.

- After sampling, filters will be desiccated as previously described.

- Chain-of-custody must be maintained for all samples (monitor will be locked when the operator is not present to prevent tampering).

2.4.1 Sample Collection Procedure

Particulate samples will be collected using the High Volume sampler as described, and operated in accordance with 40 CFR Part 50, Appendix J, and Red Book Sections 2.10, and 2.11.

- All flow calibration orifices will be traceable to a primary standard Rootsmeter. Flows will be corrected to EPA standard temperature and pressure (25^{o}C and 760 mm Hg).

- Volumetric flow controllers will be used on all PM_{10} samplers. Flows will be determined using a calibrated orifice and a digital manometer.

2.5 Laboratory Filter Analysis of Particulate Matter in Ambient Air as $PM_{2.5}$ in the $PM_{2.5}$ Performance Evaluation Program

The following is a synopsis of procedures which should be strictly adhered to for filter based sampling of $PM_{2.5}$ in air. The procedures below are a summarized version of the procedure described in the Quality Assurance Guidance Document, Method Compendium "$PM_{2.5}$ Mass Weighing Laboratory Standard Operating Procedures for the Performance Evaluation Program" which should be referenced for all low volume $PM_{2.5}$ filter analysis.

- EPA OAQPS National Filter Distribution Program will send filters to the SESD $PM_{2.5}$ Filter Weighing Laboratory

- Upon introduction into the weighing laboratory process, each individual batch of filters will go through lot stability testing to determine a minimum equilibration time period.

- Prior to use the filters will be checked for deformities, equilibrated at 20 - 23°C ± 2°C and 30 - 40 % ± 5 % relative humidity for a minimum of 24 hours, labeled, pre-weighed (auto-entry into database), loaded into cassettes, and prepared for packaging and shipping.

- During the pre-weighing session, the filter batch should pass 100µg and 200µg balance checks (+/- 3ug), two consecutive weightings demonstrating stability (+/- 5 ug), and provide an initial laboratory blank weighing.

- The SESD Filter Weighing Laboratory will ship the pre-weighed filters and chain-of- custody (COC) forms to the field personnel.

- Filters and COC forms for Region 4 are passed directly to the field scientists.

- After sampling, filters will be transported to the SESD Filter Weighing Laboratory, logged into the database, refrigerated until a batch is completed, moved to the laboratory, unloaded from the cassettes, equilibrated for at least 24 hours, and post-weighed. The filter refrigerator temperature will be monitored and recorded on a daily basis.

- During the post-weighing session, the filter batch should pass 100mg and 200mg balance checks (+/- 3ug), post batch stability check (+/- 15ug), and laboratory blank check (+/- 15ug).

- SESD will calculate the tare weight from the laboratory analyses and will calculate a concentration from the field data. All field data and laboratory data will be archived and backup data will be kept on the network.

Chain-of-custody and all accompanying field data must be maintained for all samples.

3 NON-CRITERIA POLLUTANT MONITORING FOR AIR POLLUTANTS FOR WHICH NATIONAL AMBIENT AIR QUALITY STANDARDS HAVE NOT BEEN ESTABLISHED.

3.1 Formaldehyde Sampling With Dinitrophenylhydrazine Cartridges Using Method TO-11A

The following is a synopsis of procedures which should be strictly adhered to for the handling and field use of dinitrophenylhydrazine (DNPH) cartridges for formaldehyde sampling. The following generic procedures should be adhered to at all times.

Summary of Sampling Method:

A glass cartridge using dinitrophenylhydrazine (DNPH) is placed in a special sampler and is equipped with pumps that withdraw a calibrated air volume for a specified time to capture formaldehyde and other carbonyl compounds. The cartridges are collected and shipped to the laboratory under controlled temperature.

- Quality Control requirements will be based on the method’s DQO.
- A minimum of one trip blank shall be transported per one to ten samples collected. Instrument blanks, duplicated samples or co-located samples will be based on laboratory request.
- Chain of Custody shall be maintained for all samples.
- Polyethylene or latex gloves must be worn whenever handling any of the DNPH cartridges (in the extraction laboratory, during preparation for shipment, during field set-up, in the field during preparation for return shipment, and in the laboratory during preparation for analysis and during analysis).
- All padding material shall be either clean tissue paper or polyethylene-air bubble padding. Never use polyurethane foam, cardboard, or newspaper as padding material. DNPH cartridges which have been properly prepared for shipment should be shipped in coolers with eutectic salt packs (Blue Ice).

Due to shelf life limitations, only the number of DNPH cartridges needed for a maximum of 180 days (including trip blanks and extra DNPH cartridges for breakage) should be ordered at any one time.

- On the date of shipment or when loading out for a study, the Air Monitoring Staff will remove the capped DNPH cartridges from the refrigerator in the Air Laboratory.

- An appropriate amount of packing material shall be placed in the shipping container to prevent breakage of the glass DNPH tubes. The DNPH tubes should be shipped in coolers containing eutectic salt packs (e.g., Blue Ice) to maintain a temperature of approximately 4^{o}C. The DNPH cartridges shall be shipped the same day they are packed.

- At each sampling location the cooler will be opened and one plugged DNPH cartridge will be removed (wearing clean polyethylene or latex gloves) and the two end plugs will be removed from each end of the DNPH cartridge which will be immediately placed on the sampling train. (If it is the SKC brand, the glass ends of the tubes must be broken off. A trip blank will be placed in a sampler enclosure at one of the sites for the duration of the sampling event.

- The sampler will be manually turned on and allowed to run for five minutes. An initial flowrate should be recorded on the sample data sheet. The timer should be set to turn the sampler on and off at the desired times.

- The operator should retrieve and secure the sample as soon as possible after the sampling period ends. The sampler should manually be turned on and allowed to run for five minutes and a final flowrate and the elapsed time from the time meter should be recorded on the sample data sheet. The exposed DNPH cartridge will then be removed from the sampling train (wearing clean polyethylene, latex or nylon gloves). The two end caps will be placed on the ends of the DNPH cartridge. The DNPH cartridge will be placed back into the cooler. The same procedure is to be followed at each sampling site.

- At the end of each day on which the samples are collected, the exposed DNPH cartridges shall be either placed in a refrigerator for storage overnight or stored in the cooler. The cooler will be used to transport the samples to the laboratory. The duration of the non-refrigerated period shall be kept to a minimum but absolutely not more than two days (including the sampling event).

- Upon arrival of the DNPH cartridges at the laboratory, the samples shall be stored in a refrigerator until extraction.

- Chain-of-custody and all accompanying field data must be maintained for all samples.

3.2 Volatile Organic Compounds (VOC) Sampling with SUMMA® Electropolished Stainless Steel Canisters or Silcosteel® Canisters Using Method TO-15A

The following is a synopsis of procedures which should be strictly adhered to for the cleanup and use of Summa® canisters for sampling air for Volatile Organic Compounds (VOC) analysis.

The following procedures must be followed in the preparation and use of Summa® canisters for collecting samples for VOC analysis:

- All new Summa®/Silcosteel® canisters must be individually checked for contamination by the laboratory before use. One of each batch of 10 Summa® canisters that are subsequently cleaned must be analyzed to check for contamination.

- All sampler tubing, fittings, and wetted parts of valves must be solvent washed in hexane and heated to >100° C. These parts should then be assembled and flushed with nitrogen[1] for at least 8 hours prior to use in the sample train or in the canister cleanup apparatus.

- Each canister's valve and fitting will be inspected for damage before cleaning. Any damaged valve will be replaced with a previously cleaned (see procedure above) valve. After replacing any valve, the canister will be cleaned and analyzed to verify that it is free of contamination.

- If any canister is used to sample a high concentration source, it must be cleaned and analyzed to verify it is free of contamination before it can be used again.

- Chain-of-custody must be maintained for all samples.

1. The nitrogen should be certified 99.999% pure by the manufacturer. A scrubber should be attached to the nitrogen line after the regulator to remove any trace impurities.

3.2.1 Sample Collection Procedure

Two types of VOC samples can be collected with Summa®/ Silcosteel® canisters. The canister can be opened and allowed to fill rapidly to obtain a grab sample or filled slowly by using a flow controller to collect a time integrated sample. With either type of sample, the following general procedures should be followed:

- A Chain-Of-Custody Record should be completed detailing time of sampling, sampling interval, and signed by the person collecting the sample.

- After the sample has been collected, the Summa®/Silcosteel® canister should be capped, an EPA pre-numbered tag should be completed and attached to the canister, and the canister should be placed in a shipping container with a copy of the Chain-Of-Custody Record and sealed with EPA sample custody tape.

3.2.2 Grab Sample Collection Procedure

Before a grab sample is collected for VOC analysis in a Summa®/Silcosteel® canister, the canister inlet valve should be fitted with a pre-cleaned stainless steel particulate filter. At the sample collection location, the main valve should be opened and the canister allowed to fill. After about one minute (when no audible sound of rushing gas can be heard), the main valve of the Summa® canister should be closed and capped.

3.2.3 Time Integrated Sample Collection Procedure

This sample collection method involves the use of a flow controller or a sampler containing a flow controller to slowly meter the flow of air entering a Summa®/Silcosteel® canister. With this method, a sample is collected over a longer period of time than with a grab sample. If a constant flowrate was maintained, the resulting sample will have a VOC content that is the average of the VOC concentrations during the sampling interval. The following procedures should be followed to collect time integrated samples:

- <u>Cleaning and Adjustment</u>
  All sampler systems should be checked for contamination prior to use or after any major repair. This is accomplished by metering humidified zero air to the inlet of the sampler. Excess zero air should be vented with a Swagelok® tee from the sampler inlet to atmosphere. The evacuated canister should then be filled at the normal sampling rate with the zero gas and verified by the laboratory to be free from contamination.

- <u>Sample Collection</u>
  Prior to sampling the flow controller will be calibrated a mass flow meter. The flowrate should be adjusted so that at the end of the sampling interval the ending pressure of the canister is approximately 0.9 atm. The final canister vacuum should be between 5 inches and 1 inch of Hg. The final flowrate should be at least 1 scc/min.

After sample collection, all canisters should be tagged with an EPA pre-numbered tag with all of the information filled out. Place the canister in a shipping container and seal the container with EPA sample custody tape.

3.3 Sampling for Semi-Volatile Organic Compounds (SVOC) Analysis with High Volume PUF Samplers Using Methods TO-4A & TO-13A

The following is a synopsis of procedures which should be strictly adhered to for use of the High Volume Polyurethane Foam (PUF) sampling method for collecting samples for semi-volatile organic compound (SVOC) analysis including pesticides and polychlorinated biphenyls.
The following procedures must be followed during preparation of PUF sampling media when using the High Volume PUF method for collecting samples for SVOC analysis:

- All PUF sampling media should be pre-cleaned, loaded into High Volume PUF sample cartridges, and sealed in solvent washed cans by the extraction laboratory prior to use.

- Chain-of-custody shall be maintained for all samples.

3.3.1 PUF Cleaning Procedure

- The Air Monitoring Staff has responsibility for buying the PUF media and cutting the PUF plugs. PUF media should be specified as <u>not</u> containing any fire retardants. It should be stored in the dark to prevent photo-oxidation. It should be less than two years old, and should be stored in a pesticide-free environment.

- Care should be exercised in cutting the PUF. It should be thoroughly wet with tap water prior to cutting. A drill press and stainless steel PUF cutting die should be used. The drill press area should be free of oil and a polyethylene cutting block should be used to stop the die at the bottom of the drill press stroke (do not use wood). Water should be sprayed on the die to help prevent snagging as the PUF is cut. After the plugs are cut, they should be rinsed with tap water and followed by a rinse with analyte-free water. Finally, the excess water should be squeezed out.

- The PUF/XAD cartridges are assembled using a modified glass sleeve containing an extra-extra coarse frit to retain the XAD resin in the following manner. A 3/4-inch layer of XAD resin is poured on top of the

  frit followed by a 1∠-inch PUF plug to retain the XAD resin.

- The assembled PUF/XAD cartridges are delivered to the extraction laboratory for cleaning and checking. The extraction laboratory will be given a minimum of three weeks notice for cleaning and checking the PUF/XAD cartridges. The cleaned PUF/XAD cartridges should be wrapped in aluminum foil and packed in pre cleaned metal cans or glass jars cushioned by new polyethylene bubble wrap to prevent breakage during shipment. Prepared PUF/XAD sample cartridges that are pre-packed in solvent washed metal cans will be obtained from the extraction laboratory prior to sampling. The cans should be packed inside coolers frozen eutectic salt packs (Blue Ice) to maintain a temperature of less than 15EC.

3.3.2 Sample Collection Procedure

The following procedure will be followed for all High Volume PUF/XAD sampling.

- Powder-free latex gloves will be used when handling all PUF/XAD cartridges and quartz particulate pre-filters. Assure that the red silicon upper and lower gaskets, located in the cartridge housing, are in place. Then remove the PUF/XAD cartridge from the shipping container, remove from the foil and insert the cartridge into the High Volume sampler's chamber. The pre-filter should be installed in the filter holder using caution not to over tighten the fittings. The foil should be placed back in the shipping container. The container should be labeled with site ID, operator's name, and sample date, and placed in the High Volume sampler enclosure until the sample is collected.

- The High Volume sampler should be turned on and allowed to run for two minutes. An initial flowrate should be recorded on the sample data sheet. The timer should be set to turn the sampler on and off at the desired times.

- The operator should retrieve and secure the sample as soon as possible after the sampling period ends. The sampler should then be manually turned on and allowed to run for two minutes. A final flowrate should be recorded on the sample data sheet. The final flowrate should be at least 150 liters per minute. The PUF/XAD cartridge should be removed, and the quartz pre-filter folded and placed in the top of the PUF/XAD cartridge. The PUF/XAD cartridge and pre-filter should be re-wrapped in the original aluminum foil and placed back in the shipping container. The container should then be tightly sealed. Complete the sample data sheet and Chain-Of-Custody Record and seal the shipping container with a sample custody seal. Finally, the shipping container containing the sample should be placed in a cooler containing frozen eutectic salt packs (at a nominal temperature of ~ 15^o C). When all samples are collected from all sites, the cooler should be sealed with sample custody tape for transport back to the laboratory.

- Upon arrival of the metal container at the laboratory, the samples shall be stored in the metal container in a refrigerator until submitted for extraction.

3.4 Collecting Samples for Metals Analysis Using the High Volume Sampler

The following is a synopsis of procedures which should be strictly adhered to for the collection of samples for metals analysis in air using the High Volume sampler:

- All quartz filters used will be supplied by the EPA National Filter Distribution Program, and of the same quality as supplied to the State and Local Agency Air Monitoring Stations.

- Prior to use, all filters will be checked for pinholes, and desiccated at 15^{o}C - 30^{o}C, ± 3^{o}C, and less than 50 percent relative humidity, ± 5 percent, for at least 24 hours.

- A filter field blank will be taken to the field, but not exposed. Filter field blanks will be analyzed by the laboratory to determine the concentration of metals contained in the filter matrix. The number of filter blanks will be determined based on a minimum of one blank for each ten samples collected.

- Chain-of-custody must be maintained for all samples.

3.4.1 Sample Collection Procedure

Samples will be collected using the High Volume sampler as described, and operated in accordance with 40 CFR, PART 50, APPENDIX B:

- All flow calibration orifices will be traceable to a Primary Standard Rootsmeter. Flows will be corrected to EPA standard temperature and pressure (25^{o}C and 760 mm Hg).

- Digital manometers used to determine flow rates will calibrated by the factory annually.

- Air Monitoring Staff personnel will remove a 2-inch strip of the exposed filter from one end and discard it. Two 1-inch strips will be cut from the same end and transported to the laboratory for analysis.

3.4.2 Integrated Sample Collection Procedure

The following procedure should be followed to collect time integrated samples:

- Initial and final flow rates will be determined with a calibrated orifice and a digital manometer.

- After the sample has been collected, the filter will be folded lengthwise and placed in a filter holder. The filter holder is then placed in an envelope and the envelope sealed.

- A Chain-Of-Custody Record should be completed which contains the time of sampling, the sampling interval, and the signature of the person taking the sample.

After sample collection, all sample envelopes will be placed in an appropriate container. An EPA custody seal will be placed on the container. Filters will be cut by the Air Monitoring Staff and transported to the laboratory for analysis.

3.5 Sampling for Dioxin and Dibenzofuran Analyses with High Volume PUF Samplers Using Method TO-9A

3.5.1 General Information

The following is a synopsis of procedures which should be strictly adhered to for use of the High Volume Polyurethane Foam (PUF) sampling method for collecting samples for polychlorinated dibenzo-p-dioxins and dibenzofurans analyses.

Since this method requires High-Resolution Mass Spectrometry which the Region 4 laboratory does not have, all sample media preparation and analysis will have to be contracted. At least one month's notice prior to sampling should be given to obtain a contract laboratory for any dioxin and dibenzofuran analysis. It is important that the contract specify a number of details to assure accurate results:

- All of the PUF media and a representative number of each batch of quartz pre-filters should be checked by the contract laboratory to assure that there is no contamination. Each PUF plug should be pre-spiked by the contract laboratory with dioxin and dibenzofuran surrogates as a check of the accuracy of the method.

- Each set of PUF plugs and quartz filters should be securely packed in sealed containers and in coolers to prevent damage during shipment. The sampling media should be shipped air freight to minimize the time between cleanup and sampling.

- Chain-of-custody shall be maintained for all samples.

3.5.2 Sample Collection Procedure

The following procedure will be followed for the collection of all High Volume PUF samples for dioxins and dibenzofurans analyses.

Latex gloves will be used when handling all PUF cartridges and quartz particulate pre-filters. Assure that the red silicon upper and lower gaskets are in place in the PUF cartridge housing. Remove the PUF cartridge from the shipping can. Unwrap and insert the PUF cartridge into the High Volume (Hi-Vol) PUF sampler's chamber. Install the pre-filter in the filter holder using caution not to over-tighten the fittings. The removed aluminum foil should be placed in the shipping container which then should be resealed. The container should be labeled with the site ID, the operator’s name, and the sample date, and placed in the Hi-Vol PUF sampler enclosure until the sample is collected. The Hi-Vol PUF sampler should be turned on and allowed to run for two minutes. An initial flowrate should be recorded on the sample data sheet. The timer should be set to turn the sampler on and off at the desired times.

The operator should retrieve and secure the sample as soon as possible after the sampling period ends. The sampler should then be manually turned on and allowed to run for two minutes and a final flowrate recorded on the sample data sheet. The final flowrate should be at least 150 liters/minute. The PUF cartridge should be removed, and the quartz pre-filter folded and placed in the top of the PUF cartridge. The PUF cartridge and pre-filter should be re-wrapped in the original aluminum foil and placed back in the shipping container and the container should be tightly sealed. Complete the sample data and sample custody sheets. Each shipping container should have a sample custody seal. Finally, the shipping container containing the sample should be placed in a cooler containing frozen eutectic salt packs (at a nominal temperature of ~ 4^{o}C). When all samples are collected from all sites, the cooler should be sealed with sample custody tape for shipment to the contract laboratory.

Upon arrival of the metal container at the contract laboratory, the samples shall be stored in the metal container in a refrigerator until submitted for extraction.