

Appendix L
Air Monitoring Sampling and Analysis Plan

Appendix L

Final Prescribed Burn Air Sampling and Analysis Plan Munitions Response Site 16 Former Fort Ord, California

Prepared for

Shaw Environmental & Infrastructure, Inc.
Bldg 4522 Joe Lloyd Way
Monterey, California 93944

MACTEC Project No. 4088053164 04.3

Matthew H. Walraven

Matthew H. Walraven
Project Environmental Scientist

By MLH with permission

Bruce L. Wilcer

Bruce L. Wilcer
Principal Geologist

July 28, 2006



MACTEC

Engineering and Consulting, Inc.
5341 Old Redwood Highway, Suite 300
Petaluma, CA 94954 - (707) 793-3800

Appendix L

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Munitions Response Site 16

Former Fort Ord, California

MACTEC Project No. 4088053164 04.3

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AIRMETRICS MINIVOL® PORTABLE AIR SAMPLER MACTEC SOP-104

LIST OF ACRONYMS AND ABBREVIATIONS

Army	U.S. Department of the Army
ATSDR	Agency for Toxic Substances and Disease Registry
CAAA	Clean Air Act Amendments
CARB	California Air Resources Board
CDQMP	Chemical Data Quality Management Plan
COC	Chain-of-Custody
COPC	Chemicals of Potential Concern
DQOs	Data quality objectives
DTSC	Department of Toxic Substances Control
EPA	Environmental Protection Agency
Fort Ord	The former Fort Ord
GPS	Global Positioning System
IDW	Investigation-derived waste
MACTEC	MACTEC Engineering and Consulting, Inc. (formerly Harding ESE, Inc.)
MBUAPCD	Monterey Bay Unified Air Pollution Control District
MEC	Munitions and Explosives of Concern
MRS	Munitions Response Site
PM ₁₀	Particulate Matter Less than 10 microns
POM	Presidio of Monterey
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
SAP	Sampling and Analysis Plan
Shaw	Shaw Environmental, Inc.
SMP	Smoke Management Plan
SOP	Standard Operating Procedure
SSHP	Site Safety and Health Plan
SSWP	Site Specific Work Plan
TWA	Time Weighted Average
µg/m ³	Micrograms per cubic meter
USACE	U.S. Department of the Army, Corps of Engineers
USEPA	U.S. Environmental Protection Agency

L1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) presents procedures for the collection and analysis of air samples during the prescribed burn event for Munitions Response Site (MRS) 16 at Former Fort Ord. The site history, background, historical use of munitions and explosives of concern (MEC), and details of the prescribed burn are presented in the site specific work plan (SSWP, main body of this report).

Two inter-related work activity plans, the *MRS 16 Prescribed Burn Plan, (Presidio of Monterey[POM] Fire Department, 2006)*, and *Munitions Response Site 16 Site Specific Work Plan* (main body of this report) will be implemented during the completion of the prescribed burn at MRS-16.

The Burn Plan describes the objectives of the prescribed burn; the burn area; the range of environmental conditions under which the burn will be conducted; the manpower and equipment resources required to ignite, manage, and execute the fire; and the smoke management plan. Procedures for conducting the burn within the window of environmental conditions established in the burn prescription are discussed in the Burn Plan.

The Site-Specific Work Plan (SSWP) describes the procedures, methods, and resources that Shaw and its subcontractors will use while performing MEC removal activities as well as other monitoring activities related to the program.

The Air Sampling and Analysis Plan (this document) is presented as appendix L to the SSWP, and outlines procedures for the collection and analysis of air samples during the prescribed burn. The purpose of collecting and analyzing air samples during the prescribed burn is to 1) provide data to assess the adequacy of the burn prescription relative to smoke dispersion and downwind impacts and 2) monitor and evaluate whether prescribed burns at the former Fort Ord result in downwind ambient concentrations of PM₁₀ that exceed the applicable health-based screening level (California ambient air quality standard).

L2.0 PROJECT ORGANIZATION

The work described in this SAP will be performed by MACTEC Engineering and Consulting, Inc. (MACTEC) under subcontract to Shaw Environmental, Inc (Shaw). The overall project organization is presented in the SSWP (main body of this report). Project organization for key MACTEC personnel is described below.

L2.1 Project Manager

The MACTEC Project Manager for the former Fort Ord project, Mr. Bruce Wilcer, is responsible for the implementation of USACE and regulatory agency requirements for the project, and reports directly to the Shaw Task Manager. These responsibilities may include oversight of the development and completion of project documents and related activities as well as schedule and contract management, technical oversight, report generation, implementation of three-phase QC activities, and overall project quality.

L2.2 Task Manager

The MACTEC Task Manager for the sampling program described in this SAP, Mr. Matthew Walraven, is responsible for implementation and oversight of activities related to project performance, and reports directly to the MACTEC Project Manager. This responsibility includes development of project documents, implementation of project activities, management of the schedule and contract, oversight of technical issues, generation of reports, and implementation of the QC procedures specified in this SAP.

L2.3 Project Chemist

The Project Chemist, Mr. Scott Tucker, provides input for development of this SAP, as well as management of project tasks associated with sampling and preservation requirements, general oversight of field personnel in sampling activities, and coordination of sample collection and shipment to the analytical laboratory. The Project Chemist reports directly to the MACTEC Project Manager and provides oversight of and coordination with the analytical laboratory, review and evaluation of analytical data, and implementation of three-phase QC activities and corrective actions (as necessary). The Project Chemist will also conduct a project kick-off meeting with the analytical laboratory prior to sample collection or analysis.

L3.0 PRESCRIBED BURN OPERATIONS

A description of the project background, including the location of the site, historical use, and description of MEC use at MRS-16 is provided in the SSWP. The following section summarizes the prescribed burn operations.

The Army recognizes that smoke produced by prescribed burn events has the potential to create short-term impacts on local air quality and potential temporary impacts on public health downwind. Fire and smoke management procedures are outlined in the *MRS-16 Prescribed Burn Plan* (Prescribed Burn Plan) (*POM Fire Department, 2006*). Approximately 58 acres of vegetation is expected to be burned in approximately 3 hours of active ignition, and smoke will likely be in the air for several more hours as described in the Prescribed Burn Plan. As a result of information gained from the prescribed burn performed in Ranges 43-48 in October 2003 (described in the *Draft Final, Ranges 43-48 Prescribed Burn Air Monitoring Report [MACTEC, 2004]*), the following components will comprise the prescribed burn at MRS-16:

- Cutting of approximately 28 acres of vegetation comprising the MRS-16 fuel break
- Prescribed burning of approximately 58 acres of vegetation
- Implementation of the burn prescription, which includes fuel moisture and atmospheric condition criteria as identified in the Prescribed Burn Plan. Procedures designed to enhance smoke management; including optimization of ignition patterns and modification of the burn prescription are also described in the Prescribed Burn Plan
- Notification to local communities prior to the prescribed burn event at MRS-16, using the procedures described in Appendix M - Notification Plan, in this document.

L4.0 PROJECT DESCRIPTION

This section provides a brief description of the project in terms of objective and scope. Details of the sampling program are provided in subsequent sections.

L4.1 Objective and Scope

This SAP outlines procedures for collection and analysis of air samples in areas potentially affected by air emissions from a prescribed burn at MRS-16. The results of other relevant studies and investigations have been considered during the development of this SAP to determine the chemicals of potential concern (COPCs) that will be monitored during the prescribed burn. In accordance with the California Clean Air Act (Title 17, CCR, 80100 et. seq.), a requirement for appropriate monitoring of burns that affect smoke sensitive areas is part of the Monterey Bay Unified Air Pollution Control District's (MBUAPCD) Smoke Management Program (SMP). Potential methods identified in the SMP for monitoring smoke impacts include visual observation and PM₁₀ analysis. Data collected from air monitoring will be used to plan future prescribed burns. As described earlier, the objectives of the sampling and analysis program described herein are to:

1. Provide data to assess the adequacy of the burn prescription relative to smoke dispersion and downwind impacts. The air sampling program is therefore focused on detection and quantification of particulate matter related to vegetation combustion (PM₁₀).
2. Evaluate whether prescribed burns at the former Fort Ord result in downwind ambient concentrations of PM₁₀ that exceed the applicable health-based screening level (California ambient air quality standard).

Air monitoring for PM₁₀ will be conducted starting at the active burn stage and continuing for a 24-hour period during the prescribed burn effort.

L5.0 PROJECT OVERVIEW AND DATA QUALITY OBJECTIVES

L5.1 Overview

During a prescribed burn, the smoke generated from the burning of vegetation may contain inhalable particulate matter (suspended particles less than 10 microns in diameter [PM_{10}]), which can cause temporary respiratory distress to sensitive populations during the burn event. The intense fire associated with prescribed burn conditions may also result in the incidental detonation of surface or near-surface MEC items. Detonation of MEC present at the site has the potential to release air pollutants to the atmosphere. These additional air emissions may potentially include combustion products, volatile or semivolatile organic compounds, unburned or incompletely burned energetic material, and particulate metals and metal compounds from chemical components of the MEC items. Although MEC-related emissions may also be generated during a prescribed burn, ground-level concentrations of these pollutants have been shown to remain well below human health-protective regulatory screening levels as described in the *Draft Final, Ranges 43-48 Prescribed Burn Air Monitoring Report (MACTEC, 2004)*. Data presented in the *Draft Prescribed Burn Supplemental Report, Ranges 43-48 (MACTEC, 2005)* also indicates that, with the exception of inhalable particulate matter, there is no sound technical reason for sampling air emissions resulting from combustion of vegetation such as acrolein and aldehydes. As a result, only samples for inhalable particulate matter (PM_{10}) will be collected during the MRS-16 prescribed burn. Smoke management techniques described in the SSWP will be employed by the Army to minimize public exposure to smoke from prescribed burns at the former Fort Ord. The purpose of the sampling and analysis program described herein is to:

1. Provide data to assess the adequacy of the burn prescription relative to smoke dispersion and downwind impacts.
2. Monitor and evaluate whether prescribed burns at the former Fort Ord result in downwind ambient concentrations of PM_{10} that exceed the applicable health-based screening level (California ambient air quality standard).

Data quality objectives (DQOs) for the project are discussed in the sections below.

L5.2 Data Quality Objectives

L5.2.1 Statement of the Problem

Combustion of vegetation has potential temporary smoke impacts to downwind sensitive receptors. These smoke impacts need to be evaluated relative to human health screening levels. The identity and quantity of products and residues emitted to the air from prescribed burn activities at the former Fort Ord has been documented in previous studies, including a previous ambient air sampling investigation conducted at Ranges 43-48 (*MACTEC, 2004*). This previous investigation has concluded that MEC-related air contaminants downwind of that prescribed burn at the former Fort Ord did not exceed health-based screening levels, and that the best indicator of smoke impacts at public receptors is PM_{10} (*MACTEC, 2005a*).

L5.2.2 Identification of Decisions

The primary decisions related to this project are to (1) evaluate whether prescribed burns at the former Fort Ord result in downwind ambient concentrations of PM_{10} that exceed the applicable health-based

screening level (California ambient air quality standard), and (2) provide data to support the evaluation of the burn prescription relative to smoke dispersion and downwind impacts to the public.

L5.2.3 Identification of Inputs to Decisions

Inputs to decisions necessary for evaluating downwind impacts from prescribed burn activities at former Fort Ord are as follows:

- Identify target list of COPCs
The target list of COPCs for the MRS-16 prescribed burn has been identified through evaluation of data presented in previous technical publications, including the *Draft Final Summary After-Action Report: Ranges 43-48 Prescribed Burn, Former Fort Ord, California (Army, 2004) Draft Final, Prescribed Burn Supplemental Report, Ranges 43-48 (MACTEC 2006)*, and *Health Consultation, Former Fort Ord Site (Agency for Toxic Substances and Disease Registry, 2005)*. Based on the information provided in these documents, PM₁₀ was selected as the COPC for the MRS-16 prescribed burn.
- Identify appropriate screening levels for COPCs (i.e., PM₁₀) in air.
- For PM₁₀, the California Ambient Air Quality Standard for was selected as the most appropriate screening level for the project. This decision is based on previous coordination between the Army, USACE, U.S. Environmental Protection Agency, Region 9 (EPA), California Environmental Protection Agency (Cal EPA), Department of Toxic Substances Control (DTSC), California Air Resources Board (CARB), and Monterey Bay Unified Air Pollution Control District (MBUAPCD) during the development of the sampling and analysis plan for the prescribed burn at Ranges 43-48 (*MACTEC, 2003*).
- Measure downwind concentrations of COPCs in air during a prescribed burn event.
- Record field observations, including wind speed and direction during the event, and visually identify the downwind area that received smoke impacts.

L5.2.4 Definition of Study Boundaries

The study boundary is defined as the area downwind of the prescribed burn event that includes adjacent populations that could receive smoke impacts. The maximum extent of placement of samplers (study boundary) will be based on predictions of air flow patterns from air modeling studies and information collected from previous burn events. Air samples will be collected on the day of the prescribed burn event, over a 24-hour period.

L5.2.5 Development of Decision Rules

If measured concentrations of PM₁₀ in air are less than established screening levels, then no modifications will be made to future prescribed burn operations.

If measured concentrations of PM₁₀ in air are greater than or equal to established screening levels, then modifications to future prescribed burn operations will be evaluated.

L5.2.6 Specification of Limits on Decision Errors

The null hypothesis is that, following this investigation, no modifications to future prescribed burn operations will be necessary ("future prescribed burn operations" in this context includes burn tactics for separate burns at later dates).

A false positive decision error would be to conclude that modifications are necessary when, in fact, they are not. The consequence of this error would be that unnecessary modifications or limitations to future prescribed burn operations would be made, resulting in unnecessary cost to the government.

A false negative decision error would be to conclude that modifications are not necessary when, in fact, they are. The consequence of this error would be that future prescribed burn operations with unacceptable adverse impacts to human health would occur.

This investigation employs a biased sampling strategy designed to characterize areas of maximum impact. Consequently, confidence limits on decision errors are not applicable to this investigation. The expected COPC distribution is not random; hence, the judgmental sampling strategy proposed does not lend itself to statistically derived confidence levels for decision errors. However, decision errors will be controlled by adhering to the procedures specified in this SAP, including specific QA/QC procedures as defined in the sampling and analytical methods. In particular, analytical laboratory QA/QC acceptance criteria will be used to verify that measurements are capable of supporting project decisions.

L5.2.7 Optimization of Investigation Design for Obtaining Data

The sampling investigation design includes the following factors:

- Number of air samples
- Location of air samples
- Sampling and analytical methods
- QC samples.

Rationale for the sampling investigation design includes the following factors and objectives:

- To obtain samples that characterize the degree of smoke impact to downwind public receptors
- To obtain samples that characterize the spatial extent of smoke impact
- Because the downwind COPC concentration distribution is nonrandom within the study area, a judgmental sampling design is appropriate. Also, because decisions will not be made using mean concentrations compared to the screening level, probabilistic sampling design is not useful.

Factors to consider to optimize sampling investigation design include the following:

- The location of the highest concentrations of PM₁₀ in air may vary during the event as meteorological conditions evolve throughout the day.
- Field sampling methods should facilitate rapid mobilization of sampling units to locations of predicted smoke impact the day of or day before the prescribed burn event.

The sampling strategy for PM₁₀ in air will be to conduct the following activities:

- Collect air samples in multiple areas most likely to experience smoke impacts (based on observations from previous prescribed burn events, wind direction predictions for the specific burn day, and/or pre-burn smoke dispersion modeling).
- Collect time weighted average (TWA) air samples from onset of the prescribed burn and lasting until active burning is completed (called “burn event” sample; estimated to be about 8 to 10 hours).
- After the conclusion of the burn event sample, collect a second TWA sample to complete a 24-hour sample interval (e.g., if the burn event is 8 to 10 hours, then the second sample will be 14 to 16 hours in duration).
- Samples shall be measured/analyzed for PM₁₀ as shown in Table 1.

L6.0 SAMPLING PROCESS DESIGN

Results of the DQO process described in Section 5.0 were used to develop the sampling process design for this project. This section describes the details of the planned sampling, such as the number and location of samples. The rationale and assumptions used for sample process design are presented in Section 5.7. Section 7.0 provides a description of the sampling and analytical methods.

L6.1 Objective and Basic Design

The primary objectives of this field investigation are to:

1. Determine if ground-level concentrations of PM₁₀ downwind of a burn event are above or below the applicable health-based screening level (California ambient air quality standard), and
2. Assist in assessing the adequacy of the burn prescription for minimizing downwind smoke impacts to the public.

The California Ambient Air Quality Standard human health standard for PM₁₀ is based on a time-weighted average (TWA) for a 24-hour period. In order to collect a TWA 24-hour sample, and have the ability to assess the highest impact periods during the burn, two sampling intervals will be used. The first set of samples will be collected during the burn event, which is expected to last approximately 8 to 10 hours. The burn event includes approximately 3 hours of active ignition and several subsequent hours during which visible smoke may continue to be present. A second set of samples will be collected following the burn event to comprise the TWA sample for the remainder of the 24-hour period.

This air sampling program may include support and participation from both Army and regulatory agency resources. Most sampling equipment and field personnel will be provided by MACTEC under subcontract to Shaw, who is contracted directly with the USACE.

The sampling locations and procedures described in this SAP will be followed to the maximum practicable extent during this field investigation. However, deviations from the SAP may be required because of unforeseen conditions in the field. Any deviations from the SAP must be approved by the Task Manager prior to implementation, and will be documented in writing in a Field Change Notification.

L6.2 Air Sampling Locations

A total of seven sampling locations (five that are pre-selected and two that will be located the day of, or day before the burn) are proposed for the MRS-16 Prescribed Burn. The proposed five pre-selected sampling stations are located in residential or otherwise public areas that, based on the data collected during past burn events, may be affected by smoke impacts (Plate 1). Because the actual areas of smoke impact cannot be known in advance of a burn event, the five pre-selected sampling stations will be supplemented with two sampling stations that will be deployed in one of six proposed areas, based on the smoke dispersion modeling the day before the prescribed burn. The sampling program described here for MRS-16 requires only PM₁₀ analysis, which does not require commercial electrical power to run the equipment, allowing the selected stations to be located quickly to areas of predicted smoke impacts the day before or on the day of the burn, as appropriate.

The decision on where to place the selected sampling stations on the day of the prescribed burn will be made by the Task Manager in the field based on modeling results and will focus on areas between pre-selected sampling units that may receive smoke impacts. Plate 1 presents six general candidate locations

for additional sampling stations. Two of these six locations will be selected by the task manager on or before the day of the burn based on the above observations. The actual final locations of the stations will be recorded using Global Positioning System (GPS) technology.

L7.0 SAMPLING AND ANALYTICAL METHOD REQUIREMENTS

The proposed sampling program includes collecting PM₁₀ samples as summarized in Table 1 as follows:

- TWA air samples during the active burning (burn event sample) approximately 8-10 hours
- Second set of TWA samples collected over a duration that will complete the 24-hour monitoring period.
- One trip blank sample will be submitted to the laboratory for each sampling event per day
- Duplicate (QC) air samples (one duplicate for each sampling event, per day).

Air samples will be collected at seven locations for a 24-hour period during the burn day events:

Air samples for PM₁₀ will be integrated TWA samples where samples are collected on or in a specific media for subsequent laboratory analysis. Each 24-hour TWA sample will be comprised of approximate 8- and 14-hour samples beginning at the initiation of the burn and terminating approximately 24 hours later. All samples will be collected at approximately two (2) meters above ground level (agl), which is at or near human adult breathing zone and within the probe siting criteria recommended by the USEPA (USEPA, 1987). USEPA guidance for spacing from obstructions will also be followed.

The air sampling and analytical methods that will be used to monitor PM₁₀ in this investigation are summarized in Table 1 and described in the following sections. The analytical laboratory for this project will be certified by the State of California Environmental Accreditation Program, as applicable, and analyses will be performed in accordance with the requirements set forth in USEPA Compendium Method IO-3.1. All sampling media will be maintained under strict chain-of-custody (COC) control. Sampling media will be uniquely identified and labeled, and the time, location, and duration of exposure will be recorded on COC forms. All COC forms will be completed and signed by the sampling technician before relinquishing the exposed media for shipment to the analytical laboratory. Duplicate samples will be handled identically to exposed media, and will be submitted "blind" to the laboratory for analysis.

L7.1 Particulate Matter Less than 10 Microns (PM₁₀)

Sampling Method

To maximize the flexibility of the sampling program, battery-operated low-volume sampling equipment (e.g., Airmetrics MiniVol® Sampler or equivalent) will be used to collect integrated TWA air samples for PM₁₀ analysis at the seven sampling locations. Samples will be collected using Teflon® filter media. Sampling operations will be in accordance with the Standard Operating Procedure number MACTEC SOP 104 (Appendix C) that meets the requirements established in Reference 1, USEPA Compendium Method IO-2.1 (Appendix A, modified for low volume sampling).

Analysis Method

Filter mass concentration of the low-volume air sampler will be determined gravimetrically. Pre- and post-exposure filter weights will be assessed with a microbalance. The total volume of air sampled, corrected to USEPA reference condition, is determined from the measured air flow rate and the sample time. The mass concentration of PM₁₀ in the ambient air is computed as the total mass of captured particles in the PM₁₀ size range divided by the volume of air sampled, and is expressed in micrograms per

cubic meter ($\mu\text{g}/\text{m}^3$). Filter weighing procedures will be conducted by an analytical laboratory and will meet requirements specified in Reference 2, USEPA Compendium Method IO-3.1 (Appendix B).

Performance Criteria and Quality Control

One field duplicate sample will be collected and analyzed per day of sampling, and field (trip) blanks will be submitted on a frequency of 10% of total samples (the field blank filter will be shipped to the field and returned without drawing air through the sampler). Laboratory QA/QC samples, at a minimum, will be performed at the frequency specified in the analytical method. Analytical parameters such as initial calibration and instrument conditions will be in compliance with the acceptance criteria as specified in the analytical method.

References

1. USEPA Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Compendium Method IO-2.1. Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM_{10} Using High-Volume (HV) Sampler (modified for low volume and less than 24 hour sampling). EPA/625/R-96/010a, Center for Environmental Research Information, Cincinnati, Ohio. June 1999.
2. USEPA Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Compendium Method IO-3.1. Selection, Preparation and Extraction of Filter Material. EPA/625/R-96/010a, Center for Environmental Research Information, Cincinnati, Ohio, June 1999.
3. MACTEC. Standard Operating Procedure for Airmetrics MiniVol® Sampler. MACTEC SOP 104. March 2006.

L7.2 Decontamination Procedures

Each air sample will be collected on individual sampling media as supplied by the laboratory. Decontamination procedures will not be required because all sample tubing and pumps will be downstream of the collection media, and no sampling media will be re-used.

L8.0 INVESTIGATION-DERIVED WASTE

Investigation-derived waste (IDW) consists of tools, personal protective equipment, soil cuttings, wash water, and other disposable items that may have come into contact with site contaminants. IDW is not expected as part of this investigation.

L9.0 QUALITY CONTROL

Contractor QC activities relative to the acquisition and reporting of chemical data are described in the CDQMP (*MACTEC, 2002*). The project and task managers and the project chemist for this project are described in Section 2.0 of this SAP. This section describes the QC procedures specific to the MRS-16 prescribed burn air sampling.

L9.1 Three-Phase Quality Control Process

The three-phase QC process, as described below, will be implemented for the prescribed burn air sampling activities. Each phase of QC is important for obtaining a quality product, however since the prescribed burn is anticipated to be a one-day event, the preparatory and initial inspections are most applicable to the scope of the project. Production work is not to be performed on a definable feature of work until a successful preparatory and initial phase inspection have been completed.

During these inspections, the Project Manager will verify implementation of the requirements of this SAP and the relevant requirements of the CDQMP.

L9.1.1 Preparatory Phase Inspection

Before conducting a feature of work, the Project Manager shall check that technical requirements have been planned for and that work prerequisites have been identified and met. The Project Manager's responsibility is to check that lessons learned during previous similar work have been incorporated as appropriate into the project procedures to prevent recurrence of past problems. Minimum review criteria for a preparatory phase inspection are contained in Section 4.1.3 of the CDQMP (*MACTEC, 2002*). Discrepancies among existing conditions and approved plans/procedures are to be resolved before the day of the prescribed burn, and completion of corrective actions for unsatisfactory and nonconforming conditions identified during a preparatory inspection is to be verified by the Project or Task Manager prior to granting approval to begin work. In addition, the Project Chemist will hold a project kick-off meeting with the analytical laboratory to discuss the analytical and deliverable requirements for this project as described in this SAP. Minutes of this meeting will be documented and reported to Shaw in a letter report. Laboratory kick off meetings and necessary technical systems audits will be completed prior to the submittal of samples for analysis.

L9.1.2 Initial Phase Inspection

The second QC phase consists of checks performed during the initial field activities. On the day of the prescribed burn, the Task Manager will monitor the work and verify compliance with the specifications and requirements of the contract, delivery order, and approved plan and procedures. Minimum review criteria for the initial phase inspection are provided in Section 4.1.3 of the CDQMP (*MACTEC, 2002*). The Task Manager is also responsible for verifying that a daily health and safety (H&S) inspection is performed and documented as prescribed in the site safety and health plan (SSHP). The Project Manager or designee is responsible for ensuring that discrepancies between site practices and approved specifications are identified and resolved. Discrepancies between site practices and approved plans/procedures are to be resolved and corrective actions for unsatisfactory and nonconforming conditions or practices are to be verified by the Project Manager or designee prior to granting approval to proceed. .

L9.1.3 Follow-Up Phase Inspection

The follow up phase of the three phase quality control process is designed for projects of multiple day duration, and do not apply as described in Section 4.1.3 of the CDQMP. The follow up phase inspection for this project will consist of a review of the samples collected and documentation of sample collection for compliance with the specifications described in this SAP by the Task Manager. Documentation of any deviation from the project plans will be verified by the Project Manager and provided in writing to Shaw as a non-conformance report.

L9.2 Additional Inspections

Additional inspections performed on the same definable feature of work may be required at the discretion of the USACE or the Project Manager. Additional preparatory and initial inspections are generally warranted under any of the following conditions: unsatisfactory work, as determined by program or project management or USACE; changes in key personnel; resumption of work after a substantial period of inactivity (e.g., 2 weeks or more); and changes to the project scope of work/specifications.

L9.3 Deliverables

The following deliverables will be prepared:

- Laboratory kick off meeting minutes,
- Comprehensive certificate of analysis (from analytical laboratory), and
- Non-Conformance Report (as needed).

L9.4 Data Validation

Data validation will consist of review and re-calculation of the laboratory raw data to verify accuracy of concentrations reported. The laboratory will provide the equivalent of an EPA Level 4 data package for each data set submitted for analysis. The Project Chemist will be responsible for coordinating or performing data validation efforts.

L10.0 REFERENCES

Agency for Toxic Substances and Disease Registry (ATSDR), 2005. *Health Consultation, Former Fort Ord Site*. February 3.

Harding Lawson Associates (HLA), 1994. *Draft Final Technical Memorandum, Preliminary Remediation Goals, Fort Ord, California*. June 24.

Harding ESE, Inc. (Harding ESE), 2001. *Technical Memorandum, Air Emissions from Incidental Ordnance Detonation During a Prescribed Burn on Ranges 43-48, Former Fort Ord, California*. November 9.

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TABLES

**Table L1. Screening Level, Sampling and Analytical Methods
and QA/QC Requirements for PM₁₀
Prescribed Burn Air Sampling and Analysis Plan
Former Fort Ord, California**

Analyte Class	Analyte	Air Screening Level ($\mu\text{g}/\text{m}^3$)	Screening Level Reference
Particulate Matter	Particulate Matter less than 10 microns (PM ₁₀)	50(24-hour)	California AAQS ¹

¹ California Ambient Air Quality Standard

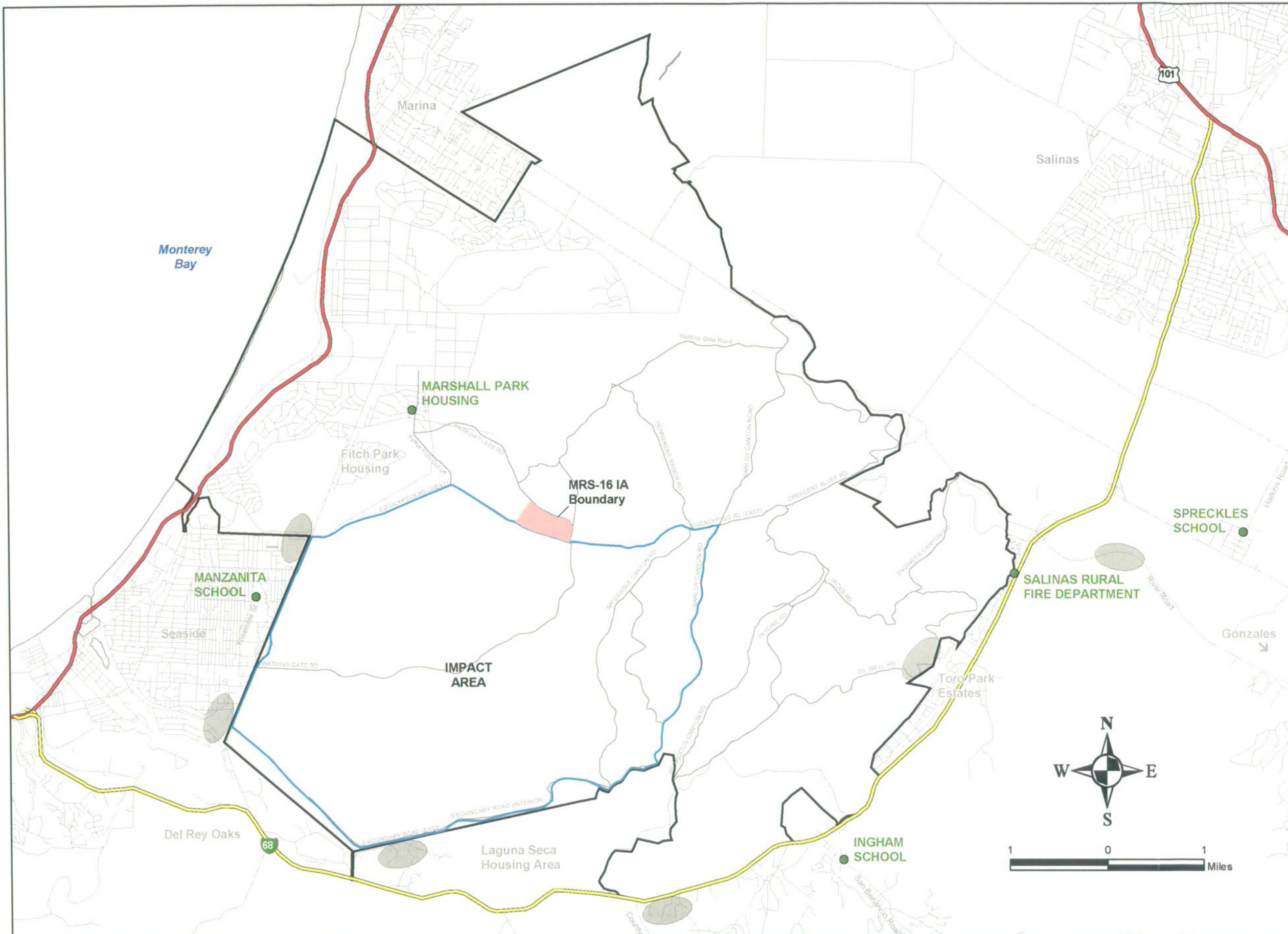
Pollutant	Sampling Equipment	Sampling Method	Analytical Method	Quality Assurance / Quality Control		
				Field Duplicates	Field Blanks	Lab QA/QC
Particulate Matter < 10 microns (PM ₁₀)	Low Volume Sampler with Size-Selective Inlet equipped with Teflon filter (Airmetrics MiniVol®)	USEPA Compendium Method IO-2.1, modified for low volume and less than 24 hour sampling	USEPA Compendium Method IO-3.1	One per day of sampling	10%	See Note 1

Note 1: Laboratory QA/QC samples, at a minimum, will be performed at the frequency specified in the analytical method. Analytical parameters such as initial calibrations and instrument conditions will be in compliance with the acceptance criteria as specified in the analytical method.

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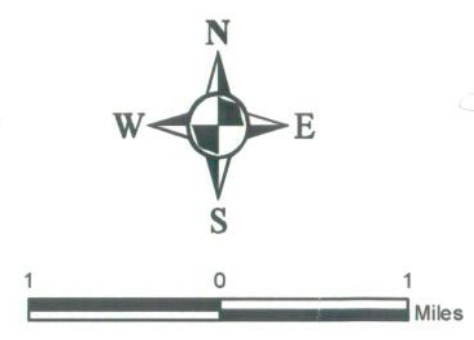
Approved: BLW

PLATES



- EXPLANATION**
- Proposed Monitoring Station
 - Potential Monitoring Station*
 - Impact Area
 - ▭ Munitions Response Site 16 Interim Action Boundary
 - ▭ Fort Ord Boundary
 - Road Classification
 - Limited Access Highway
 - Highway
 - Roads

* Specific locations will be selected the day before or the day of the burn based on meteorological modeling results.



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DATE: 05/2006	DATE:



Prescribed Burn
Air Sampling and Analysis Plan
Former Fort Ord, California

Munition Response Site 16 and
Proposed Monitoring Locations

PLATE
1

ATTACHMENT L-A

USEPA COMPENDIUM METHOD IO-2.1

**Compendium of Methods
for the Determination of
Inorganic Compounds
in Ambient Air**

Compendium Method IO-2.1

**SAMPLING OF AMBIENT AIR
FOR TOTAL SUSPENDED
PARTICULATE MATTER (SPM)
AND PM₁₀ USING
HIGH VOLUME (HV) SAMPLER**

Center for Environmental Research Information
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268

June 1999

Method IO-2.1

Acknowledgments

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- James L. Cheney, U.S. Army Corps of Engineers, Omaha, NE
- Michael F. Davis, U.S. EPA, Region 7, KC, KS
- Joseph B. Elkins Jr., U.S. EPA, OAQPS, RTP, NC
- Robert G. Lewis, U.S. EPA, NERL, RTP, NC
- Justice A. Manning, U.S. EPA, ORD, Cincinnati, OH
- William A. McClenny, U.S. EPA, NERL, RTP, NC
- Frank F. McElroy, U.S. EPA, NERL, RTP, NC
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

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Author(s)

- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

Peer Reviewers

- David Brant, National Research Center for Coal and Energy, Morgantown, WV
- John Glass, SC Department of Health and Environmental Control, Columbia, SC
- Jim Cheney, U.S. Army Corps of Engineers, Omaha, NE
- Margaret Zimmerman, Texas Natural Resource Conservation Commission, Austin, TX
- Lauren Drees, U.S. EPA, NRMRL, Cincinnati, OH

DISCLAIMER

This Compendium has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Method IO-2.1
Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM₁₀ Using High Volume (HV) Sampler

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Chapter IO-2 Integrated Sampling of Suspended Particulate Matter (SPM)

Method IO-2.1 SAMPLING OF AMBIENT AIR FOR TOTAL SUSPENDED PARTICULATE MATTER (SPM) AND PM₁₀ USING HIGH VOLUME (HV) SAMPLER

1. Scope

1.1 Suspended particulate matter (SPM) in air generally is a complex, multi-phase system of all airborne solid and low vapor pressure liquid particles having aerodynamic particle sizes from below 0.01-100 μm and larger. Historically, SPM measurement has concentrated on total suspended particulates (TSP), with no preference to size selection.

1.2 The U. S. Environmental Protection Agency (EPA) reference method for TSP is codified at 40 CFR 50, Appendix B. This method uses a high-volume sampler to collect particles with aerodynamic diameters of approximately 100 μm or less. The high-volume samples 40-60 ft^3/min of air with the sampling rate held constant over the sampling period. The high-volume design causes the TSP to be deposited uniformly across the surface of a filter located downstream of the sampler inlet. The TSP high-volume can be used to determine the average ambient TSP concentration over the sampling period, and the collected material subsequently can be analyzed to determine the identity and quantity of inorganic metals present in the TSP.

1.3 Research on the health effects of TSP in ambient air has focused increasingly on particles that can be inhaled into the respiratory system, i.e., particles of aerodynamic diameter less than 10 μm . The health community generally recognizes that these particles may cause significant adverse health effects. Recent studies involving particle transport and transformation strongly suggest that atmospheric particles commonly occur in two distinct modes: the fine (< 2.5 μm) mode and the coarse (2.5-10.0 μm) mode. The fine or accumulation mode (also termed the respirable particulate matter) is attributed to growth of particles from the gas phase and subsequent agglomeration, while the coarse mode is made of mechanically abraded or ground particles. Particles that have grown from the gas phase (either because of condensation, transformation, or combustion) occur initially as very fine nuclei--0.05 μm . These particles tend to grow rapidly to accumulation mode particles around 0.5 μm which are relatively stable in the air. Because of their initially gaseous origin, particle sizes in this range include inorganic ions such as sulfate, nitrate, ammonia, combustion-form carbon, organic aerosols, metals, and other combustion products. Coarse particles, on the other hand, are produced mainly by mechanical forces such as crushing and abrasion. Coarse particles, therefore, normally consist of finely divided minerals such as oxides of aluminum, silicon, iron, calcium, and potassium. Coarse particles of soil or dust mostly result from entrainment by the motion of air or from other mechanical action within their area. Since the size of these particles is normally > 2.5 μm , their retention time in the air parcel is shorter than the fine particle fraction.

1.4 On July 1, 1987, the U. S. Environmental Protection Agency (EPA) promulgated a new size-specific air quality standard for ambient particulate matter. This new primary standard applies only to particles with aerodynamic diameters \leq 10 micrometers (PM₁₀) and replaces the original standard for TSP. To measure concentrations of these particles, the EPA also promulgated a new federal reference method (FRM). This method is based on the separation and removal of non-PM₁₀ particles from an air sample, followed by filtration and gravimetric analysis of PM₁₀ mass on the filter substrate.

1.5 The new primary standard (adopted to protect human health) limits PM_{10} concentrations to $150 \mu\text{g}/\text{std. m}^3$ averaged over a 24-h period. These smaller particles are able to reach the lower regions of the human respiratory tract and, therefore, are responsible for most of the adverse health effects associated with suspended particulate pollution. The secondary standard, used to assess the impact of pollution on public welfare, has also been established at $150 \mu\text{g}/\text{std. m}^3$.

1.6 Ambient air SPM measurements are used (among other purposes) to determine whether defined geographical areas are in attainment or non-attainment with the national ambient air quality standards (NAAQS) for PM_{10} . These measurements are obtained by the States in their State and local air monitoring station (SLAMS) networks as required under 40 CFR Part 58. Further, Appendix C of Part 58 requires that the ambient air monitoring methods used in these EPA-required SLAMS networks must be methods that have been designated by the EPA as either reference or equivalent methods.

1.7 Monitoring methods for particulate matter are designated by the EPA as reference or equivalent methods under the provisions of 40 CFR Part 53, which was amended in 1987 to add specific requirements for PM_{10} methods. Part 53 sets forth functional specifications and other requirements that reference and equivalent methods for each criteria pollutant must meet and explicit test procedures by which candidate methods or samplers are to be tested against those specifications. General requirements and provisions for reference and equivalent methods are also given in Part 53, as are the requirements for submitting an application to the EPA for a reference or equivalent method determination.

1.8 Several methods are available for measuring SPM in ambient air. As mentioned earlier, the most commonly used device is the high-volume sampler, which consists essentially of a blower and a filter, and which is usually operated in a standard shelter to collect a 24-h sample. The sample is weighed to determine concentration and may be analyzed chemically. The high volume sampler is considered a reliable instrument for measuring the mass concentration of TSP in ambient air. When EPA first regulated TSP, the NAAQS was stated in terms of SPM captured on a filter with aerodynamic particle size of $< 100 \mu\text{m}$ as defined by the TSP sampler; therefore, the TSP sampler was used as the reference method.

1.9 Under Part 53 requirements, reference methods for PM_{10} must be shown to use the measurement principle and meet other specifications set forth in 40 CFR 50, Appendix J. They must also include a PM_{10} sampler that meets the requirements specified in Subpart D of 40 CFR 53. Appendix J specifies a measurement principle based on extracting an air sample from the atmosphere with a powered sampler that incorporates the inertial separation of PM_{10} size range particles followed by the collection of PM_{10} particles on a filter over a 24 h period. The average PM_{10} concentration for the sample period is determined by dividing the net weight gain of the filter over the sample period by the total volume of air sampled, corrected to EPA's standard temperature (25EC) and standard pressure (760 mm Hg). Other specifications for flow rate control and measurement, flow rate measurement device calibration, filter media characteristics and performance, filter conditioning before and after sampling, filter weighing, sampler operation, and correction of sample volume to EPA reference temperature and pressure are prescribed in Appendix J. In addition, sampler performance requirements in Subpart D of Part 53 include sampling effectiveness (the accuracy of the PM_{10} particle size separation capability) at each of the three wind speeds and at "50% cutpoint" (the primary measure of 10- μm particle size separation). Field tests for sampling precision and flow rate stability are also specified. In spite of the instrumental nature of the sampler, this method is basically a manual procedure, and all designated reference methods for PM_{10} are therefore defined as manual methods.

1.10 This document describes the procedures for sampling SPM in ambient air for both TSP and PM₁₀ based upon active sampling using a high volume air sampler. The ambient particles are collected on quartz fiber filters. The sampler collects TSP or PM₁₀ ambient particles depending upon the type of inlet selected.

2. Applicable Documents

2.1 ASTM Documents

- D4096 *Application of the High Volume Sample Method for Collection and Mass Determination of Airborne Particulate Matter.*
- D1356 *Definition of Terms Related to Atmospheric Sampling and Analysis.*
- D1357 *Practice for Planning the Sampling of the Ambient Atmosphere.*
- D2986 *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test.*

2.2 Other Documents

- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: A Field Guide for Environmental Quality Assurance*, EPA/600/R-94/038a.
- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition)*, EPA/600/R-94/038b.
- *Reference Method for the Determination of Particulate Matter in the Atmosphere*, 40 CFR 50, Appendix J.
- *Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)*, 40 CFR 50, Appendix B.
- *Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air*, 40 CFR 50, Appendix G.
- *Reference Method for this Determination of Suspended Particulates in the Atmosphere (PM₁₀ Method)*, 40 CFR 50, Appendix J.

3. Summary of Method

3.1 The sampling of a large volume of atmosphere, 1,600-2,400 m³ (57,000-86,000 ft³), with a high-volume blower, typically at a rate of 1.13-1.70 m³/min (40-60 ft³/min), is described in this method. The calibration and operation of typical equipment used in this sampling is also described.

3.2 Air is drawn into the sampler and through a glass fiber or quartz filter by means of a blower, so that particulate material collects on the filter surface. Without a 10 µm size-selective inlet, particles of 100 µm size and less enter the sampling inlet and are collected on the downstream filter. The collection efficiencies for particles larger than 20 µm decreases with increasing particle size, and it varies widely with the angle of the wind with respect to the roof ridge of the sampler shelter. When glass fiber filters are used, particles 100-0.1 µm or less in diameters are ordinarily collected. With a size-select inlet, particles 10 µm diameter or less are collected on the quartz filter.

3.3 The upper limit of mass loading is determined by plugging the filter medium with sample material, which causes a significant decrease in flow rate. For very dusty atmospheres, shorter sampling periods will be necessary.

3.4 The volume of air sampled is determined by a flow-rate indicator. The instrument flow-rate indicator is calibrated against a reference orifice meter. The latter is a working standard which, in turn, has been calibrated against a master flow meter certified by the National Institute of Standards and Technology (NIST).

3.5 Airborne particulate matter retained on the filter may be examined or analyzed chemically by a variety of methods (ICP, ICP/MS, AA, GFAA, and NAA) as delineated in Inorganic Compendium Methods IO-3.2 through IO-3.7.

4. Significance

4.1 The area of toxic air pollutants has been the subject of interest and concern for many years. Recently the use of receptor models has resolved the elemental composition of atmospheric aerosol into components related to emission sources. The assessment of human health impacts resulting in major decisions on control actions by federal, state and local governments is based on these data. Accurate measures of toxic air pollutants at trace levels is essential to proper assessments.

4.2 The high volume sampler is commonly used to collect the airborne particulate component of the atmosphere. A variety of options available for the sampler provides broad versatility and allows the user to develop information about the size and quantity of airborne particulate material and, using subsequent chemical analytical techniques, information about the chemical properties of the particulate matter. The advent of inductively coupled plasma spectroscopy has improved the speed and performance of metals analysis in many applications.

5. Definitions

[Note: Definitions used in this document are consistent with those used in ASTM Methods. All pertinent abbreviations and symbols are defined within this document at point of use.]

5.1 High-Volume Air Sampler (HV). A device for sampling large volumes of an atmosphere for collecting the contained particulate matter by filtration. Consists of a high-capacity blower, a filter to collect suspended particles, and a means for measuring the flow rate.

5.2 Working Flow-Rate Standard. A flow-rate measuring device, such as a standard orifice meter, that has been calibrated against a master flow-rate standard. The working flow-rate standard is used to calibrate a flow measuring or flow rate indicating instrument.

5.3 Master Flow-Rate Standard. A flow-rate measuring device, such as a standard orifice meter, that has been calibrated against a primary standard.

5.4 Primary Flow-Rate Standard. A device or means of measuring flow rate based on direct primary observations such as time and physical dimensions.

5.5 Spirometer. A displacement gasometer consisting of an inverted bell resting upon or sealed by liquid (or other means) and capable of showing the amount of gas added to or withdrawn from the bell by the displacement (rise or fall) of the bell.

5.6 Absolute Filter. A filter or filter medium of ultra-high collection efficiency for very small particles (submicrometer size) so that essentially all particles of interest or of concern are collected. Commonly, the efficiency is 99.95% or higher for a standard aerosol of 0.3 μm diameter.

5.7 Calibration. The process of comparing a standard or instrument with one of greater accuracy (small uncertainty) to obtain quantitative estimates of the actual values of the standard being calibrated, the deviation of the actual value from a nominal value, or the difference between the value indicated by an instrument and the actual value.

5.8 Differential Pressure Meter. Any flow measuring device that operates by restricting air flow and measuring the pressure drop across the restriction.

5.9 Emissions. The total of substances discharged into the air from a stack, vent, or other discrete source.

5.10 Flowmeter. An instrument for measuring the rate of flow of a fluid moving through a pipe or duct system. The instrument is calibrated to give volume or mass rate of flow.

5.11 Impaction. A forcible contact of particles of matter. A term often used synonymously with impingement.

5.12 Impactor. A sampling device that employs the principle of impaction (impingement).

5.13 Impingement. The act of bringing matter forcibly in contact. As used in air sampling, refers to a process for the collection of particulate matter in which the gas being sampled is directed forcibly against a surface.

5.14 Inhalable Particles. Particles with aerodynamic diameters of $< 10 \mu\text{m}$ that are capable of being inhaled into the human lung.

5.15 Interference. An undesired positive or negative output caused by a substance other than the one being measured.

5.16 Mass Flowmeter. Device that measures the mass flow rate of air passing a point, usually using the rate of cooling or heat transfer from a heated probe.

5.17 Matter. The substance of which a physical object is composed.

5.18 Orifice Meter. A flowmeter, employing as the measure of flow rate the difference between the pressures measured on the upstream and downstream sides of the orifice (that is, the pressure differential across the orifice) in the conveying pipe or duct.

5.19 Aerodynamic Diameter (a.d.). The diameter of a unit density sphere having the same terminal settling velocity as the particle in question. Operationally, the size of a particle as measured by an inertial device.

5.20 Particle. A small discrete mass of solid or liquid matter.

5.21 Particulate. Solids or liquids existing in the form of separate particles.

5.22 Precision. The degree of mutual agreement between individual measurements, namely repeatability and reproducibility.

5.23 Pressure Gage. The difference in pressure existing within a system and that of the atmosphere. Zero gage pressure is equal to atmospheric pressure.

5.24 Rotameter. A device, based on the principle of Stoke's law, for measuring rate of fluid flow. It consists of a tapered vertical tube having a circular cross section, and containing a float that is free to move in a vertical path to a height dependent upon the rate of fluid flow upward through the tube.

5.25 Sampling. A process consisting of the withdrawal or isolation of a fractional part of a whole. In air or gas analysis, the separation of a portion of an ambient atmosphere with or without the simultaneous isolation of selected components.

5.26 Standard. A concept that has been established by authority, custom, or agreement to serve as a model or rule in the measurement of quantity of the establishment of a practice or procedure.

5.27 Traceability to NIST. Documented procedure by which a standard is related to a more reliable standard verified by the National Institute of Standards Technology (NIST).

5.28 Uncertainty. An allowance assigned to a measured value to take into account two major components of error: The systematic error and the random error attributed to the imprecision of the measurement process.

6. Apparatus Description

6.1 General Description

6.1.1 The essential features of a typical non size-specific TSP high-volume sampler are shown in Figure 1. The high volume sampler is a compact unit consisting of a protective housing; an electric motor driven; a high-speed, high-volume blower; a filter holder capable of supporting a 203 x 254-mm (8 in. by 10 in.) filter; and a flow-controller for controlling the air-flow rate through the instrument at 40-60 ft³/min.

6.1.2 In operation, this traditional TSP sampler draws ambient air into the sampler through the air inlet gap between the cover and the sampler housing walls (see Figure 2). The air inlet is uniform on all sides of the sampler to provide an effective particle capture air velocity between 20-35 cm/sec. at the recommended flow rate between 40-60 ft³/min. The gable roof design of the sampler allows the sampled air to be evenly distributed over the surface of a downstream filter, where TSP is collected.

6.1.3 For PM₁₀ measurement, the traditional gable roof of the TSP sampler is replaced with an impactor design size-select inlet, as illustrated in Figure 3. For the impactation design, an air sample enters a symmetrical (therefore wind-direction insensitive) hood and is deflected upward into a buffer chamber. The buffer chamber is evacuated at a rate of 68 m³/h (40 cfm) through multiple circular nozzles. Particles are accelerated as they pass through the nozzles to an impactation chamber (see Figure 4). Because of their momentum, particles having diameters larger than the inlet's 10- μ m cut design impact the surface of the

impaction chamber. Smaller particles rise through the impaction chamber at speeds slow enough to minimize reentrainment of the impacted particles and then pass through multiple vent tubes to the high-volume sampler's filter where they are collected.

6.1.4 The second size-select design for PM₁₀ measurement is the cyclone inlet, as illustrated in Figure 5. The omnidirectional cyclone used for fractionation in this inlet allows particles to enter from all angles of approach. An angular velocity component is imparted to the sample air stream and the particles contained in it by a series of evenly spaced vanes. Larger particle removal occurs in an inner collection tube. This tube incorporates a "perfect absorber," which is an oil-coated surface to eliminate particle bounce and reentrainment. The sample flow (with the unremoved smaller particles) then enters an intermediate tube, where the trajectory is altered to an upward direction. An additional turn is then made to alter the flow to a downward trajectory to allow the remaining particles (i.e., PM₁₀ fraction) to deposit on a filter for subsequent analysis. As with the impaction inlet, control of air velocities in the cyclonic inlet is critical to maintain the correct particle size cutpoint. Maintaining the correct design volumetric flow rate through the inlet is important. This design flow rate is specified by the manufacturer in the instruction manual. For example, a popular cyclonic impaction inlet has a design flow rate of 1.13 m³/min.

6.2 Filter Medium

6.2.1 Selecting a filtration substrate for time-integrated SPM monitoring must be made with some knowledge of the expected characteristics and a pre-determined analytical protocol. For any given standard test method, the appropriate medium will normally be specified.

6.2.2 Of the various types of filters listed in Table 1 of Chapter IO-2 Overview, four general types of filter material have been used to capture SPM. They include cellulose fiber, quartz/glass fiber, mixed fiber and membrane filter types. Selecting a filter depends upon variables such as background metal content, artifact formation, and affinity for moisture. The basic characteristics of the types of filter material used in air monitoring are outlined in Table 1, while useful filter properties are identified in Table 2. Several characteristics are important in selection of filter media. They are:

- **Particle Sampling Efficiency.** Filters should remove more than 99% of SPM from the air drawn through them, regardless of particle size or flow rate.
- **Mechanical Stability.** Filters should be strong enough to minimize leaks and wear during handling.
- **Chemical Stability.** Filters should not chemically react with the trapped SPM.
- **Temperature Stability.** Filters should retain their porosity and structure during sampling.
- **Blank Correction.** Filters should not contain high concentrations of target compound analytes.

6.2.3 Quartz fiber filters are the most commonly used filters for SPM sampling for determining mass loading. Typical characteristics of quartz fiber filters are (1) a fiber content of high purity quartz, (2) a binder of below 5% (zero for binderless types), (3) a thickness of approximately 0.5 mm, (4) a surface with no pinholes, and (5) an allowance of no more than 0.05% of smoke particles to pass through the filter at a pressure of 100 mm of water with a flow rate of 8.53 m/min (28 ft/min), as determined by a DOP smoke test (see ASTM Method D2986).

6.2.4 Quartz fiber filters are made from finely spun glass fiber by combining the fiber with an organic binder and compressing this material in a paper machine. These filters are increasingly used in air sampling. These filters have the ability to withstand high temperatures (up to 540EC). They are further typified by high-collection efficiency. In some cases, the organic binder may interfere with subsequent analysis, so the filter is flash-fired to remove the binder material. This action causes some loss in tensile strength and usually requires that a backing material be used during sampling. The quartz filters are nonhygroscopic, thus allowing them to be used in areas where humidity is high. Because they are glass, they are the filter choice for most corrosive atmospheres. All the filters in this category are fragile and must be handled with care. Quartz fiber filters, because of the high silicate content, are extremely difficult to ash by chemicals or heat.

Therefore, extraction procedures are performed on these filters to remove the sample for subsequent chemical analysis. For this reason, flash-fired quartz filters are the major atmospheric sampling filters.

6.3 Flow Control System

The high-volume sampler employs two basic types of flow control systems. One is a mass-flow-control (MFC) system; the other is a volumetric-flow-control (VFC) system. Because the calibration and standard operating procedures differ considerably between these two types of flow-control systems, this method presents procedures that are control-system-specific. PM₁₀ inlets can be used with either the MFC and VFC systems.

6.3.1 Mass-flow-control (MFC) system. The flow rate in a MFC system is actively sensed and controlled at a predetermined set point. Air is pulled through the filter into the intake of a blower and subsequently exits the sampler through an exit orifice, which facilitates measurement of the flow with a manometer or pressure recorder. The flow rate is controlled by an electronic mass-flow controller, which uses a flow sensor installed below the filter holder to monitor the mass flow rate and related electronic circuitry to control the speed of the motor accordingly to provide a constant sampling rate. The controlled flow rate can be changed by an adjustment knob on the flow controller.

6.3.2 Volumetric-flow-control (VFC) system. A VFC system maintains a constant volumetric flow rate through the inlet, rather than a constant mass flow rate as in the MFC system. In a popular commercial VFC system, a choked-flow venturi is operated such that the air attains sonic velocity in the throat of the device. In this "choked" mode, the flow rate is unaffected by downstream conditions, such as motor speed or exit pressure and is a predictable function of upstream conditions, such as the stagnation pressure ratio and temperature. Thus, the volumetric flow is controlled without any moving parts or electronic components. In this type of flow control system, no means is provided for adjusting the controlled flow rate. The controlled flow rate is set by the manufacturer through engineering design of the venturi.

7. Calibration

7.1 Introduction

[Note: All sampling equipment must be properly calibrated. Calibration is the relationship between an instrumental output and the input of a known reference standard. The objective of this section is to provide technically sound flow-rate calibration procedures for the MFC and VFC HV samplers.]

[Note: Consistency of temperature and barometric pressure is required. All temperatures should be expressed in kelvin ($K = EC + 273$). All barometric pressures should be expressed in mm Hg. Avoid calibrating an HV sampler using one set of units and then performing sample calculations using another set.]

7.1.1 HV sampler inlet. Two types of size-selective inlets available are impaction and cyclonic for monitoring inhalable particles ($< 10 \mu\text{m}$). The particle size discrimination characteristics of both the impaction and cyclonic type inlets depend critically on maintaining certain air velocities within the inlet; a change in velocity will result in a change in the nominal particle size collected. For this reason, the flow rate through the inlet must be maintained at a constant value that is as close as possible to the inlet's design flow rate. The design flow rate for a given sampler is specified in the sampler's instruction manual. The manual may also provide tolerance limits (or upper and lower limits) within which the sampler flow must be maintained. If the tolerance is not specified by the manufacturer, it should be assumed to be $\pm 10\%$.

7.1.1.1 The symmetrical design of the impaction inlet (see Figure 4) ensures wind-direction insensitivity. Ambient air that is drawn into the inlet is evacuated from the buffer chamber through nine acceleration nozzles into the first impaction chamber, where initial particle separation occurs. The air is then accelerated through an additional 16 jets into a second impaction chamber. The acceleration jets have critical diameters calculated by the manufacturer to provide the necessary changes in velocity to effect correct particle size fractionation within the impaction chambers. The air flow finally exits the inlet through nine vent tubes onto a sample filter. Because air velocities are critical to maintain the correct particle size cutpoint within the inlet, maintaining the correct design flow rate through the inlet is important. This design flow rate is specified by the manufacturer in the instruction manual. For example, the design flow rate for one popular impaction inlet is 1.13 m³/min.

7.1.1.2 The omnidirectional cyclone inlet (see Figure 5) used for fractionation allows particles to enter from all angles of approach. A angular velocity component is imparted to the sample air stream and the particles contained in it by a series of evenly spaced vanes. Larger particle removal occurs in an inner collection tube. This tube incorporates a "perfect absorber," an oil-coated surface to eliminate particle bounce and reentrainment. The sample flow (with the unremoved smaller particles) then enters an intermediate tube, where the trajectory is altered to an upward direction. An additional turn is then made to alter the flow to a downward trajectory to allow the remaining particles (i.e., PM₁₀ fraction) ultimately to deposit on a filter for subsequent analysis. As with the impaction inlet, control of air velocities in the cyclonic inlet is critical to maintain the correct particle size cutpoint. Maintaining the correct design volumetric flow rate through the inlet is important. This design flow rate is specified by the manufacturer in the instruction manual. For example, as in the case of the impaction inlet, a popular cyclonic inlet also has a design flow rate of 1.13 m³/min.

7.1.2 Total suspended particulate (TSP). As illustrated in Figure 2, particles of less than 100 µm are collected at a flow rate of 1.13-1.70 m³/min (40-60 ft³/min) using the conventional high-volume sampler, without size selection.

7.2 Summary of Calibration Procedures

[Note: During calibration, a closure plate perforated with a number of circular orifices is connected to the inlet of the sampler. The pressure drop across this orifice plate provides a measure of instrument air flow rate at any time. This pressure drop may be indicated by a rotameter, manometer, or other pressure-responsive device traceable to an NIST certified standard.]

7.2.1 A simple and sufficiently accurate method of calibrating is to compare the sampler meter with an orifice meter (working standard) that has been calibrated against a primary or master standard such as a Roots meter.

7.2.2 The preferable primary standard is a Roots meter of sufficient capacity to allow an accurate time-volume reading, which would be at least 30 s.

7.2.3 A positive displacement pump or blower may be used as a master flow-rate standard. In this case, the delivery rate of the master standard must be known accurately and the equipment must be in sound mechanical condition (no bypass leakage).

7.3 Certification of an Orifice Transfer Standard

[Note: The following certification procedure is applicable to an orifice transfer standard such as those that have been used previously in the calibration of both the traditional HV sampler and the PM₁₀ samplers. Two common types of orifice devices are available: one equipped with a set of fixed resistance plates (e.g., a reference flow [Ref] device or a top-hat orifice) and one with an externally variable resistance valve. The

series of plates normally provided by the orifice manufacturer include an 18-, 13-, 10-, 7-, and 5-hole plate. Unfortunately, the 5-hole plate provides too low a flow rate to be useful for HV calibration, and other plates may produce flow rates substantially outside the design flow-rate range of the commercially available HV inlets. One may opt to fabricate or procure a different series of resistance ranges or use the variable-resistance type orifice device.]

7.3.1 Orifice Calibration Procedure.

7.3.1.1 Assemble the following equipment (see Figure 6):

- Orifice transfer standard (i.e., top-hat orifice, variable orifice, or ReF device) to be calibrated.
- Water or oil manometer with a 0-400 mm (0-16") range and minimum scale divisions of 1 mm (0.1"). This manometer should be permanently associated with the orifice transfer standard.
- Variable voltage transformer, a set of resistance plates, or available flow orifice (see Figure 7).
- Calibrated positive displacement, standard volume meter (such as a Roots Meter®) traceable to National Institute of Standards and Technology (NIST).

[Note: As they are sold, standard volume meters may not be traceable to NIST. Traceability can be established directly through NIST or indirectly through the meter manufacturer's repair department. Periodic recertification is not normally required under clean service conditions unless the meter has been damaged and must be repaired. In general, damage will be indicated by a substantial (e.g., 50%) increase in the pressure drop across the meter. The meter's traceability certificate should contain a graph of the pressure drop as a function of flow rate.]

- High-volume air mover (e.g., a blower motor from a HV sampler).
- Accurate stopwatch.
- Mercury manometer, with a 0-200 mm (0-8") range and minimum scale divisions of 1 mm (0.1").
- Thermometer, capable of accurately measuring temperatures over the range of 0-50EC (273-323 K) to the nearest ± 1 EC and referenced to an NIST or ASTM thermometer within ± 2 EC at least annually.
- Barometer, capable of accurately measuring ambient barometric pressure over the range of 500-800 mm Hg (66-106 kPa) to the nearest mm Hg and reference within ± 5 mm Hg of a barometer of known accuracy at least annually.
- Orifice transfer standard certification worksheet (see Figure 8).

7.3.1.2 Record on the orifice transfer standard certification worksheet the standard volume meter's serial number; orifice transfer standard's type, model, and serial number; the person performing the certification; and the date.

7.3.1.3 Observe the barometric pressure and record it as Pa. Read the ambient temperature in the vicinity of the standard volume meter and record it as T_a ($K = EC + 273$).

7.3.1.4 Connect the orifice transfer standard to the inlet of the standard volume meter. Connect the mercury manometer to measure the pressure at the inlet of the standard volume meter. Connect the orifice (water or oil) manometer to the pressure tap on the orifice transfer standard. Connect a high-volume air mover to the outlet side of the standard volume meter. Make sure that all gaskets are present and are in good condition.

7.3.1.5 Check that the standard volume meter table is level and adjust its legs if necessary.

7.3.1.6 Check for leaks by temporarily clamping both manometer lines (to avoid fluid loss) and blocking the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Start the high-volume air mover and note any change in the standard volume meter's reading. The reading should

remain constant. If the reading changes, locate any leaks by listening for a whistling sound and/or retightening all connections, making sure that all gaskets are properly installed.

[Note: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime; it can raise temperatures to the point of defeating the electrical insulation which could result in fire or electric shock to the user.]

7.3.1.7 After satisfactorily completing the leak check, turn off the high-volume air sampler, unblock the orifice, and unclamp both manometer lines. Zero the water and mercury manometers by sliding their scales so that their zero lines are even with the bottom of the menisci.

7.3.1.8 Turn on the high-volume air sampler. Adjust the variable voltage transformer to achieve an appropriate flow rate (i.e., within the approximate range of 0.9-1.3 m³/min (32-46 ft³/min)). If necessary, use fixed resistance plates or the variable resistance valve to achieve the appropriate flow rate (see Figure 7). The use of fixed resistance plates is discouraged (but not prohibited) because the leak check must be repeated each time that a plate is installed.

7.3.1.9 After setting a flow rate, allow the system to run for at least 1 min to attain a constant motor speed. Observe the standard volume meter dial reading and simultaneously start the stopwatch. Error in reading the meter dial can be minimized by starting and stopping the stopwatch on whole number dial readings (e.g., 4091.00).

7.3.1.10 Record the initial volume that the meter dial indicated when the stopwatch was started. Maintain this constant flow rate until at least 3 m³ of air have passed through the standard volume meter. Record the standard volume meter's inlet pressure manometer reading as) Hg and the orifice manometer reading as) H₂O. If) H₂O changes significantly during the run, abort the run and start again.

7.3.1.11 When at least 3 m³ of air have passed through the system, note the standard volume meter reading and simultaneously stop the stopwatch. Record the final volume that the meter dial was indicating when the stopwatch was stopped. Record the elapsed time (Time) indicated on the stopwatch.

7.3.1.12 Calculate the volume measured by the standard volume meter (Vol.) using the following equation:

$$\text{) Vol.} = \text{Final Volume} - \text{Initial Volume}$$

7.3.1.13 Correct this volume to ambient atmosphere pressure.

$$V_a = \text{) Vol.} (\text{Pa} - \text{) Hg}) / \text{Pa}$$

where:

- V_a = actual volume at ambient barometric pressure, m³.
-) Vol. = actual volume measured by the standard volume meter, m³.
- Pa = ambient barometric pressure during calibration, mm Hg.
-) Hg = differential pressure at inlet to volume meter, mm Hg.

7.3.1.14 Calculate the actual volumetric flow rate (m³/min).

$$Q_a = V_a / \text{Time}$$

where:

- Qa = actual volumetric flow rate through the orifice, m³/min.
) time = elapsed time, min.

7.3.1.15 Repeat Sections 7.3.1.8 through 7.3.1.14 for at least four additional flow rates within the approximate range of 0.9-1.3 m³/min (32-46 ft³/min). At least five evenly distributed different flow rates are required, and at least three flow rates must be in the specified inlet flow-rate interval (1.02-1.24 m³/min [36-44 ft³/min]). Better calibration precision may be obtained by running additional flow rates or repeating the flow rates.

7.3.1.16 For each flow, compute $[(\text{H}_2\text{O})(T_a/P_a)]^{1/2}$, and plot these values against the corresponding values of Qa. Draw the orifice transfer standard's certification curve. For the model $[(\text{H}_2\text{O})(T_a/P_a)]^{1/2} = m(Qa) + b$, calculate the linear least squares regression's slope (m), intercept (b), and correlation coefficient (r) of the certification relationship. Plot the regression line on the same graph as the calibration data, as illustrated in Figure 9. A certification graph should be readable to 0.02 m³/min.

7.3.1.17 If any calibration point does not fall within $\pm 2\%$ of the line, rerun the point, recalculate, and replot.

7.3.1.18 For subsequent use of the orifice transfer standard, calculate Qa from the calibration relationship as:

$$Qa(\text{orifice}) = \{[(\text{H}_2\text{O})(T_a/P_a)]^{1/2} - b\} \{1/m\}$$

where:

- Qa(orifice) = actual volumetric flow rate as indicated by the orifice transfer standard, m³/min
) H₂O = pressure drop across the orifice, mm H₂O.
Ta = ambient temperature during use, K (K = EC + 273).
b = intercept of the orifice calibration relationship.
m = slope of the orifice calibration relationship.

7.3.2 Orifice Transfer Standard Calibration Frequency. Upon receipt and at 1-yr intervals, the calibration of the orifice transfer standard should be certified with a standard volume meter (such as a Roots Meter®) traceable to NIST. An orifice transfer standard should be visually inspected for signs of damage before each use and should be recalibrated if the inspection reveals any nicks or dents.

7.4 Procedure for a Mass-Flow-Controlled (MFC) High Volume Sampler

The MFC sampler calibration procedure presented in this section relates known flow rates to the pressure in the exit orifice plenum. The known flow rates are determined by an orifice transfer standard that has been certified according to the procedure presented in Section 7.3.1. The exit orifice plenum is the area within the motor housing (below the motor unit) that contains the air flow just before it is exhausted to the atmosphere through the exit orifice. This exit orifice plenum pressure should be measured with a 25-cm (10") water or oil manometer. Also, each sampler should have its own dedicated manometer, which can be conveniently mounted to the side of the sampler housing. Other types of pressure measurement devices may be used provided they have comparable accuracy. The 4" continuous pressure (flow) recorders of the type often supplied with high volume PM₁₀ samplers are generally not sufficiently accurate and are not recommended for quantitative sampler pressure or flow measurements. These flow recorders should be used only for nonquantitative determination that the flow was approximately constant and uninterrupted over the sample period. The flow recorder may be connected in parallel with the manometer or other pressure

measuring device, using a tee or "y" tubing connection. For this MFC calibration procedure, the following conditions are assumed:

- The high volume PM₁₀ sampler is equipped with a mass flow controller to control its sample flow rate.
- The sampler flow rate is measured by measuring the exit orifice plenum pressure, using a water or oil manometer [or, if necessary, a continuous-flow recording device using square-root-scale chart paper].
- The transfer standard for the flow-rate calibration is an orifice device equipped with either a series of resistance plates or an integral variable-resistance valve. The pressure drop across the orifice is measured by an associated water or oil manometer.

[Note: Because flow recorders are still widely used for quantitative flow measurements, the calibration procedure includes specific instructions for quantitatively calibrating a flow recorder. These flow recorder instructions are enclosed in brackets [] and should be used only when a manometer or other pressure measurement device cannot be used.]

7.4.1 Calibration Equipment.

7.4.1.1 Orifice transfer standard with calibration traceable to NIST (see Section 7.3).

7.4.1.2 An associated water or oil manometer, with a 0-400 mm (0-16") range and an minimum scale division of 2 mm (0.1")

[Note: Digital manometers may also be used in place of water or oil manometers, especially in cold/frigate climates. Ensure the battery in the manometer is new before use.]

7.4.1.3 A water or oil manometer, with a 0-400 mm (0-16") range and a minimum scale division of 2 mm (0.1") for measurement of the sampler exit orifice plenum pressure. This manometer should be associated with the sampler.

[Note: Manometers used for field calibration may be subject to damage or malfunction and should thus be checked frequently.]

7.4.1.4 Thermometer, capable of accurately measuring temperature over the range of 0-50EC (273-323 K) to the nearest ± 1 EC and referenced to an NIST or ASTM thermometer within ± 2 EC at least annually.

7.4.1.5 A portable aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient barometric pressure over the range of 500-800 mm Hg (66-106 kPa) to the nearest mm Hg and referenced within ± 5 mm Hg of a barometer of known accuracy at least annually.

7.4.1.6 Miscellaneous handtools, calibration data sheets or station log book, and 51 mm (2") duct tape.

7.4.2 Multipoint Flow-Rate Calibration. The procedure presented here is basic and generic, given the assumptions listed in Section 7.4. More detailed calibration procedures, variations, or alternative procedures may be presented in the manufacturer's instruction manual. The manual should be reviewed carefully and the various calibration variations or alternative procedures should be evaluated. In-house equipment and personnel, procedural simplicity and uniformity, and subsequent data applications should be considered in establishing the specific, detailed calibration procedure to be implemented.

[Note: Do not attempt to calibrate the MFC sampler under windy conditions. Short-term wind velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The calibration will be less precise because of pressure variations.]

7.4.2.1 Set up the calibration system as recommended by the manufacturer. A typical MFC PM₁₀ sampler calibration configuration is illustrated in Figure 10. MFC samplers are calibrated without a filter or filter cassette installed.

7.4.2.2 Disconnect the motor from the flow controller and plug it directly into a stable line voltage source (i.e., the sampler's on-off timer, if so equipped, or other source of the line voltage).

7.4.2.3 Install the orifice transfer standard and its adapter faceplate on the sampler. Check all gaskets and replace any questionable ones.

[Note: Tighten the faceplate nuts evenly on alternate corners to properly align and seat the gaskets. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.]

7.4.2.4 Select the first calibration flow rate and install the appropriate resistance plate or adjust the variable orifice valve. At least four flow rates are required to define the calibration relationship. For resistance plate orifices, make sure that the orifice and resistance plate gaskets are in place and the orifice is not cross-threaded on the faceplate.

7.4.2.5 To leak check, block the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Seal the pressure port with a rubber cap or similar device. Turn on the sampler. Gently rock the orifice transfer standard and listen for a whistling sound that would indicate a leak in the system. A leak-free system will not produce an upscale response in the sampler's exit orifice manometer or flow recorder. Leaks are usually caused either by a damaged or missing gasket between the orifice transfer standard and the faceplate or by cross-threading of the orifice transfer standard on the faceplate. All leaks must be eliminated before proceeding with the calibration. When the system is determined to be leak-free, turn off the sampler and unblock the orifice.

[Note: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime and can raise temperatures to the point of defeating the electrical insulation, which could result in fire or electric shock to the user.]

7.4.2.6 Inspect the connecting tubing of both manometers for crimps or cracks. Open the manometer valves (if present) and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometers' sliding scales so that their zero lines are at the bottom of the menisci. Connect the orifice transfer standard manometer to the orifice transfer standard. Connect the sampler's exit orifice manometer [and the continuous-flow recorder, if used] to the exit orifice plenum port. Ensure that one side of each manometer is open to atmospheric pressure. Make sure that the tubing fits snugly on the pressure ports and on the manometer.

7.4.2.7 If a continuous flow recorder is to be used quantitatively in lieu of a manometer, record the site location, sampler S/N, date, and the operator's initials on the blank side of a clean recorder chart. Make sure the chart has a square-root scale. Open the front door of the sampler and install the clean recorder chart.

7.4.2.8 Read and record the following parameters on the HV data sheet. An example calibration data sheet for the MFC sampler is illustrated in Figure 11.

- Date, location, and operator's signature.
- Sampler S/N and model.
- Ambient Pa, mm Hg.
- Ambient temperature (T_a), K ($K = EC + 273$).
- Orifice S/N and calibration relationship.

[Note: Consistency of temperature and barometric pressure units is required. All temperatures should be expressed in kelvin ($K = EC + 273$). Also, all barometric pressures should be expressed in mm Hg. Avoid calibrating a sampler using one set of units and then performing sampler calculations using another set.]

[Note: Ideally, the temperature of the air in the exit orifice plenum should be measured because it will be somewhat higher than ambient temperature. However, an adequate approximation of this temperature may be obtained by adding 30 K to the ambient temperature. This addition is incorporated in the calculations given in Section 7.4.3.]

7.4.2.9 Turn on the sampler and allow it to warm up to operating temperature (3-5 min). Then read and record the orifice transfer standard's manometer deflection, ΔH_2O (in. H₂O), and the corresponding sampler's manometer deflection, ΔP_{ex} [or flow recorder chart reading, I].

[Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2" of clearance at the bottom for air flow and for the manometer tubing.]

7.4.2.10 Install the other resistance plates or adjust the variable orifice value to obtain each of the other calibration flow rates and repeat Section 7.4.2.9 for each. At least four calibration flow rates are required.

7.4.2.11 Plot the calibration data on a sheet of graph paper as specified in Section 7.4.3.4.

[Note: The data should be plotted in the field as the calibration is occurring, rather than afterwards back at the laboratory.]

Repeat Section 7.4.2.9 for any data that are questionable on the plot.

[Note: Running additional calibration points at differing flow rates or repeating the calibration points at the same flow rates is encouraged to improve the precision of the calibration.]

7.4.2.12 Turn off the sampler and remove the orifice transfer standard.

7.4.2.13 Reconnect the sampler motor to the flow controller.

7.4.2.14 Perform the calibration calculations presented in the following section. The data generated will be used to set the mass flow controller (see Section 7.4.4) to a value that will result in optimal volumetric flow based on the seasonal average temperature and barometric pressure at the monitoring site.

7.4.3 Calibration Calculations. Gather all calibration data, including the orifice calibration information and the sampler calibration data sheet (and, if used, the flow recorder chart, which should graphically display the various calibration flow rates).

[Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that have already been obtained.]

7.4.3.1 Verify that the orifice transfer standard calibration relationship is current and traceable to an acceptable primary standard.

7.4.3.2 Calculate and record Q_a for each calibration point from the orifice calibration information using the following equation.

$$Q_a(\text{orifice}) = \{ \} H_2O(T_a/P_a)^{1/2} - b \} \{1/m\} \text{ where:}$$

- Q_a(orifice) = actual volumetric flow rate as indicated by the transfer standard orifice, m³/min
-) H₂O = pressure drop across the orifice, in. H₂O.
- T_a = ambient temperature during use, K (K = EC + 273).
- P_a = ambient barometric pressure during use, mm Hg.
- b = intercept of the orifice calibration relationship.
- m = slope of the orifice calibration relationship.

7.4.3.3 Calculate and record the quantity for each calibration point as:

$$) P_{ext} = [] P_{ex}(T_a + 30)/P_a)^{1/2}$$

where:

-) P_{ext} = transformed manometer reading.
-) P_{ex} = sampler manometer reading, in. H₂O T_a = ambient temperature, K (K = EC + 273).
- P_a = ambient barometric pressure, mm Hg.

[If a continuous-flow recorder is used quantitatively, calculate and record the quantity [It] as follows:

$$[It] = I[T_a + 30)/P_a)^{1/2}$$

where:

- [It] = transformed flow recorder chart reading.
- I = flow recorder chart reading, arbitrary units on square root scale.

[Note: If recorder charts with linear scales are used, substitute (I)^{1/2} for I in the above equation.]

7.4.3.4 On a sheet of graph paper, plot the calculated Q_a(orifice) flow rates on the x-axis and the transformed sampler manometer response,) P_{ext} [or the transformed flow recorder reading, It], on the y-axis.

Because determining the sampler's average operational flow rate (Q_a) during a sample period depends on the ambient average temperature and pressure, using a graphic plot of the calibration relationship is not recommended for subsequent data reduction. This plot is used only to visually assess the calibration points to see if any should be rerun. Plot the regression line on the same graph paper as the calibration data. For the regression model y = mx + b, let y + 2) P_{ext} and x = Q_a(Orifice) so that the model is given by:

$$) P_{ext} = m[Q_a(\text{orifice})] + b$$

For the flow recorder, the model is:

$$It = m[Q_a(\text{orifice})] + b]$$

Using a programmable calculator or a calculation data form, determine the linear regression slope (m), intercept (b), and correlation coefficient (r) and record them on the data sheet. A five-point calibration should yield a regression equation with a correlation coefficient of r > 0.990, with no point deviating more

than $\pm 0.04 \text{ m}^3/\text{min}$ from the value predicted by the regression equation. Plot the regression line on the same graph paper that has the individual calibration points.

7.4.3.5 For subsequent sample periods, the sampler's average actual operational flow rate, \overline{Q}_a , is calculated from the calibration slope and intercept using the equation.

$$\overline{Q}_a = \{ \overline{P}_{ex} (T_{av} + 30) / P_{av} \}^{1/2} - b \} \{ l/m \}$$

where:

- \overline{Q}_a = the sampler's average actual flow rate, m^3/min .
- \overline{P}_{ex} = average of initial and final sampler manometer readings $(P_{ex_i} + P_{ex_f}) / 2$, mm Hg.
- T_{av} = average ambient temperature for the sample period, K ($K = EC + 273$).
- P_{av} = average ambient pressure for the sampling period, mm Hg.
- b = intercept of the sampler calibration relationship.
- m = slope of the sampler calibration relationship.

[For the flow controller,

$$\overline{Q}_a = \{ \overline{I} (T_{av} + 30) / P_{av} \}^{1/2} - b \} \{ l/m \}$$

where:

- \overline{I} = average flow recorder reading for the sample period.]

[*Note: If recorder charts with linear scales are used, substitute $(I)^{1/2}$ for (I) in the above equation.*]

7.4.4 Mass Flow Controller Adjustment Procedure. The controlled flow rate of an MFC sampler is adjustable and must be set to the proper design flow rate. The constant mass flow maintained by the MFC causes the actual volumetric flow rate through the inlet to fluctuate as the ambient temperature and barometric pressure change at the monitoring site. Normally, the range of these fluctuations is within the allowable tolerance limits for the inlet. However, the flow-rate set point of the mass flow controller must be correctly adjusted so that the deviations are "centered" with respect to the seasonal average temperature and barometric pressure at the site, not the temperature and pressure prevailing at the time of setting. The correct seasonal volumetric setpoint flow rate (SFR) at T_a and P_a has had the same mass flow rate as the inlet design volumetric flow rate at T_s and P_s .

[*Note: The correct SFR may differ from day to day and may be somewhat higher or lower than the inlet design flow rate on any particular day.*]

7.4.4.1 Determine the seasonal average temperature (T_s) and seasonal average pressure (P_s) at the site and record them on the calibration data sheet. (Determination of the number of "seasons," i.e., the number of different seasonal average temperatures needed for the year, is left to the discretion of the operator.)

7.4.4.2 Calculate SFR and record on the calibration data sheet:

$$\text{SFR} = (1.13) (\text{Ps}/\text{Pa})(\text{Ta}/\text{Ts})$$

where:

- SFR = set-point actual volumetric flow rate for adjustment of the mass flow controller, based on seasonal average temperature and average pressure at site, m³/min.
1.13 = inlet design flow rate (as specified by the manufacturer), m³/min.
Ps, Pa = seasonal average and current ambient barometric pressure at the site, respectively, mm Hg.
Ts, Ta = seasonal average and current ambient temperature, respectively, K (K = EC + 273).

7.4.4.3 Calculate and record on the sampler's calibration data sheet the sampler set-point manometer reading [or flow recorder reading] that corresponds to the SFR calculated in Section 7.4.4.2.

$$\text{SSP} = [\text{Pa}/(\text{Ta} + 30)][\text{m}(\text{SFR}) + \text{b}]^2$$

where:

- SSP = sampler set-point manometer reading, in H₂O.
Pa = ambient barometric pressure, mm Hg.
Ta = ambient temperature, K (K = EC + 273).
m = slope of the sampler's calibration relationship.
SFR = set-point flow rate from 7.4.4.2, m³/min.
b = intercept of the sampler's calibration relationship.
[For the flow recorder,

$$\text{SSP} = [\text{m}(\text{SFR}) + \text{b}] [\text{Pa}/(\text{Ta} + 30)]^{1/2}$$

7.4.4.4 Visually check to make sure the motor is connected to the mass flow controller and the manometer is properly connected.

7.4.4.5 Install a clean filter (in a filter cassette) in the sampler according to the manufacturer's instructions. [If the continuous flow recorder is used quantitatively, install a clean chart and verify that the recorder is zeroed (i.e., the pen rests on the innermost circle of the chart).]

7.4.4.6 Turn on the sampler and allow it to warm up to operating temperature (3-5 min).

7.4.4.7 Following the manufacturer's instructions, adjust the mass flow controller until the manometer reading [or flow recorder response] indicates the sampler set point (SSP) as calculated in Section 7.4.4.3.

7.4.4.8 Verify that the flow controller will maintain this flow rate for at least 10 min. Turn off the sampler.

7.4.4.9 The sampler can now be prepared for the next sample run day.

7.5 Procedure for a Volumetric-Flow-Controlled (VFC) Sampler

The VFC sampler calibration procedure presented in this section relates known flow rates (Qa, as determined by an orifice transfer standard) to the ratio of the stagnation pressure to the ambient barometric pressure (PI/Pa). The stagnation pressure (PI) is the air pressure inside the sampler in the area just under the filter. VFC samplers have a stagnation pressure tap or port through which the stagnation pressure can be measured. A VFC sampler may also have an exit orifice below the motor similar to those in MFC samplers. In this case, the sampler flow rate can be measured and calibrated using the exit orifice plenum pressure generally described in Section 7.4. However, using the stagnation pressure generally provides a more accurate

indication of sampler flow rate. Additionally, a continuous-flow recorder may be connected to the exit orifice pressure tap for nonquantitative determination that the flow rate was constant and uninterrupted over the sample period.

The stagnation pressure should be measured with a 0-1000 mm (0-36") oil, water, or digital manometer. Also, each sampler should have its own dedicated manometer, which can be conveniently mounted to the side of the sampler housing. Other types of pressure measurement instruments may be used provided they have comparable accuracy. However, the 4" continuous pressure (i.e., flow) recorders often supplied with HV samplers are generally not sufficiently accurate and are **not recommended** for quantitative sampler pressure or flow rate measurements.

The VFC sampler's flow control system is a choke-flow venturi. This system must be precisely sized for a given average annual temperature and pressure because no means is provided for the user to adjust the operational flow rate. Therefore, the purchasing agency should notify the manufacturer of the **operational** location of the sampler; differences in temperature and pressure between the shipping address and the monitoring site may result in an incorrect operational flow rate. As with the MFC sampler, both the ambient temperature and barometric pressure readings must be determined or estimated during the sampling period for the subsequent calculation of total sampler volume in standard volume units.

For this VFC calibration procedure, the following conditions are assumed:

- The VFC sampler uses a choked-flow venturi to control the actual volumetric flow rate.
- The sampler flow rate is measured by measuring the stagnation pressure ratio, and the sampler is not equipped with a continuous flow recorder.
- The sampler inlet is designed to operate at a constant actual volumetric flow rate of 1.13 m³/min.
- The transfer standard for the flow-rate calibration is an orifice device equipped with either a series of resistance plates or an integral variable-resistance valve. The pressure drop across the orifice is measured by an associated water or oil manometer.
- The sampler will be calibrated in actual volumetric flow-rate units (Q_a), and the orifice transfer standard is also calibrated in Q_a, as specified in Section 7.3.

7.5.1 Calibration Equipment.

7.5.1.1 Orifice transfer standard with proper calibration traceable to NIST (see Section 7.3).

7.5.1.2 An associated water, oil, or digital manometer, with a 0-400 mm (0-16") range and minimum scale divisions of 2 mm (0.1").

7.5.1.3 An oil, water, or digital manometer, with a 0-1000 mm (0-36") range and minimum scale divisions of 2 mm (0.1") or other pressure measurement device for measurement of the sampler stagnation pressure. Ideally, this manometer (or other pressure instrument) should be associated with the sampler.

[Note: Manometers used for field calibration may be subject to damage or malfunction and should thus be checked frequently.]

7.5.1.4 Thermometer, capable of accurately measuring temperature over the range of 0-50EC (273-323 K) to the nearest ± 1EC and referenced to an NIST or ASTM thermometer within ± 2EC at least annually.

7.5.1.5 A portable, aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient barometric pressure over the range of 500-800 mm Hg to the nearest mm Hg and referenced within ± 5 mm Hg to a barometer of known accuracy at least annually.

7.5.1.6 Calibration data sheets or the station log book and 51 mm (2")-wide duct tape.

7.5.1.7 A clean filter.

7.5.2 Multipoint Flow-Rate Calibration Procedure - VFC Sampler. The procedure presented here is basic and intended to be generic, given the assumptions listed in Section 7.5. More detailed calibration procedures, variations, or alternative procedures may be presented in the manufacturer's instruction manual. The manual should be reviewed carefully and that the various calibration variations or alternative procedures be evaluated. In-house equipment and personnel, procedural simplicity and uniformity, and subsequent data applications should be considered in establishing the specific, detailed calibration procedure to be implemented.

[Note: The calibration of some VFC samplers may be affected by changes in line voltage, particularly if the line voltage is below normal (normal is about 115 V). For this reason, VFC samplers should always be calibrated at the monitoring site. Further, if the line voltage at the site is low and likely to fluctuate significantly, a line voltage booster or regulator may be advisable. Also, be sure that replacement blower motors are of the correct type.]

[Note: Do not attempt to calibrate the VFC sampler under windy conditions. Short-term velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The calibration will be less precise because of the pressure variations.]

7.5.2.1 Set up the calibration system as recommended by the manufacturer. A typical VFC sampler calibration configuration is illustrated in Figure 12. The VFC sampler manufacturer may specify that the sampler be calibrated with a filter installed, which generally precludes calibration flow rates higher than normal operating flow rate. Additional calibration flow rates obtained without a filter may be appropriate, as discussed in Section 7.5.2.8.

7.5.2.2 Install the orifice transfer standard and its adapter faceplate on the sampler. First inspect all gaskets and seals and replace any doubtful ones.

[Note: Tighten the faceplate nuts evenly on alternate corners to properly align and uniformly seat the gaskets. The nuts should be hand-tightened only; too much compression can damage the sealing gasket.]

7.5.2.3 Select a calibration flow rate and install the appropriate resistance plate (or no plate) or adjust the variable resistance valve. At least four flow rates are required to define the calibration relationship. At least three flow rates should be within the acceptable flow-rate range (i.e., 1.02-1.24 m³/min) for the sampler inlet. For resistance plate orifices, make sure the orifice and resistance plate gaskets are in place and the orifice is not cross-threaded on the faceplate.

7.5.2.4 Leak check the system by blocking the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Seal the pressure port with a rubber cap or similar device. Turn on the sampler. Gently rock the orifice transfer standard and listen for a whistling sound that would indicate a leak in the system. Leaks are usually caused either by a damaged or missing gasket between the orifice transfer standard and the faceplate or by crossthreading of the orifice transfer standard on the faceplate. All leaks must be eliminated before proceeding with the calibration. When the system is determined to be leak-free, turn off the sampler and unblock the orifice.

[Note: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime. It can raise temperatures to the point of defeating the electrical insulation, which could result in fire or electric shock to the user.]

7.5.2.5 Inspect the connecting tubing of the manometers for crimps or cracks. Open the manometer valves (if present) and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometers' sliding scales so that their zero lines are at the bottom of the menisci. Connect the transfer standard manometer to the transfer standard and the sampler stagnation pressure manometer (or other pressure instrument) to the stagnation pressure port. Ensure that one side of each manometer is open to atmospheric pressure. Make sure the tubing fits snugly on the pressure ports and on the manometers.

7.5.2.6 Read and record the following parameters on the VFC Sampler Data Sheet. An example calibration data sheet for the VFC sampler is illustrated in Figure 13.

- Date, location, and operator's signature.
- Sampler S/N and model.
- Ambient barometric pressure (Pa), mm Hg.
- Ambient temperature (Ta), EC and K (K = EC + 273).
- Orifice S/N and calibration relationship.

[Note: Consistency of temperature and barometric pressure units is required. All temperatures should be expressed in kelvin (K = EC + 273). Also, all barometric pressures should be expressed in mm Hg. Avoid calibrating a HV sampler using one set of units and then performing sampler calculations using another set.]

7.5.2.7 Turn on the sampler and allow it to warm to operating temperature (3-5 min). Read and record the orifice transfer standard's manometer reading, H₂O, and the corresponding sampler relative stagnation pressure manometer reading, Pstg, on the data sheet. (Relative stagnation pressure is a negative pressure [i.g., a vacuum] relative to atmospheric pressure as measured by a manometer with one leg open to the atmosphere.) Be sure to convert the manometer reading to mm Hg using the following equation before recording the reading on the calibration data sheet:

$$\text{mm Hg} = 25.4 (\text{in. H}_2\text{O}/13.6)$$

[Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2" of clearance at the bottom of air flow and for the manometer tubing.]

7.5.2.8 Install the other resistance plates or adjust the variable orifice value to obtain each of the other calibration flow rates and repeat Section 7.5.2.7 for each. At least four calibration flow rates are required with at least three in the acceptable flow-rate range. Difficulties may be encountered in obtaining flow rates in the acceptable range. Even with modified resistance plates (or with no plates) installed, it may be impossible to obtain three acceptable flow rates with a filter mounted on the sampler. Lower flow rate calibration points may be used by extrapolation into the acceptable range without a filter installed in the sampler. If additional calibration points are obtained without a filter, they should be examined carefully to make sure they are consistent with the calibration points obtained with a filter (i.e., they fall on a smooth curve through all the calibration points).

7.5.2.9 Plot the calibration data on a sheet of graph paper as specified in Section 7.5.3.5 of the next section. Repeat Section 7.5.2.7 for any data that are questionable on the plot. Running additional calibration points at differing flow rates or repeating the calibration points at the same flow rates is encouraged to improve the precision of the calibration.

[Note: The data should be plotted in the field as the calibration is occurring, rather than afterwards back at the laboratory.]

7.5.2.10 Turn off the sampler and remove the orifice transfer standard.

7.5.2.11 Install a clean filter on the sampler in the normal sampling mode (use a filter cassette if one is normally used). Turn on the sampler and allow it to warm up to operating temperature.

7.5.2.12 Read the relative stagnation pressure as in Section 7.5.2.7 and record it on the data sheet in the row for the operational flow rate.

7.5.2.13 Perform the calibration calculations presented in the following sections.

7.5.3 Calibration Calculations. Gather together all the calibration data, including the orifice transfer standard's calibration information and the sampler calibration data sheet.

[Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that have already been obtained.]

7.5.3.1 Verify that the orifice transfer standard calibration relationship is current and traceable to an acceptable primary standard.

7.5.3.2 Calculate the record $Q_a(\text{orifice})$ for each calibration point from the orifice calibration information and the equation.

$$Q_a(\text{orifice}) = \left\{ \left[\frac{H_2O(T_a/P_a)}{m} \right]^{1/2} - b \right\} \{l/m\}$$

where:

$Q_a(\text{orifice})$ = actual volumetric flow rate as indicated by the transfer standard orifice, m^3/min .

H_2O = pressure drop across the orifice, in. H_2O .

T_a = ambient temperature during use, K ($K = EC + 273$).

P_a = ambient barometric pressure during use, mm Hg.

b = intercept of the orifice transfer standard's calibration relationship.

m = slope of the orifice transfer standard's calibration relationship.

7.5.3.3 Calculate and record the value of the absolute stagnation pressure ratio, $[PI]$, for each calibration point:

$$[PI] = P_a - P_{\text{stg}}$$

where:

$[PI]$ = absolute stagnation pressure, mm Hg.

P_a = ambient barometric pressure, mm Hg.

P_{stg} = relative stagnation pressure, mm Hg.

7.5.3.4 Calculate and record the stagnation pressure ratio:

$$\text{Stagnation pressure ratio} = PI/P_a$$

7.5.3.5 On a sheet of graph paper, plot the calculated orifice transfer standard's flow rates, $Q_a(\text{orifice})$, on the x-axis vs. the corresponding stagnation pressure ratios, PI/P_a , on the y-axis. Draw a smooth curve through the plotted data. If necessary, extrapolate the curve to include the acceptable flow-rate range.

7.5.3.6 If the sampler manufacturer has provided a factory calibration table (i.e., the lookup table) for the sampler, compare $Q_a(\text{orifice})$ for several points on the calibration plot with $Q_a(\text{sampler})$ determined from the factory calibration. Calculate the percentage difference between $Q_a(\text{orifice})$ and $Q_a(\text{sampler})$ using the following equation.

$$\% \text{ Difference} = \frac{Q_a(\text{sampler}) - Q_a(\text{orifice})}{Q_a(\text{orifice})} \cdot 100$$

If the agreement is within a few (i.e., 2 or 4) percent, the factory calibration is validated and may be used for subsequent sample periods. Proceed to Section 7.5.5.

7.5.3.7 If the agreement is not within a few percentage points, recheck the accuracy of the orifice transfer standard and recheck the calibration procedure. Look for leaks, manometer reading errors, incorrect temperature or pressure data, or miscalculations. Also check for abnormally low line voltage at the site (it should be at least 110 V ac), for the correct blower motor, and for the presence of a gasket between the motor and the choked-flow venturi. A factory calibration is not likely to be substantially incorrect, and any discrepancy of more than a few percent is probably due to some problem with the sampler or with the calibration procedure. However, if no errors or problems with the sampler or with the calibration can be found, or if no factory calibration is provided by the manufacturer, proceed as described in Section 7.5.4.

7.5.4 Generation of Calibration Relationship - VFC Sampler.

7.5.4.1 For each calibration point, calculate and record the quantity,

$$[(P/P_a)T_a]^{1/2}$$

where:

P/P_a = stagnation pressure ratio from the equation in Section 7.5.3.

T_a = ambient temperature during sampler calibration, K ($K = EC + 273$).

7.5.4.2 For the general linear regression model, $y = mx + b$, let $y = [(P/P_a)T_a]^{1/2}$ and let $x = Q_a(\text{orifice})$, such that the model is given by:

$$[(P/P_a)T_a]^{1/2} = m[Q_a(\text{orifice})] + b$$

Calculate the linear regression slope (m), intercept (b), and correlation coefficient (r).

[Note: Inspect the plotted calibration curve to determine whether any of the calibration points that are substantially outside of the acceptable flow-rate range need to be eliminated so that they do not result in an inappropriate linear regression line.]

7.5.4.3 For subsequent sample periods, the sampler's average actual operating flow rate, \bar{Q}_a , is calculated from the calibration slope and intercept using the following equation.

$$\bar{Q}_a(\text{sampler}) = \{[\bar{P}/P_{av}]T_{av}\}^{1/2} - b \} \{1/m\}$$

where:

$\bar{Q}_a(\text{sampler})$ = the sampler's average actual flow rate, m^3/min .

\bar{P}/P_{av} = average stagnation pressure ratio for the sampling period.

T_{av} = average ambient temperature for the sampling period, K ($K = EC + 273$).

b = intercept of the sampler calibration relationship.

m = slope of the sampler calibration relationship.

[Note: The average value for P_l should be calculated from stagnation pressure measurements taken before and after the sampling period. P_{av} should be estimated from barometric pressure for the sampling period. See also Section 9.4 for additional information.]

7.5.4.4 If a calibration (Lookup) table is desired, evaluate the above equation for various appropriate values of P_l/P_a and T_a and list the corresponding values of $Q_a(\text{sampler})$ in tabular form.

7.5.5 Single-Point Operational Flowrate Ventilation. This procedure compares the VFC sampler's normal operating flow rate to the design flow rate of the inlet (e.g., 1.13 m³/min).

7.5.5.1 Determine the value of P_l/P_a for the operational flow rate obtained with only the filter cassette installed (see Section 7.5.2.11 and Section 7.5.2.12).

7.5.5.2 Determine the new sampler flow rate, $Q_a(\text{sampler})$ from the lookup table that corresponds to this value of P_l/P_a . Use the manufacturer's calibration table if it has been validated in 7.5.3.6; otherwise, use the equation in Section 7.5.4.3.

7.5.5.3 Compare $Q_a(\text{sampler})$ with the inlet design flow rate (e.g., 1.13 m³/min) using the following equation:

$$\text{Design flow rate\% difference} = \frac{Q_a(\text{sampler}) \& 1.13}{1.13} \times 100$$

This design flow rate percentage difference must be less than the allowable flow rate tolerance (i.e., ± 10 , if not otherwise specified by the manufacturer). However, this value should be well within ± 7 to allow for some variation with ambient temperature. If this value is not within ± 7 , recheck the calibration procedure and data for errors. Check the sampler for leaks, bad motor brushes, missing gaskets, incorrect motor type, or abnormally low line voltage. Because the VFC flow rate is not adjustable, the VFC manufacturer must be consulted to resolve cases of substantially incorrect VFC flow rates.

7.6 Sampler Calibration Frequency

To ensure accurate measurement calibrate HV samplers upon installation and recalibrate as follows:

7.6.1 At least quarterly or annually (see 40 CFR 58, Appendix A for a description of the quality assurance requirements);

7.6.2 After any repairs that might affect sampler calibration (e.g., replacing the motor);

7.6.3 After relocation of the sampler to a different site;

7.6.4 If the results of a field flow-check exceed quality control limits (e.g., greater than $\pm 7\%$ from the sampler's indicated flow rate); or

7.6.5 Whenever a field flow-check or performance audit indicates that the sampler is out (or nearly out) of the acceptable flow-rate range.

[Note: Multipoint flow-rate calibrations should be distinguished from single-point, quality control flow checks (see Section 13). The latter are done more frequently than calibrations and are intended to check if the sampler flow rate, $Q_a(\text{sampler})$, or the calibration relationship has changed significantly since the last calibration.]

8. Filters

8.1 Pre-weighing of Filters

8.1.1 Filters ready for field use have been pre-weighed in the laboratory, under prescribed climate control conditions of temperature and relative humidity, using Inorganic Compendium Method IO-3.1, *Selection, Extraction and Preparation of Filter Material*.

8.1.2 Within Method IO-3.1, the user is provided guidance on proper selection of filter material in order to meet project specific data quality objectives (DQOs), how to visually inspect a new lot of filters for consistency and identification of defects, and initial weighing of the filters so a net concentration of particulate matter can be calculated after sampling.

8.1.3 The user should follow the procedures outlined within Method IO-3.1 as part of meeting the program's standard operating procedures (SOPs) and quality control (QC) requirements.

8.2 Filter Handling

8.2.1 Filter material may be brittle and subject to shearing and breakage. Laboratory and field personnel must be aware of these characteristics and handle sample filters with care.

8.2.2 For convenience, filters can be packed in groups of 50 or less in their original containers or in a box of comparable size. The filters should be separated by a sheet of 8 ½ x 11" tracing paper. Filter inventory can be controlled by stacking the filters in numerical order so that the operator will use the proper filter first. One side of the shipping box can be cut away to allow the operator to remove the filter easily without damaging the corners.

8.2.3 A filter identification number must be assigned to each filter. Because of difficulty in seeing the "up" side (i.e., the side with the slightly rougher texture) of the filter, consistency in labeling these filters will allow the operator easy access to the filter ID number for documentation and cross-referencing laboratory data forms. This consistency will also eliminate confusion in loading the filter cassettes for subsequent sampling. If the filter ID number is embossed by the operating agency, gentle pressure must be used to avoid filter damage, and extreme care must be taken to avoid duplication or missed numbers.

8.2.4 If samples are to be mailed, the field operator should be supplied with reinforced envelopes and manila folders for protection of the exposed filters during their return to the analytical laboratory. These manila folders may be printed to serve as sample data sheets.

8.3 Visual Filter Inspection

All filters must be visually inspected for defects, and defective filters must be rejected if any are found. Batches of filters containing numerous defects should be returned to the supplier.

The following are specific defects to look for:

- **Pinhole** - a small hole appearing as a distinct and obvious bright point of light when examined over a light table or screen, or as a dark spot when viewed over a black surface.
- **Loose material** - any extra loose material or dirt particles on the filter that must be brushed off before the filter is weighed.
- **Discoloration** - any obvious visible discoloration that might be evidence of a contaminant.
- **Filter nonuniformity** - any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity across the face of the filter.
- **Other** - a filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

9. Sampling Procedure

[Note: This section describes routine operation of a monitoring site using an HV sampler and covers an array of topics, ranging from initial site selection to final data documentation. The procedures herein are intended to serve as guidelines for developing a monitoring program that will accurately reflect trends in local or regional air quality. The effectiveness of the monitoring program depends on responsible day-to-day operation of the monitoring site. The operators who conduct sampling activities offer a unique perspective on the sampler's performance, and their awareness and attention to detail will salvage data that may otherwise be lost. Note, however, that "routine" does not mean "unimportant." The site operator provides cohesiveness in a sampling program.]

9.1 Summary

9.1.1 The PM₁₀ sampler can be used in a number of ways. Procedure variations may include the kind of filter medium, the surface area of the filter, prescreening to exclude particles up to a given size, and the manner of placing and exposing the filter during the test. The procedure most commonly used will be described here.

9.1.2 Calibrate the sampler as described in the Section 7. Do not make any change or adjustment on the sampler flow indicator after calibrating. Remove the calibrating orifice. The filters may be packed into a box with sheets of glassine between the filters, or they may be individually packed in self-sealing plastic bags for transportation to the field.

9.1.3 Mount the filter sheet in the filter holder taking care not to lose any of the fiber. Clamp it in place by means provided. Seal into place easier by facing the smooth side into the housing if there is a difference in texture. If the filter holder is separate from the sampler, mount the holder on the intake port, making sure that the coupling gasket is in place and that it is tight.

9.1.4 Place the sampler in the position and location called for in the test, which is with the filter face up, in a horizontal plane, and inside a housing. The dimensions and clearances specified are intended to provide uniformity in sampling practice.

9.1.5 Start the sampler motor and record the time and date. Read the flow-rate indicator and record this reading and the corresponding flow rate as read from the calibration curve. Note also the temperature and barometric pressure. An electric clock should be connected to the same line as the motor so as to detect any loss of test time due to power interruption. A continuous record of the sampling flow rate and sampling time can be obtained by the use of a continuous pressure (or flow rate) recorder.

9.1.6 Allow the sample to run for the specified length of time, which is commonly 24 h, \pm 1 h. During this period several readings of flow rate, temperatures, barometric pressure, and time should be taken if this is feasible. A final set of reading is taken at the end of the test period. If only initial and final readings are made, assume that change of readings is linear over the period of test. Intermediate readings will improve the accuracy of volume measurement.

9.1.7 At the end of the sampling period, record all final readings. Remove the filter from the mount very carefully so as not to lose any of the fiber material or collected particulate matter. Fold the filter in half upon itself with the collected material enclosed within. Place the folded filter in a clean tight envelope and mark it for identification. In some applications it may be desirable to place the used filter in a tight metal container to prevent any loss or damage to the filter.

9.1.8 In the laboratory remove the filter from its container. Tap the container and knock any loose fiber or particulate matter onto the inside surface of the folded filter. Examine the inside surface and, with a pair of tweezers, remove any accidental objects such as insects.

9.2 Siting Requirements

9.2.1 As with any type of air monitoring study in which sample data are used to draw conclusions about a general population, the validity of the conclusions depends on the representativeness of the sample data. Therefore, the primary goal of a monitoring project is to select a site or sites where the collected particulate mass is representative of the monitored area.

9.2.2 Basic siting criteria for the placement of high-volume sampler (either TSP or PM₁₀) are documented in Table 3. This list is not a complete listing of siting requirements; instead, an outline to be used by the operating agency to determine a sampler's optimum location. Complete siting criteria are presented in 40 CFR 58, Appendix E.

9.2.3 Additional factors not specified in the Code of Federal Regulations (CFR) must be considered in determining where the sampler will be deployed. These factors include accessibility under all weather conditions, availability of adequate electricity, and security of the monitoring personnel and equipment. The sampler must be situated where the operator can reach it safely despite adverse weather conditions. If the sampler is located on a rooftop, care should be taken that the operator's personal safety is not jeopardized by a slippery roof surface during inclement weather. Consideration also should be given to the fact that routine operation (i.e., calibrations, filter installation and recovery, flow checks, and audits) involves transporting supplies and equipment to and from the monitoring site.

9.2.4 To ensure that adequate power is available, consult the manufacturer's instruction manual for the sampler's minimum voltage and power requirements. Lack of a stable power source can result in the loss of many samples because of power interruptions.

9.2.5 The security of the sampler itself depends mostly on its location. Rooftop sites with locked access and ground-level sites with fences are common. In all cases, the security of the operating personnel as well as the sampler should be considered.

9.3 Sampler Installation Procedures

9.3.1 On receipt of a high-volume sampler (TSP or PM₁₀) from the manufacturer, visually inspect it and account for all components. Compare the equipment delivered with the enclosed packing slip. Notify the manufacturer immediately of any missing or damaged equipment.

9.3.2 Perform a laboratory check to determine if the sampler is operational. Turn on the sampler and observe the vacuum motor performance and shift the recorder response (if so equipped).

9.3.3 Carefully transport the sampler to the field site. If possible, install the sampler in the center of the site platform. This practice will ensure easy access to the sampler's inlet during maintenance procedures and will reduce inlet damage if the sampler should topple over.

9.3.4 Following manufacturer's instructions, carefully assemble the base and inlet of the sampler. The sampler must be bolted down to a secure mounting surface.

9.3.5 Check all tubing and power cords for crimps, cracks, or breaks.

9.3.6 Plug the power cord into a line voltage outlet. If possible, this outlet should be protected by a ground fault interrupter (GFI) for the operator's safety. The use of waterproof interlocking electrical connectors is also recommended to ensure operator safety and to avoid shorts or power interruptions. Do not allow any electrical connections to be submerged during periods of inclement weather.

9.3.7 Turn on the sampler and make sure that it is still working properly. Investigate and correct any malfunctions before proceeding. Operate the sampler for approximately 30 min to ensure that the motor brushes are properly seated and that the motor is operating at full performance.

9.3.8 Perform a multipoint flow-rate calibration, as described in Section 7.

9.4 Sampling Operations

9.4.1 General.

9.4.1.1 Operational procedures will vary according to the sampler model and options (e.g., the types of flow-rate controller and timer) selected for use in the monitoring program. Consult the instrument manual before putting the sampler into operation. Significant differences exist in the field operation of the two types of flow-controlling systems and, hence, in the determination of operational flow rates. The following assumptions are made in this section:

- The flow rate through a sampler that is equipped with a mass-flow controller is indicated by the exit orifice plenum pressure. This pressure is measured with a manometer (or a flow recorder).
- The flow rate through a sampler that is equipped with a volumetric-flow controller is indicated by the stagnation pressure. This pressure is measured with a manometer.
- The sampler has been calibrated according to procedure presented in Section 7.

9.4.1.2 The sampler has been calibrated according to procedures presented in Section 7.

9.4.1.3 The average actual flow rate for MFC samplers is calculated by determining the following:

- The average of the initial and final manometer readings of the exit orifice plenum pressure (or the average flow recorder reading).
- The average ambient temperature (T_{av}).
- The average ambient barometric pressure (P_{av}) during the sampling period.

These values are then applied to the sampler's calibration relationship. The 4" pressure flow recorders often supplied with HV samplers generally are not sufficiently accurate and are *not recommended* for quantitative sampler pressure or flow rate measurements. These flow recorders should be used only for nonquantitative determination that the flow was approximately constant and uninterrupted over the sampling period. The flow recorder may be connected in parallel with the manometer or other pressure measuring device using a tee or "Y" tubing connector.

[Note: Because flow recorders are still widely used for quantitative flow rate measurements, the procedures in this section include specific instructions for the use of a flow recorder. These flow recorder instructions are enclosed in brackets.]

9.4.1.4 The average actual flow rate for VFC samplers is calculated by determining the following:

- The average of the initial and final relative stagnation pressures (P_{stg}).
- The average ambient temperature (T_{av}).
- The average barometric pressure (P_{av}) during the sampling period and then by applying these values to the calibration relationship.

*[Note: Consistency of temperature and barometric pressure units is required. All temperatures should be expressed in kelvin ($K = EC + 273$). Also, all barometric pressures should be expressed in either mm Hg or kPa (**but don't mix the two units**). Avoid calibrating a PM_{10} sampler using one set of units and then performing sample calculations using another set.]*

9.4.2 Presampling Filter Preparation Procedures.

9.4.2.1 Most high-volume samplers (TSP or PM_{10}) have been designed to accept filter cassettes. Loading these cassettes in the laboratory will minimize damage; however, if extreme care is exercised, they can be loaded at the site when ambient conditions permit. Wear protective gloves when handling filters to avoid contaminating the filters with body oils and moisture. Keep the filters in protective folders or boxes. Never bend or fold unexposed filters. The analytical laboratory (and/or filter manufacturer) will give each filter an ID number. Because it is extremely difficult to see the "up" side of a quartz filter (i.e., the side with the slightly rougher texture), the filters should be consistently labeled on one side. When a filter that has

been labeled on its "down" side is folded for transport to the laboratory, its sample number will be readily accessible for documentation on laboratory log sheets upon arrival at the laboratory.

9.4.2.2 Following the manufacturer's instructions, carefully load the pre-weighted filter in the filter cassette. The filter should be centered on the wire screen so that the gasket will form an airtight seal on the outer edge of the filter when the faceplate is in place. Poorly aligned filters show uneven white borders after exposure. Care should be taken to ensure that the filter cassette is not excessively tightened, as the filter may stick or the gasket may be permanently damaged. Check that the gasket is in good condition and has not deteriorated.

9.4.3 Sampling Procedures--MFC Sampler.

9.4.3.1 Filter Installation Procedure.

9.4.3.1.1 Following the manufacturer's instructions, loosen the nuts that secure the inlet to the base and gently tilt back the inlet to allow access to the filter support screen.

9.4.3.1.2 Examine the filter support screen. If the screen appears dirty, wipe it clean. If the filter cassette is equipped with a protective cover, remove it and place the loaded cassette in position on the sampler support screen. Tighten the thumb nuts to hold the filter cassette securely. Check that the gasket is in good condition and has not deteriorated.

Caution: Tighten the thumb nuts evenly on alternate corners to properly align and seat the gasket. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.

9.4.3.1.3 If an inlet is being used, lower the sample inlet. Inspect the sample inlet to make sure that it is resting on the filter cassette and not on the sampler's frame. Secure the sample inlet to the sampler base.

9.4.3.1.4 Open the front door of the sample and examine the flow recorder. Remove any moisture inside by wiping it with a clean cloth. Record the sampler S/N, filter ID number, site location, and sampling data on the back of a clean chart and install the chart in the flow recorder.

[Note: Charts used for PM₁₀ samplers normally have square-root-function scales; however, linear-function scales may be used. If charts with linear-function scales are used, Equations in Section 7.4.3.3 and Section 7.4.3.5 will have to be modified from their current form by replacing I with (I)^{1/2}]

[Note: While installing the chart, do not bend the pen arm beyond its limits of travel. Raise the pen head by pushing on the very top of the pen air (or by using the pen lift). Be sure that the chart tab is centered on the slotted drive to ensure full 360° rotation in 24 h. Make sure that the chart edges are properly located beneath the retainers. Lower the pen arm and tap the recorder face lightly to make certain that the pen is free.]

[Note: During periods of inclement weather, the chart tends to stick to the recorder face. Two charts can be installed simultaneously to enable the sample (top, annotated) chart to rotate freely.]

9.4.3.1.5 Using a coin or slotted screwdriver, advance the chart and check to see that the pen rests on zero--the smallest circle diameter. If necessary, adjust the zero set screw while gently tapping on the side of the flow recorder. If a chart with a linear function scale is used, some positive zero offset may be desirable to allow for normal variation in the zero readings.

9.4.3.1.6 Turn on the sampler and allow it to equilibrate to operating temperature (3-5 min).

9.4.3.1.7 While the sampler is equilibrating, record the following parameters on the MFC Sampler Field Data Sheet (see Figure 14):

- Site Location.
- Sample date.
- Filter ID number.
- Sampler model and S/N.
- Operator's initials.

9.4.3.1.8 Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer while watching for the free flow of the fluid. Adjust the manometer's sliding scale so that its zero line is at the bottom of the menisci.

9.4.3.1.9 Measure the initial exit orifice plenum pressure (Pex) using an oil or water manometer, with a 0-200-mm (0-8") range and a minimum scale division of 1 mm (0.1"). Record the initial Pex on the MFC Sampler Field Data Sheet. If Pex is substantially different than for previous samples or otherwise appears abnormal, carry out a Quality Control (QC) flow check as described in Section 13.1.

9.4.3.1.10 Verify that the flow recorder (if used) is operational and that the pen is inking. Note the flow recorder reading. If it is substantially different than for previous samples or otherwise appears abnormal, carry out a QC flow-check as described in Section 13.1.

9.4.3.1.11 Turn the sampler off.

9.4.3.1.12 Check the time indicated by the time-set pointer on the flow recorder. If it is in error, rotate the chart clockwise by inserting a screwdriver or coin in the slotted drive in the center of the chart face until the correct time is indicated.

9.4.3.1.13 Reset the elapsed time meter to 0000 min and the sampler timer for the next run day. Close the sampler door, taking care not to crimp the vacuum tubing or any power cords. The sampler is now ready to sample ambient air.

9.4.3.2 Filter Recovery Procedure. As soon as possible after sampling, the operator should return to the monitoring site to retrieve the exposed filter. Particle loss or filter damage will result if the filter is left in the sampler for extended periods.

9.4.3.2.1 Turn on the sampler and allow it to equilibrate to operating temperature (3-5 min).

9.4.3.2.2 Measure the final Pex and record it on the MFC Sampler Field Data Sheet.

9.4.3.2.3 Turn off the sampler.

9.4.3.2.4 Open the door of the sampler, remove the flow recorder chart, and examine the recorder trace. If the trace indicates extensive flow fluctuations, investigate and correct before the next sampling day.

9.4.3.2.5 Record the following parameters on the MFC Sampler Field Data Sheet:

- Elapsed time of the sampling period, min.
- Average recorder response, arbitrary units.
- Average ambient temperature for the run day (Tav), K ($K = EC + 273$).
- Average ambient barometric pressure for the run day (Pav), mm Hg or kPa.

[Note: Tav and Pav readings may be recorded or estimated on site or may be obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the evaluation are not available, seasonal average temperature (Ts) and barometric pressure (Ps) may be substituted for Tav and Pav, respectively. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. Therefore, seasonal values may represent actual values within 20EC and 40 mm Hg.]

The calculations presented in this section assume that the sampler has been calibrated in terms of actual temperature and barometric pressure and that the substitution of seasonal values is used only to determine the sampler's operational flow rate during a sample period. Although additional calculations to convert the sampler's calibration curve to seasonal can be made, the error represented by this method is negligible.

9.4.3.2.6 Calculate and record the average actual flow rate (as determined by the sampler's calibration relationship) on the MFC Sampler Field Data Sheet and on the back of the chart. Attach the chart to the data sheet.

$$Q_a = \{I\} \overline{P_{ex}} (T_{av} + 30)/P_a]^{1/2} - b\} \{1/m\}$$

or for the flow recorder,

$$\overline{Q_a} = \{[\overline{I}] (T_{av} + 30)/P_a]^{1/2} - b\} \{1/m\}$$

where:

$\overline{Q_a}$ = average sampler flow rate, actual m³/min.

$\overline{P_{ex}}$ = average exit orifice plenum pressure, mm Hg.

I = average flow recorder response, arbitrary units.

T_{av} = average ambient temperature for the run day, K.

P_{av} = average ambient pressure for the run day, mm Hg.

b = intercept of the MFC sampler calibration relationship.

m = slope of the MFC sampler calibration relationship.

[Note: If charts with linear-function scales are used, substitute (I)^{1/2} for I.]

9.4.3.2.7 Observe conditions around the monitoring site; note any activities that may affect filter particle loading (e.g., paving, mowing, fire) and record this information on the MFC Sampler Field Data Sheet.

9.4.3.2.8 Raise the sampler inlet and remove the filter cassette. Replace the cassette protective cover (if so equipped). To avoid particle loss, be careful to keep the cassette as level as possible.

9.4.3.2.9 The sampler may now be readied for the next run day.

9.4.3.2.10 Keeping the filter cassette level, carefully transport it, the data sheet, and the flow recorder chart to the laboratory sample custodian.

9.4.4 Sampling Procedures--VFC Sampler.

9.4.4.1 Filter Installation Procedure.

9.4.4.1.1 Following the manufacturer's instructions, loosen the nuts that secure the inlet to the base and gently tilt back the inlet to allow access to the filter support screen.

9.4.4.1.2 Examine the filter support screen. If the screen appears dirty, wipe it clean. If the filter cassette is equipped with a protective cover, remove it and place the loaded cassette in position on the sampler support screen. Tighten the thumb nuts sufficiently to hold the filter cassette securely. Check that the gasket is in good condition and has not deteriorated.

Caution: Tighten the thumb nuts evenly on alternate corners to properly align and seat the gasket. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.

9.4.4.1.3 If an inlet is used, lower the sample inlet and secure it to the sampler base. For impaction inlets, inspect the sample inlet to make sure that it is resting on the filter cassette and not on the sampler's frame. Secure the sampler inlet to the sampler base.

9.4.4.1.4 Record the following parameters on the VFC Sampler Field Data Sheet (see Figure 15):

- Site location.
- Sample date.
- Filter ID number.
- Sampler model and S/N.
- Operator's initials.

9.4.4.1.5 Turn on the sampler and allow it to reach a stable operating temperature (3-5 min).

9.4.4.1.6 Bring an oil or water manometer to the side of the sampler. This manometer should have a range of 0-400 mm (0-16") and a minimum scale division of 1 mm (0.1").

[Note: Be sure to convert the manometer reading to mm Hg using the following equation before recording the reading on the VFC Sampler Field Data Sheet.]

$$\text{mm Hg} = (\text{in. H}_2\text{O}/13.6)$$

Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer, while watching for the free flow of the fluid.

Adjust the manometer's sliding scale so that its zero line is at the bottom of the menisci.

9.4.4.1.7 Remove the vacuum cap from the stagnation pressure port located on the side of the sampler base. Using the connecting tubing, attach one side of the manometer to the port. Leave the other side of the manometer open to atmospheric pressure. Make sure the tubing snugly fits the port and the manometer.

9.4.4.1.8 Measure the initial relative stagnation pressure () Pstg) and record this reading on the VFC Sampler Field Data Sheet.

9.4.4.1.9 Turn off the sampler, disconnect the manometer, and replace the vacuum cap on the stagnation pressure port.

9.4.4.1.10 Reset the elapsed-time meter to 0000 min and the sampler timer for the next run day. The sampler is now ready to sample ambient air.

9.4.4.2 Filter Recovery Procedure. As soon as possible after sampling, the operator should return to the monitoring site to retrieve the exposed filter. Particle loss or filter damage will result if the filter is left in the sampler for extended periods.

9.4.4.2.1 Turn on the sampler and allow it to warm up to operating temperature (3-5 min).

9.4.4.2.2 While the sampler is equilibrating, record the following parameters on the VFC Sampler Field Data Sheet:

- Elapsed time of the sampling period, min.
- Average ambient temperature for the run day (Tav), EC and K.
- Average ambient barometric pressure for the run day (Pav), mm Hg.

[Note: Tav and Pav readings may be recorded or estimated on site or may be obtained from a nearby U.S. National Weather Service Forecast Office, National Weather Service (NWS) station, or an airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If Tav and Pav readings are not available, seasonal average temperature (Ts) and barometric pressure (Ps) can be substituted. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. Therefore, seasonal values may represent actual values within 20EC and 40 mm Hg.]

9.4.4.2.3 Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer, while watching for the free flow of the fluid. Adjust the manometer sliding scale so that its zero line is at the bottom of the menisci.

9.4.4.2.4 Remove the vacuum cap from the stagnation pressure port located on the side of the sampler base. Using the connecting tubing, attached one side of the manometer to the port. Make sure that the tubing snugly fits the port and the manometer. Leave the other side open to atmospheric pressure.

9.4.4.2.5 Record the final Pstg on the VFC Sampler Field Data Sheet. Turn off the sampler and replace the vacuum cap.

[Note: Be sure to convert the manometer reading to mm Hg using the following equation before recording the reading on the Sampler Field Data Sheet.]

$$\text{mm Hg} = 25.4 (\text{in. H}_2\text{O}/13.6)$$

9.4.4.2.6 Calculate the average relative stagnation pressure (\overline{Pstg}) and record it on the data sheet.

9.4.4.2.7 Calculate the average absolute stagnation pressure ($\overline{P1}$) for the sample run day and record it on the data sheet.

$$\overline{P1} = P_{av} - \overline{Pstg}$$

where:

$\overline{P1}$ = average absolute stagnation pressure, mm Hg.

P_{av} = average ambient barometric pressure for the run day (not the retrieval day), mm Hg.

\overline{Pstg} = average stagnation pressure drop, mm Hg.

9.4.4.2.8 Calculate and record the average stagnation pressure ratio:

$$\text{Average stagnation pressure ratio} = P1/P_{av}$$

where:

P1 = average absolute stagnation pressure, mm Hg.

Pav = average ambient barometric pressure on the sample run day, mm Hg.

9.4.4.2.9 Using the manufacturer's lookup table (or an alternate calibration relationship as described in Section 7.5.4), locate the column and row corresponding to $\overline{P1}/P_{av}$ and the Tav value for the sample run day. Read and record the indicated \overline{Qa} value.

9.4.4.2.10 Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the VFC Sampler Field Data Sheet.

9.4.4.2.11 Raise the sampler inlet and remove the filter cassette. Replace the cassette protective cover (if so equipped). To avoid particle loss, be careful to keep the cassette as level as possible.

9.4.4.2.12 The sampler may now be readied for the next sampling period.

9.4.4.2.13 Keeping the filter cassette level, carefully transport it and the Sampler Field Data Sheet to the laboratory sample custodian.

9.4.5 Post-Sampling Filter Handling Procedures. If a sample will not be analyzed immediately, the sample custodian should store the filter within a protective covering. Because filter cassettes often prove too expensive and unwieldy for storage purposes, the use of a manila folder and a protective envelope of comparable size to that of the filter is recommended. Laboratory personnel should adhere to the following procedure:

9.4.5.1 Following the manufacturer's instructions, remove the top frame of the filter cassette.

9.4.5.2 Conduct a secondary check of a sample's validity as presented in "Laboratory Validation Criteria" (see Section 9.5).

9.4.5.3 Carefully slip a manila folder underneath the edge of the exposed filter. The filter may stick in the cassette because of overcompression of the filter cassette gasket. Be extremely careful to avoid damage to the brittle quartz filter.

9.4.5.4 Center the filter on the folder. If the filter must be touched, do not touch or jar the deposit. Fold the manila folder lengthwise at the middle with the exposed side of the filter in. If the collected sample is not centered on the filter (i.e., the unexposed border is not uniform around the filter), fold it so that only deposit touches deposit. **Do not crease the folder**--the sample filter may tear. If the filter shears or breaks, ensure that all pieces of the filter are included within the folder.

9.4.5.5 Insert the folder into the protective envelope.

9.4.5.6 Deliver the filter in its protective folder and envelope, accompanied by the completed data sheet, to the analytical laboratory.

9.5 Sample Validation and Documentation

9.5.1 **Field Validation Criteria.** After each sampling period, calculate the percentage difference between Q_a and the design flow rate (1.13 m³/min) using the following formula:

$$\% \text{ Difference} = 100 \frac{\overline{Q_a} - 1.13}{1.13}$$

Record this value on a control chart for the field validation of the sampler's actual volumetric flow rate as is shown in Figure 16.

- Decreases in flow rate during sampling (due to mechanical problems) of more than 10% from the initial set point result in sample invalidation. Recalibrate the sampler. A sample flow rate may also fluctuate due to heavy filter loading. If a high concentration is suspected, the operator should indicate this on the field data sheet. The laboratory supervisor will make the final decision regarding the sample's validity.
- Changes in flow-rate calibration of more than 10%, as determined by a field QC flow-rate check (see Section 13), will invalidate all samples collected back to the last calibration or valid flow check. Recalibrate the sample.

9.5.2 Laboratory Validation Criteria.

9.5.2.1 Upon receiving the filter from the field, check the filter for signs of air leakage by observing the border around the filter. If the border is clear, then the gasket on the sampler is still usable. However, if particulate matter is on the border, then air leakage has occurred and the gasket on the sampler should be changed. Leakage may result from a worn or improperly installed faceplate gasket. A gasket generally deteriorates slowly. The sample custodian should be able to decide well in advance (by the increased fuzziness of the sample outline) when to change the gasket before total gasket failure results. If signs of leakage are observed, void the sample, determine the cause, and instruct the operator to take corrective actions before starting another sampling period.

9.5.2.2 Check the exposed filter for physical damage that may have occurred during or after sampling. Physical damage after sampling would not invalidate the sample if all pieces of the filter were put in the

folder; however, complete losses of loose particulate after sampling (e.g., loss when folding the filter) would void the sample. Mark such samples as "void" on the HV data sheet.

9.5.2.3 Check the appearance of the particles. Any changes from normal color may indicate new emission sources or construction activity in the area. Note any change on the data sheet.

9.5.2.4 The filters should be weighed according to the procedures described in Inorganic Compendium Method IO-3.1, Section 5, *Gravimetric Analysis*.

9.5.3 Data Documentation. Recordkeeping is a critical part of the QA program. Careful documentation of sampling data will salvage samples that may otherwise be lost. The sheer repetition of recording data may result in errors; however, this cross-referencing between data sheets, log books, and (for those samplers so equipped) the continuous-flow recorder charts will allow the operator to pinpoint discrepancies that may result in a sample's invalidation.

[Note: The use of log books at monitoring sites is highly encouraged.]

9.5.3.1 Presampling Documentation and Inspection. The following information should be recorded on the Sampler Field Data Sheet (SFDS), sampler recorder chart (RC), flow-rate control chart (CC), and in the site log book (LB):

- Site Location.
- Sample Date.
- Filter ID number.
- Sample model and S/N.
- Operator's initials.

9.5.3.2 Post-Sampling Documentation and Inspection. Upon receipt of exposed filters from the field, the sample custodian should adhere to the following procedures.

9.5.3.2.1 Examine the field data sheet. Determine whether all data needed to verify sample validity and to calculate mass concentration are provided (e.g., average flow rate, ambient temperature, barometric pressure, and elapsed time). Void the sample if data are missing or unobtainable from a field operator or if a sampler malfunction is evident.

9.5.3.2.2 If the exposed filter was packaged for shipment, remove the filter from its protective envelope and examine the shipping envelope. If sample material has been dislodged from a filter, recover as much as possible by brushing it from the envelope onto the deposit on the filter with a soft camel's-hair brush.

9.5.3.2.3 Match the filter ID number with the correct laboratory data/coding form on which the original balance ID number, filter ID number, filter tare weight, and other information are inscribed. The sample custodian should group filters according to their recorded balance ID numbers. Initial separation of filters by balance ID number will decrease the probability of a balance error that could result from the use of different balances for tare and gross weights.

9.5.3.2.4 Remove the filter from the protective manila folder. Should the filter be retained in its filter cassette, loosen the nuts on the top and remove the filter. Overtightening the nuts may cause the filter to adhere to the cassette gasket. Gently remove it by the extreme corners to avoid damage. Inspect the filters for any damage that may have occurred during sampling. Conduct a secondary check of a sample's validity (as presented in Section 9.4). If insects are embedded in the sample deposit, remove them with Teflon®-tipped tweezers and disturb as little of the sample deposit as possible. If more than 10 insects are observed, refer the sample to the supervisor for a decision on acceptance or rejection of the filter for analysis.

9.5.3.2.5 Place defect-free filters in protective envelopes and forward them to the laboratory for weighing and analysis. File the data sheets for subsequent mass concentration calculations.

9.5.3.2.6 Place defective filters, with the type of defect(s) listed, in separate clean envelopes. Label the envelopes and submit them to the laboratory supervisor for final approval of filter validity.

10. Interferences

10.1 Large extraneous objects, such as insects, may be swept into the filter.

10.2 Liquid aerosols, such as oil mists and fog droplets, are retained by the filter. If the amount of liquid so collected is sizeable, the filter can become wet and its function may be impaired.

10.3 Any gaseous or vaporous constituent of the atmosphere under test that is reactive with or absorbed on the filter will be retained.

10.4 As the filter becomes loaded with collected matter, the sampling rate is reduced. If a significant drop in flow rate occurs, the average of the initial and final flow rate will not give an accurate estimate of total flow during the sampling period. The magnitude of such errors will depend on the amount of reduction of airflow rate and on the variation of the mass concentration of dust with time during the 24-h sampling period. As an approximate guideline, any sample should be suspect if the final flow rate is less than one-half the initial rate.

10.5 Power failure or voltage change during the test period will lead to an error, depending on the extent and time duration of such failure.

10.6 The passive loading of the filter left in place for any time prior to or following a sampling period can introduce an error. The timely installation and removal of the filter is advisable, or a sampler with shutters may be used.

10.7 If two or more samplers are used at a given location, they should be placed at least 2 meters apart so that one sampler will not affect the results of an adjacent sampler.

10.8 Recent wind tunnel studies have shown significant possible sampling errors as a function of sampler orientation in atmospheres containing high relative concentrations of large particles.

10.9 Metal dusts from motors, especially copper, may significantly contaminate samples under some conditions.

10.10 Under some conditions, atmospheric SO₂ and NO_x may interfere. Artifact formation errors are caused by the retention of sulfur dioxide in the form of sulfate particulate on alkaline filters. Experiments involving a variety of filters indicate that sulfate loading errors of 0.3-3.0 µg/m³ can be expected with the use of common glass fiber filters under normal sampling conditions and that larger sulfate errors are possible under extreme sampling conditions. A neutral or low-alkalinity filter medium will eliminate excessive artifact formation.

10.11 Guidelines to help prevent post-sampling particle loss are presented in Section 8.

11. Calculations, Validations, and Reporting of TSP and PM₁₀ Data

11.1 Basic Information Used for Calculations

11.1.1 The design flow rate is specified as an actual volumetric flow rate (Q_a), measured at existing conditions of temperature (T_a) and pressure (P_a). The sampler's operational flow rate should be very close to the design flow rate. All samplers have some means for measuring the operational flow rate, and that flow rate measurement system must be calibrated periodically with a certified flow rate transfer standard. Usually, measurements (or estimates) of ambient temperature and barometric pressure are required to get an accurate indication of the operational flow rate. To determine the average sampler flow rate over a sample period, use the average temperature (T_{av}) and average barometric pressure (P_{av}) over the sample period. However, if average temperature and pressure values (or reasonable estimates) cannot be obtained for each sample period, seasonal average temperature (T_s) and barometric pressure (P_s) for the site may be substituted.

[Note: T_{av} and P_{av} readings may be recorded on site or estimated from data obtained from a nearby U.S. National Weather Service Forecast Office, NWS station, or local airport weather station. Barometric pressure readings obtained from airports or other sources must be at station pressure (i.e., not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If individual T_{av} and P_{av} readings cannot be obtained for each sample period and seasonal averages for the site are routinely substituted, care must be taken to ensure that the actual temperature and barometric pressure at the site are reasonably represented by such averages. Therefore, seasonal average temperature and pressure values (T_s and P_s) for the site by should be used only when these values are within 20 K and 40 mm Hg (5 kPa) of the actual average temperature and barometric pressure (T_{av} and P_{av}) for the sample period.]

11.1.2 The calculations presented in this section assume that the sampler has been calibrated in actual volumetric flow rate units (Q_a) and that individual average temperature and barometric pressure values are used for each sample period. If seasonal average temperature and pressure values for the site are to be used, T_s may be substituted for T_{av} , and P_s may be substituted for.

11.1.3 The true or actual flow rate through the sampler inlet must be known and controlled. A common source or error in a monitoring program is confusion of various air volume flow-rate measurement units. Although the sampler's operational flow rate must be monitored in terms of actual volume flow rate units (Q_a), sampler flow rates can be corrected to standard volume flow rate units (Q_{std}) at EPA standard conditions of temperature (25°C) and pressure (760 mmHg).

- Q_a : Actual volumetric air flow rates, measured and expressed at existing conditions of temperature and pressure and denoted by Q_a (Q_{actual}). Typical units are L/min and m³/min. Inlet design flow rates for PM₁₀ samplers are always given in actual volumetric flow rate units.
- Q_{std} : Airflow rates that have been corrected to equivalent standard volume flow rates at EPA standard conditions of temperature and pressure (25°C or 298 K and 760 mm Hg or 101 kPa) and denoted by Q_{std} ($Q_{standard}$). Typical units are std. L/min, and std. m³/min. Standard volume flow-rate units are often used by engineers and scientists because they are equivalent to mass flow units.

11.1.4 The Q_a and Q_{std} measurement units must not be confused or interchanged. The flow-rate units can be converted as follows, provided the existing temperature and pressure (or in some cases the average temperature and pressure over a sampling period) are known:

$$\begin{aligned}\overline{Q_{std}} &= \overline{Q_a} (P_a/P_{std})(T_{std}/T_a) \\ Q_{std} &= (P_{av}/P_{std})(T_{std}/T_{av}) \\ Q_a &= Q_{std}(P_{std}/P_a)(T_a/T_{std})\end{aligned}$$

where:

- Q_{std} = standard volume flow rate, std m³/min.
- Q_a = actual volume flow rate, actual m³/min.
- P_a = ambient barometric pressure, mm Hg.
- P_{std} = EPA standard barometric pressure, 760 mm Hg.
- T_{std} = EPA standard temperature, 298 K.
- T_a = standard temperature, K (K = EC + 273).
- $\overline{Q_{std}}$ = average standard volume flow rate for the sample period, std. m³/min.
- $\overline{Q_a}$ = average actual volume flow rate for the sample period, m³/min.
- P_{av} = average ambient barometric pressure during the sample period, mm Hg.
- T_{av} = average ambient temperature during the sample period, K.

Inorganic Compendium Method IO-2.4 provides guidance on calculating sample volume corrected to EPA standard temperature and pressure.

11.2 Flow-Rate Calculations. Because flow control methods (and hence, calibration procedures) vary among different sampler models, the calculations necessary to determine the average actual flow rate during a sample run will also differ. The following general procedures are recommended for calculating the average ambient flow rate of the sampler. In this section, it is assumed that the samplers have been calibrated according to procedures outlined in Section 7.

[Note: Consistency in units is required. Adoption of uniform designations of K for temperature and mm Hg (or kPa) for pressure is recommended in all calculations.]

11.2.1 MFC Sampler.

11.2.1.1 The average actual flow rate for sample period is calculated by determining:

- The average of the initial and final manometer readings ($\overline{P_{ex}}$) [or the average flow recorder trace];
- The average ambient temperature (T_{av}); and
- The average ambient barometric pressure (P_{av}) during the sampling period and applying these values to the calibration relationship.

11.2.1.2 Each sampler's flow measurement system should be calibrated periodically, and the calibration should be described by a mathematical expression (e.g., a least-squares linear regression equation) that indicates the slope and intercept of the calibration relationship. Following the procedure in Section 7, this expression is in the form of:

$$\overline{Q_a} = \{[\overline{P_{ex}}(T_{av} + 30)/P_{av}]^{1/2} - b\} \{1/m\}$$

where:

- $\overline{Q_a}$ = the sampler's average actual flow rate for the sample period, m³/min.
- $\overline{P_{ex}}$ = average of initial and final sampler manometer readings, $(P_{ex_i} + P_{ex_f})/2$, in. H₂O.
- T_{av} = average barometric pressure for the sample period, K (K = EC + 273).
- P_{av} = average barometric pressure for the sample period, mm Hg.
- b = intercept of the sampler calibration relationship.
- m = slope of the sampler calibration relationship.

For the flow recorder,

$$\overline{Q_a} = \{[\overline{I} (T_{av} + 30)/P_{av}]^{1/2} - b\} \{1/m\}$$

where:

- \overline{I} = average flow recorder reading for the sample period.

11.2.1.3 The average actual flow rate is then corrected to EPA-standard conditions, calculated as:

$$\overline{Q_{std}} = \overline{Q_a} (P_{av}/P_{std})(T_{std}/T_{av})$$

where:

- $\overline{Q_{std}}$ = average sampler flow rate corrected to EPA-standard volume flow rate units, std. m³/min.
- $\overline{Q_a}$ = average actual sampler flow rate for the sample period, m³/min.
- P_{std} = standard barometric pressure, 760 mm Hg.
- T_{std} = standard temperature, 288 K.

11.2.2 VFC Sampler.

11.2.2.1 The average actual flow rate for the sample period is calculated by determining the ratio of the average absolute stagnation pressure of the average ambient barometric pressure ($\overline{P_1}/P_{av}$) and the ambient average temperature (T_{av}) for the sampler period.

11.2.2.2 Calculate the value of P_1 in mm Hg:

$$\overline{P_1} = P_{av} - \overline{P_{stg}}$$

where:

- P_1 = average absolute stagnation pressure for the sample period, mm Hg.
- P_{av} = average barometric pressure for the sample period, mm Hg.
- $\overline{P_{stg}}$ = average of initial and final relative stagnation pressure readings, mm Hg.

[Note: Be sure to convert a water manometer reading to mm Hg using the following equation before recording the reading on the data sheet:]

$$\text{mmHg} = 25.4 (\text{H}_2\text{O}/13.6)$$

11.2.2.3 Calculate and record the value of the average stagnation pressure ratio.

$$\text{Average stagnation pressure ratio} = (\overline{P_1}/P_{av})$$

11.2.2.4 Use the manufacturer's lookup table (or alternate calibration relationship; see Section 7) to determine Q_a from the average stagnation pressure ratio ($\overline{P_1}/P_{av}$) and T_{av} for the sample period. The value of $\overline{Q_a}$ is the average volumetric flow rate for the sampler period.

11.2.2.5 The average actual flow rate is then corrected to EPA-standard conditions using the following equation:

$$\overline{Q_{std}} = \overline{Q_a} (P_{av}/P_{std})(T_{std}/T_{av})$$

where:

$\overline{Q_{std}}$ = average sampler flow rate corrected to EPA-standard volume flow rate units, std. m^3/min .

$\overline{Q_a}$ = average actual sampler flow rate for the sample period, m^3/min .

P_{std} = standard barometric pressure, 760 mm Hg.

T_{std} = standard temperature, 298 K.

11.3 The total standard volume of air sampled is calculated by the following equation:

$$V_{std} = (\overline{Q_{std}})(t)$$

where:

V_{std} = total volume of air sampled in standard volume units, std m^3 .

$\overline{Q_{std}}$ = average sampler flow rate corrected to EPA-standard conditions, std m^3/min .

t = total elapsed sampling time, min.

11.4 Percent Difference

11.4.1 After each sampling period, calculate the percentage difference between Q_a and the design flow rate (1.13 m^3/min) using the following formula:

$$\% \text{ Difference} = 100 \frac{Q_a - 1.13}{1.13}$$

Record this value on a control chart for the field validation of the sampler's actual volumetric flow rate as is shown in Figure 14.

11.4.2 The following criteria should be used as the basis for determining a sample's validity:

- Decreases in flow rate during sampling (due to mechanical problems) of more than 10% from the initial set point cause sample invalidation. A sample flow rate may also fluctuate due to heavy filter loading. If a high concentration is suspected, the operator should indicate it on the field data sheet. The laboratory supervisor will make the final decision regarding the sample's validity.
- Changes in flow-rate calibration of more than 10%, as determined by a field QC flow-rate check, will invalidate all samples collected back to the last calibration or valid flow check.

12. Records

12.1 MFC Sampler

Record the following parameters on the MFC Sampler Field Data Sheet (see Figure 14):

- Final Pex.
- Elapsed time of the sampling period, min.
- Average record response, arbitrary units.
- Tav for the run day K ($K = EC + 273$).
- Pav for the run day, mm Hg.

12.2 VFC Sampler

Record the following parameters on the VFC Sampler Field Data Sheet (see Figure 15):

- Site location.
- Sample date.
- Filter ID number.
- Sampler model and S/N
- Operator's initials.
- Initial Relative Stagnation Pressure (Pstg).
- Elapsed time of the sampling period, min.
- Tav for the run day Tav, EC and K.
- Pav for the run day Pav, mm Hg.
- Pstg, mm Hg.
- Relative Stagnation Pressure.
- Absolute Stagnation Pressure.
- Qa value (from chart generated in Section 7.5.4.).

12.3 Tav and Pav readings may be recorded or estimated on site or may be obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level); they may have to be corrected for differences between elevation of the monitoring site and that of the airport. If Tav and Pav readings are not available, seasonal average temperature (Ts) and barometric pressure (Ps) may be substituted for Tav and Pav, respectively. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. Therefore, seasonal values should represent actual values within 20EC and 40 mm Hg.

12.4 Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the VFC Sampler Field Data Sheet.

Document any factors that may cause a sample's invalidation on the sample data sheet. Forward the data sheet and the filter to the laboratory supervisor, who will make the final decision regarding the sample's validity.

12.5 Record the percentage difference between Qa and the design flow rate on Figure 16.

12.6 Recordkeeping is a critical part of the QA program. Careful documentation of sampling data will salvage samples that may otherwise be lost. The sheer repetition of recording data may result in errors;

however, this cross-referencing between data sheets, log books, and (for those samplers so equipped) the continuous-flow-recorder charts will allow the operator to pinpoint discrepancies that may result in a sample's invalidation.

[Note: The use of log books at monitoring sites is highly encouraged. The following information should be recorded on the Sampler Field Data Sheet (SFDS), sampler recorder chart (RC), in the site log book (LB), and on the flow-rate control chart (CC).]

12.6.1 The following information should be recorded by the operator who starts the sample. (The designation in parentheses indicates where the data must be inscribed):

- Site designation and locations (SFDS)(RC)(LB). This information should be recorded in the log book only once, at the initiation of a monitoring program.
- Sampler model and S/N (SFDS)(RC)(LB). This information needs to be recorded in the log book only at the commencement of monitoring, unless there is more than one sampler or a new sampler has been deployed.
- Filter ID number (SFDS)(RC)(LB).
- Sample date (SFDS)(RC)(LB).
- Initial Pex for MFC or initial) Pstg for VFC (SFDS)(LB).
- Unusual conditions that may affect the results (e.g., subjective evaluation of pollution that day, construction activity, weather conditions) (SFDS)(LB).
- Operator's initials (SFDS).
- Signature (LB).

12.6.2 The following information should be recorded by the operator who removes the samples.

- Elapsed time of the sample run (SFDS)(RC)(LB).
- Final) Pex [or mean I] for MFC or final) Pstg, $\overline{P_1}$, and $\overline{P_1}/P_{av}$ for VFC (DS)(LB)[RC].
- The calculated standard average flow rate (Qstd) in std m³/min (SFDS)(LB).
- The percentage difference between the actual and design flow rates (CC).
- Average ambient temperature and barometric pressure on the sample run day (SFDS)(LB).
- Seasonal average temperature and pressure, if needed (SFDS/LB). This information needs to be recorded in the logbook once, at the change of each season.
- Existing conditions that may affect the results (SFDS)(LB).
- Explanations for voided or questionable samples (SFDS)(LB).
- Operator's initials (SFDS).
- Signature (LB).

13. Field QC Procedure

For HV samplers, a field-calibration check of the operational flow rate is recommended at least once per month. The purpose of this check is to track the sampler's calibration stability. A control chart (e.g., Figure 14) that contains the percentage difference between a sampler's indicated and measured flow rates should be maintained. This chart is a quick reference of instrument flow-rate drift problems and is useful for tracking the performance of the sampler. Either the sampler log book or a data sheet must be used to document flow-check information. This information includes, but is not limited to, instrument and transfer standard model and serial numbers, ambient temperature and pressure conditions, and collected flow-check data.

In this section, the following is assumed:

- The flow rate through sampler that is equipped with a mass-flow controller is indicated by the exit orifice plenum pressure. This pressure is measured with a manometer [or a flow recorder].
- The flow rate through a sampler that is equipped with a volumetric flow controller is indicated by the stagnation pressure. This pressure is measured with a manometer.
- The acceptable flow-rate fluctuation range is 10% of the design flow rate.
- The transfer standard will be an orifice device equipped with a water or oil manometer.
- The orifice transfer standard's calibration relationship is in terms of the actual volumetric flow rate (Qa).

13.1 QC Flow-Check Procedure--MFC Sampler. The indicated flow rate [Qa (sampler)] for MFC samplers is calculated by determining:

- The manometer reading of the exit orifice plenum pressure [or the flow recorder reading],
- The ambient temperature (Ta), and
- The barometric pressure (Pa) during the flow check.

These values are then applied to the sampler's calibration relationship. The 4" pressure (flow) recorders of the type often supplied with high-volume PM₁₀ samplers are generally not sufficiently accurate and are not recommended for quantitative sampler pressure or flow measurements. The flow recorder may be connected in parallel with the manometer or other pressure measuring device, using a tee or "Y" tubing connector. An alternate QC flow-check procedure may be presented in the manufacturer's instruction manual. The manual should be reviewed and the various methods evaluated. Inhouse equipment and procedural simplicity should be considered in determining which method to use.

[Note: Do not attempt to conduct a flow check of samplers under windy conditions. Short-term wind velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The flow check will be less precise because of the pressure variations.]

13.1.1 Collect the following equipment and transport it to the monitoring station:

[Note: An independent person should perform the QC flow check, with an outside observer present.]

- A water, oil, or digital manometer with a 0-200 mm (0-8") range and a minimum scale division of 1 mm (0.1") for measuring the sampler's exit orifice plenum pressure. This manometer should be the same as is used routinely for sampler flow rate measurements.
- An orifice transfer standard and its calibration relationship (different from initial orifice standard).
- An associated water or oil manometer with a 0- to 400-mm (0- to 16") range and a minimum scale division of 1 mm (0.1") for measuring the orifice transfer standard.
- A thermometer capable of accurately measuring temperature 0-50EC (273-323 K) to the nearest ± 1EC and referenced to an NIST or ASTM thermometer within ± 2EC at least annually.
- A portable aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient pressure 500-800 mm Hg (66-106 kPa) to the nearest millimeter Hg and referenced within ± 5 mm Hg of a barometer of known accuracy at least annually.
- The sampler's calibration information.
- Spare recorder charts and a clean flow-check filter.
- MFC Sampler Flow-Check Data Sheet or site log book.

13.1.2 Record the site location, sampler S/N, and date on the back of a clean chart and install it in the flow recorder. While installing the chart, do not bend the pen arm beyond its limits of travel. Raise the pen head by pushing on the very top of the pen arm (or by using the pen lift) and simultaneously insert the chart.

13.1.3 Lower the pen arm and tap the recorder face lightly to make certain that the pen can move freely.

13.1.4 Using a coin or slotted screwdriver, advance the chart and check to see that the pen head rests on zero (i.e., that smallest diameter circle). If necessary, adjust the zeroset screw while gently tapping on the side of the recorder. A quarter turn of the set screw usually results in large offsets; adjust the set screw carefully.

13.1.5 Set up the flow-check system as previously illustrated in Figure 10. MFC samplers are normally flow-checked with a filter in line (i.e., between the orifice transfer standard and the motor). Install a clean filter in the sampler. Place the filter directly upon the sampler's filter screen. Do not use a filter cassette. A flow-check filter should never be used for subsequent sampling because particles larger than 10 Fm can be collected on the filter while the inlet is raised. The sample mass will be biased as a result of using a filter for both a flow check and subsequent sampling.

13.1.6 Install the orifice transfer standard and its faceplate on the sampler. Do not restrict the flow rate through the orifice (i.e., by using fixed resistance plates or closing the variable-resistance valve).

Caution: Tighten the faceplate nuts on alternate corners first to eliminate leaks and to ensure even tightening. The nuts should be hand-tightened; too much compression can damage the sealing gasket. Make sure the orifice transfer standard gasket is in place and the orifice transfer standard is not cross-threaded on the faceplate.

13.1.7 Connect the orifice manometer to the pressure port of the orifice transfer standard and the sampler manometer to the sampler's exit orifice plenum. Inspect the manometers' connecting tubings for crimps and cracks. Open the manometer valves and blow gently through the tubings. Watch for the free flow of fluid. Adjust the manometers' scales so that their zero lines are at the bottom of the meniscuses. Make sure that the connecting tubing snugly fits the manometer and the pressure port.

13.1.8 Turn on the sampler and allow it to warm up to operating temperature (3-5 min).

[Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2" of clearance at the bottom for air flow and for the manometer tubing.]

13.1.9 Read and record the following parameters on the MFC Sampler Flow-Check Data Sheet:

- Sampler location and date.
- Sampler model and S/N.
- Ambient temperature (T_a), EC and K.
- Ambient barometric pressure (Pa), mm Hg.
- Unusual weather conditions.
- Orifice transfer standard S/N and calibration relationship.
- Operator's signature.

13.1.10 Observe the H_2O across the orifice by reading the manometer deflection. Record the manometer deflection on the MFC Sampler Flow-Check Data Sheet (see Figure 11).

13.1.11 Measure the exit orifice plenum pressure (P_{ex}) by reading the manometer deflection. Record the manometer deflection on the MFC Sampler Flow-Check Data Sheet.

13.1.12 Using a coin or small screwdriver, advance the recorder chart to read the sampler's corresponding response (I) and record on the data sheet. A gentle tap on the recorder face is often necessary to ensure that the pen is not sticking to the chart.

13.1.13 Turn off the sampler and remove the orifice transfer standard, but not the filter. Turn on the sampler and repeat Section 13.1.11 [or Section 13.1.12] to check the flow rate under normal operating conditions. Turn off the sampler and remove the filter.

13.1.14 Calculate and record $Q_a(\text{orifice})$ at actual conditions using the following equation:

$$Q_a(\text{orifice}) = \{[(\text{H}_2\text{O})(T_a/P_a)]^{1/2} - b\} \{l/m\}$$

where:

- $Q_a(\text{orifice})$ = actual volumetric flow rate as indicated by the orifice transfer standard, m^3/min
- H_2O = pressure drop across the orifice, in. H_2O .
- T_a = ambient temperature, K.
- P_a = ambient barometric pressure, mm Hg.
- b = intercept of the orifice calibration relationship.
- m = slope of the orifice calibration relationship.

13.1.15 Calculate and record the corresponding sampler flow rate at actual conditions using the following equation:

$$Q_a(\text{sampler}) = \{P_{\text{ex}} (T_a + 30)/P_a\}^{1/2} - b \} \{l/m\}$$

or use the following if a flow recorder is being used to measure the exit orifice plenum pressure:

$$Q_a(\text{sampler}) = \{I(T_a + 30)/P_a\}^{1/2} - b \} \{l/m\}$$

where:

- $Q_a(\text{sampler})$ = sampler flow rate, actual m^3/min .
- P_{ex} = exit orifice plenum pressure, in. H_2O .
- T_a = ambient temperature during the flow check, K ($K = \text{EC} + 273$).
- P_a = ambient barometric pressure during the flow check, mm Hg.
- b = intercept of the MFC sampler calibration relationship.
- m = slope of the MFC sampler calibration relationship.

[Note: If charts with linear-function scales are used, substitute $(I)^{1/2}$ for I .]

13.1.16 Using this information and the formulas provided on the MFC Sampler Flow-Check Data Sheet, calculate the QC check percentage differences.

$$\text{QC\&check \% difference} = \frac{[Q_a(\text{sampler}) \& Q_a(\text{orifice})]}{Q_a(\text{orifice})} [100]$$

where:

$Q_a(\text{sampler})$ is measured with the orifice transfer standard being installed.

Record this value on the MFC Sampler Flow-Check Data Sheet and plot on the QC control chart. If the sampler flow rate is within 93-107% ($\pm 7\%$ difference) of the calculated $Q_a(\text{orifice})$ flow rate (in actual volumetric units), the sampler calibration is acceptable. If these limits are exceeded, investigate and correct any malfunction. Recalibrate the sampler before sampling is resumed. Differences exceeding $\pm 10\%$ may result in the invalidation of all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the orifice transfer standard's calibration and all calculations.

13.1.17 Calculate the corrected sampler flow rate, $Q_a(\text{corr. sampler})$, using the following equation:

$$Q_a(\text{corr. sampler}) = [Q_a(\text{sampler})] \frac{[(100 \pm \% \text{ difference})]}{100}$$

where:

$Q_a(\text{sampler})$ is measured without the orifice transfer standard being installed and where the QC-check percentage difference was obtained from the equation above.

[Note: Take care to use the correct sign (i.e., positive or negative) for the percent difference.]

13.1.18 Calculate and record on the MFC Sampler Flow-Check Data Sheet the percentage difference between the inlet's design flow rate and the corrected sampler flow rate as:

$$\text{Design flow rate \% difference} = \frac{[Q_a(\text{corr. sampler}) \pm 1.13]}{1.13} [100]$$

[Note: The author assumes in this section that the inlet is designed to operate at a flow rate of 1.13 actual m^3/min . If the design flow rate percentage difference is less than or equal to $\pm 7\%$, the sampler calibration is acceptable. If the difference is greater than $\pm 7\%$, investigate potential error sources and correct any malfunction. Recalibrate the sampler before sampling is resumed. Differences exceeding $\pm 10\%$ may invalidate all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calculations.]

[Note: Deviations from the design flow rate may be caused in part by deviations in the site temperature and pressure from the seasonal average conditions. Recalculate the optimum set-point flow rate (SFR) according to Section 7.4.4 to determine if the flow controller should be adjusted.]

13.1.19 Set up the sampler for the next sampling period according to the operating procedure in Section 9.4.

13.2 QC Flow-Check Procedure--VFC Sampler

The indicated flow rate ($Q_a(\text{sampler})$) for VFC samplers is calculated by determining:

- The relative stagnation pressure (Pstg),
- The ambient temperature (T_a), and
- The barometric pressure (P_a) during the flow check.

These values are then applied to the sampler's calibration relationship. An alternative QC flow-check procedure may be presented in the manufacturer's instruction manual. The manual should be reviewed and the various methods evaluated. Inhouse equipment and procedural simplicity should be considered in determining which method to use.

[Note: Do not attempt to conduct a flow check of samplers under windy conditions. Short-term wind velocity fluctuations will provide variable pressure readings by the orifice transfer standard's manometer.]

The flow check will be less precise because of the pressure variations.

13.2.1 Collect the following equipment and transport it to the monitoring station:

- An orifice transfer standard and its calibration relationship in actual volumetric flow units (Qa).
- An associated oil, water, or digital manometer, with a 0-400 mm (0-16") range and minimum scale divisions of 1 mm (0.1").
- An oil, water, or digital manometer, with a 0-400 mm (0-16") range and minimum scale divisions of 1 mm (0.1") or other pressure measurement device for measurement of the sampler stagnation pressure. Ideally, this manometer (or other pressure measurement device) should be associated with the sampler.

[Note: Manometers used for QC flow-checks may be subject to damage or malfunction and thus should be checked frequently.]

- A thermometer capable of accurately measuring temperature from 0E-50EC (273-323 K) to the nearest ± 1 EC and referenced to an NIST or ASTM thermometer within 2EC at least annually. To calculate the orifice flow rates, convert EC to K.
- A portable aneroid barometer (e.g., a climber or engineer's altimeter) capable of accurately measuring ambient barometric pressure over the range of 500-800 mm Hg to the nearest millimeter Hg and referenced within 5 mm Hg of a barometer of known accuracy at least annually.
- The sampler's calibration relationship (i.e., lookup table or alternative calibration relationship).
- A clean flow-check filter loaded into a filter cassette.
- A VFC Sampler Flow-Check Data Sheet (see Figure 13) or a site log book.

13.2.2 Set up the flow-check system as previously illustrated in Figure 12. VFC samplers are normally flow-checked with a loaded filter cassette in line (i.e., between the orifice transfer standard and the motor). The orifice transfer standard should be installed without fixed resistance plates or with the adjustable resistance value fully open. A flow-check filter should never be used for subsequent sampling because particles larger than 10 Fm can be collected on the filter while the inlet is raised. The sample mass will be biased as a result of using a filter for both a flow check and subsequent sampling.

Caution: Tighten the faceplate nuts on alternate corners first to eliminate leaks and to ensure even tightening. The fittings should be hand-tightened; too much compressing can damage the sealing gasket. Make sure the orifice gasket is in place and the orifice transfer standard is not cross-threaded on the faceplate.

13.2.3 Turn on the sampler and allow the sampler to warm up to operating temperature (3-5 min).

[Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2" of clearance at the bottom for air flow and for the manometer tubing.]

13.2.4 Read and record the following parameters on the VFC Sampler Flow-Check Data Sheet (see Figure 13):

- Sampler location and date.
- Sampler S/N and model.
- Ambient temperature (Ta), EC and K.
- Ambient barometric pressure (Pa), mm Hg.
- Unusual weather conditions.
- Orifice transfer standard S/N and calibration relationship.
- Operator's signature.

13.2.5 Inspect the manometers for crimps or cracks in the connecting tubing. Open the valves and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometers' sliding scales so that the zero lines are at the bottom of the meniscuses.

13.2.6 Connect the orifice manometer to the orifice transfer standard and the sampler manometer to the sampler stagnation pressure port located on the side of the sampler base. Ensure that one side of each manometer is open to atmospheric pressure. Be sure that the connecting tubing snugly fits the pressure ports and the manometers.

13.2.7 Read the pressure drop as indicated by the orifice manometer (ΔP_{H_2O}) and record the value on the VFC Sampler Flow-Check Data Sheet. Read the stagnation pressure drop and record it as ΔP_{stg} (mm Hg) on the data sheet.

[Note: Be sure to convert the manometer reading to mm Hg using the following equation before recording the reading on the data sheet.]

$$\text{mm Hg} = 25.4(\text{in. H}_2\text{O}/13.6)$$

13.2.8 Turn off the sampler and remove the orifice transfer standard.

13.2.9 With only a loaded filter cassette in line, turn on the sampler and allow it to warm up to operating temperature.

13.2.10 Read and record the stagnation pressure drop (ΔP_{stg}) for the normal operating flow rate. Turn off the sampler. Replace the vacuum cap on the stagnation pressure port.

13.2.11 Calculate and record $Q_a(\text{orifice})$ flow rate for the flow-check point, as in the equation, reproduced below:

$$Q_a(\text{orifice}) = \{[(\Delta P_{H_2O})(T_a/P_a)]^{1/2} - b\} [l/m]$$

where:

- $Q_a(\text{orifice})$ = actual volumetric flow rate as indicated by the transfer standard orifice, m^3/min .
- ΔP_{H_2O} = pressure drop across the orifice, in. H_2O .
- T_a = ambient temperature, K ($K = \text{EC} + 273$).
- P_a = ambient barometric pressure, mm Hg.
- b = intercept of the orifice calibration relationship.
- m = slope of the orifice calibration relationship.

13.2.12 Calculate and record the value of P_l (mm Hg) for the measurements, with and without the orifice installed, according to the following equation:

$$P_l = [P_a - \Delta P_{stg}]$$

where:

- P_l = stagnation pressure, mm Hg.
- P_a = ambient barometric pressure, mm Hg.
- ΔP_{stg} = stagnation pressure drop, mm Hg.

13.2.13 Calculate and record the stagnation pressure ratio for the measurements, with and without the orifice installed, according to the following equation:

$$\text{Stagnation pressure ratio} = P_l/P_a$$

where:

- P_l = stagnation pressure, mm Hg.
- P_a = ambient barometric pressure, mm Hg.

13.2.14 Refer to the instrument manufacturer's lookup table (or alternative calibration relationship as described in Section 7.5.4) and determine the $Q_a(\text{sampler})$ flow rates (m^3/min) for the measurements with

and without the orifice installed as indicated for the ratio of Pl/Pa and ambient temperature in EC. Record these values on the VFC sampler flow check data sheet.

13.2.15 Using $Qa(\text{orifice})$ and $Qa(\text{sampler})$ for the measurements with the orifice installed, calculate the QC-check percentage difference as:

$$\text{QC\&check \% difference} = \frac{[Qa(\text{sampler}) \& Qa(\text{orifice})]}{Qa(\text{orifice})} [100]$$

Record this value on the VFC Sampler Flow-Check Data Sheet and plot it on the control chart for QC flow checks. If the QC-check percentage difference is less than or equal to $\pm 7\%$, the sampler calibration is acceptable. Those differences exceeding $\pm 7\%$ will require recalibration. Differences exceeding $\pm 10\%$ may invalidate all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calibrations.

13.2.16 Using this percentage difference and $Qa(\text{sampler})$ from the measurements without the orifice installed (i.e., for the normal operating flow rate), calculate the corrected sampler flow rate as:

$$Qa(\text{corr. sampler}) = [Qa(\text{sampler})] \frac{[(100 \& \% \text{ difference})]}{100}$$

Record $Qa(\text{corr. sampler})$ on the VFC Sampler Flow-Check Data Sheet.

13.2.17 Determine the design flow rate percentage difference between the PM_{10} sampler inlet design flow rate (e.g., $1.13 \text{ m}^3/\text{min}$) and $Qa(\text{corr. sampler})$ as:

$$\text{QC\&check \% difference} = \frac{[Qa(\text{sampler}) \& Qa(\text{orifice})]}{Qa(\text{orifice})} [100]$$

Record this design flow rate percentage difference on the VFC Sampler Flow-Check Data Sheet and plot it on the control chart for the field validation of flow rates. When plotting this value, use a different symbol than is normally used for plotting values that are obtained during sampling periods. If the design flow rate percentage difference is less than or equal to $\pm 7\%$, the sampler calibration is acceptable. Those differences exceeding $\pm 7\%$ will require recalibration. Differences exceeding $\pm 10\%$ may invalidate all data obtained subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calculations.

14. Maintenance

Maintenance is defined as a program of positive actions aimed toward preventing failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and provide more complete data acquisition. The general maintenance procedures for HV samplers are outlined in this section. For more complete information on a particular sampler or on laboratory equipment maintenance, refer to the manufacturer's instruction manual for the individual instrument. Maintenance activities for the HV sampler are summarized in Table 4. Records should be maintained for the maintenance schedule of each HV sampler. Files should reflect the history of maintenance, including all replacement parts, suppliers, costs, expenditures, and in inventory of on-hand spare equipment for each sampler. Check sheets should be used to record preventive and/or corrective maintenance activities and the subsequent sampler calibration curve.

14.1 Maintenance Procedures

The HV sampler is comprised of two basic components: the inlet and the flow control system. Because of the differences between sampler models, refer to the manufacturer's instruction manual for specific maintenance guidelines and necessary supplies.

14.2 Recommended Maintenance Schedules

14.2.1 MFC Base. The MFC base is equipped with the following items:

14.2.1.1 Connecting tubing and power lines, which must be checked for crimps, cracks, or obstructions on sample recovery days. Fittings should be inspected periodically for cross-threading and tightness.

14.2.1.2 A filter screen, which should be inspected on sample recovery days for any impacted deposits.

14.2.1.3 Filter cassette gaskets, which need to be inspected each time a cassette is loaded. A worn cassette gasket is characterized on exposed filters by a gradual blending of the boundary between the collected particulate and the filter border.

14.2.1.4 Motor and housing gaskets, which should be checked at 3-month intervals and replaced as necessary.

14.2.1.5 Blower motor brushes, which should be replaced before they become worn to the point that damage may occur. Although motor brushes usually require replacement after 600-1,000 hours of operation, the optimum replacement interval must be determined by experience. A pumice stone can be used against the motor's contacts to ensure high conductivity. Change the brushes according to manufacturer's instructions and perform the operator's field-calibration check as presented in Section 13. If the sampler's indicated flow rate exceeds the manufacturer-specified design-flow-rate range, adjust the sampler before the next run day.

To achieve the best performance, new brushes should be properly seated on the motor's commutator before full voltage is applied to them. After the brushes have been changed, operate the sampler at 50-75% of normal line voltage for approximately 30 min. The motor should return to full performance after an additional 30-45 min at normal line voltage.

[Note: The motors that are used for HV samplers are higher-current versions of the motors that have been used for HV total suspended particulate samplers. The brushes for the two types of motors are different. Make sure that the correct replacement brushes are used for the maintenance of HV samplers. If a motor needs to be replaced, be sure to use the higher-current versions that are needed for HV sampling. When lower-current motors are installed in HV samplers, the flow rate has been found to vary with changes in the line voltage.]

14.2.1.6 A flow controller should be replaced if the flow recorder indicates no flow, low flow, excessive flow, or erratic flow. Minor adjustments can be made to alter sampling flow rates; however, the controller generally cannot be repaired in the field.

[Note: A flow recorder requires very little maintenance, but does deteriorate with age. Difficulty in zeroing the recorder and/or significant differences (i.e., greater than 0.3 m³/min) in average flow rates obtained from consecutive sampling periods usually indicate a faulty recorder. The recorder pens should be replaced every 30 recording days. In dry climates, a more frequent replacement schedule may be required.]

14.2.2 VFC Base. The VFC base is equipped with the following items:

14.2.2.1 Power lines, which must be checked for crimps or cracks on sample recovery days. Fittings should be inspected periodically for cross-threading and tightness.

14.2.2.2 A filter screen at the throat of the choked-flow venturi, which should be inspected on sample recovery days for any impacted deposits.

14.2.2.3 Filter cassette gaskets, which should be checked each time a filter is installed. A worn cassette gasket is characterized on exposed filters by a gradual blending of the boundary between the collected particulates and the filter border.

14.2.2.4 Motor and housing gaskets, which should be checked at 3-month intervals and replaced as necessary.

14.2.2.5 Blower motor brushes, which should be replaced before they become worn to the point that damage may occur. Although motor brushes usually require replacement after 600-1,000 hours of operation, the optimum replacement interval must be determined by experience. A pumice stone can be used against the motor's contacts to ensure high conductivity. Change the brushes according to manufacturer's instructions, and perform the operator's field-calibration check as presented in Section 13. If the sampler's indicated flow rate exceeds the manufacturer-specified design flow-rate range, recalibrate the sampler before the next run day.

To achieve the best performance, new brushes should be seated properly on the motor's commutator before full voltage is applied to them. After the brushes have been changed, operate the sampler at 50-75% of normal line voltage for approximately 30 min. The motor should return to full performance after an additional 30-45 min at normal line voltage.

Caution: Motors that are used for HV PM₁₀ samplers are higher-current versions of the motors that have been used for HV total suspended particulate samplers. The brushes for the two types of motor are different. Make sure that the correct replacement brushes are used for the maintenance of HV PM₁₀ samplers.

14.2.2.6 If a motor needs to be replaced, be sure to use the higher-current versions that are needed for HV PM₁₀ sampling. When lower-current motors are installed in HV PM₁₀ samplers, the flow rate has been found to vary with changes in the line voltage.

14.3 Refurbishment of HV Samplers

If operated in the field for extended periods, HV PM₁₀ samplers may require major repairs or complete refurbishment. If so, refer to the manufacturer's instrument manual before work is undertaken. A sampler that has undergone major repairs or refurbishment must be leak-checked and calibrated prior to sample collection.

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TABLE 1. EXAMPLE OF BASIC CHARACTERISTICS OF SOME COMMON FILTER MATERIAL

<p>QUARTZ FIBER FILTER (Glass Spun with Organic Binder)</p>
<p>! Whatman QMA Filter</p> <ul style="list-style-type: none"> ! Maximum temperature of up to 540EC ! High Collection Efficiency ! Non-hydroscopic ! Good for Corrosive Atmospheres ! Fragile ! Lowest background metals content
<p>CELLULOSE FIBER FILTER (Cellulose Pulp)</p>
<p>! Whatman # 41/MSA "s"</p> <ul style="list-style-type: none"> ! Low Ash ! Maximum Temperature of 150EC ! High Affinity for Water ! Enhanced Artifact Formation for SO₄ and NO₃ ! Good for X-Ray/Neutron Activation Analysis ! Low Metal Content
<p>MEMBRANE FILTER (Dry Gel of Cellulose Esters)</p>
<p>! Whatman #41</p> <ul style="list-style-type: none"> ! Fragile, Therefore Requires Support Pad ! High Pressure Drop ! Low Residue when Ashed

TABLE 2. EXAMPLE OF SUMMARY OF USEFUL FILTER PROPERTIES

Filter and Filter Composition	Density mg/cm ²	pH	Filter Efficiency %
Teflon® (Membrane) (CF ₂) _n (2µm Pore Size)	0.5	Neutral	99.95
Cellulose (Whatman 41) (C ₆ H ₁₀ O ₅) _n	8.7	Neutral (Reacts with HNO ₃)	58% at 0.3 µm
Glass Fiber (Whatman GF/C)	5.16	Basic pH - 9	99.0
"Quartz" Gelman Microquartz	6.51	pH - 7	98.5
Polycarbonate (Nuclepore) C ₁₅ H ₁₄ + CO ₃ (0.3µm Pore Size)	0.8	Neutral	93.9
Cellulose Acetate/Nitrate Millipore (C ₉ H ₁₃ O ₇) _n (1.21 µm Pore Size)	5.0	Neutral (Reacts with HNO ₃)	99.6

TABLE 3. EXAMPLE OF MINIMUM SAMPLER SITING CRITERIA

Scale	Height above ground, meters	Distance from supporting structure, meters		Other spacing criteria
		Vertical	Horizontal ^a	
Micro	2 to 7	> 2	> 2	1. Should be > 20 meters from trees. 2. Distance from sampler to obstacle, such buildings, must be twice the height and the obstacle protrudes above the sampler. 3. Must have unrestricted airflow 270 degrees around the sampler inlet. 4. No furnace or incineration flues should be nearby. ^b 5. Spacing from roads varies with traffic (see 40 CFR 58, Appendix E). 6. Sampler inlet is at least 2 m but not greater than 4 m from any collocated PM ₁₀ sampler. (See 40 CFR 58, Appendix E.)
Middle, neighborhood, urban, and regional scale	2 to 15	> 2	> 2	

^aWhen inlet is located on rooftop, this separation distance is in reference to walls, parapets, or penthouses located on the roof.

^bDistance depends on the height of furnace or incineration flues, type of fuel or waste burned, and quality of fuel (sulfur, ash, or lead content). This is to avoid undue influences from minor pollutant sources.

TABLE 4. EXAMPLE OF ROUTINE MAINTENANCE ACTIVITIES
 FOR SAMPLERS

Equipment	Frequency and/or method	Acceptance limits	Action if requirements are not met
Sampler inlet	Dismantle and clean at manufacturer-specified intervals	No obvious particulate deposits or damage	Clean, replace damaged equipment before sampling
Sampler base			
Power lines	Check for crimps or cracks	No obvious damage	Replace as necessary
Filter screen and throat	Visually check on sample-recovery days	No obvious deposits; clean with wire brush	Clean
Gaskets	At 3-mo intervals, inspect all gaskets in the sampler	No leaks; no compression damage evident	Replace as necessary
Brushes	Replace after 600-1,000 h of operation	Stable flow rate	Replace as necessary
Motor	Replace if needed	Correct model must be used	Obtain correct model
Flow controller	Check when flowrate changes are evident	Stable flow rate throughout sample run	Replace or repair if possible
Recording device	Inspection with experiencing difficulty in zeroing, or when large changes in flow rates occur	Recorder stays zeroed; chart advances; pen inks	Replace or repair if possible
Tubing, fittings	Visually inspect on sample-recovery days	No crimps, cracks, or obstructions; no crossthreading	Replace as necessary.

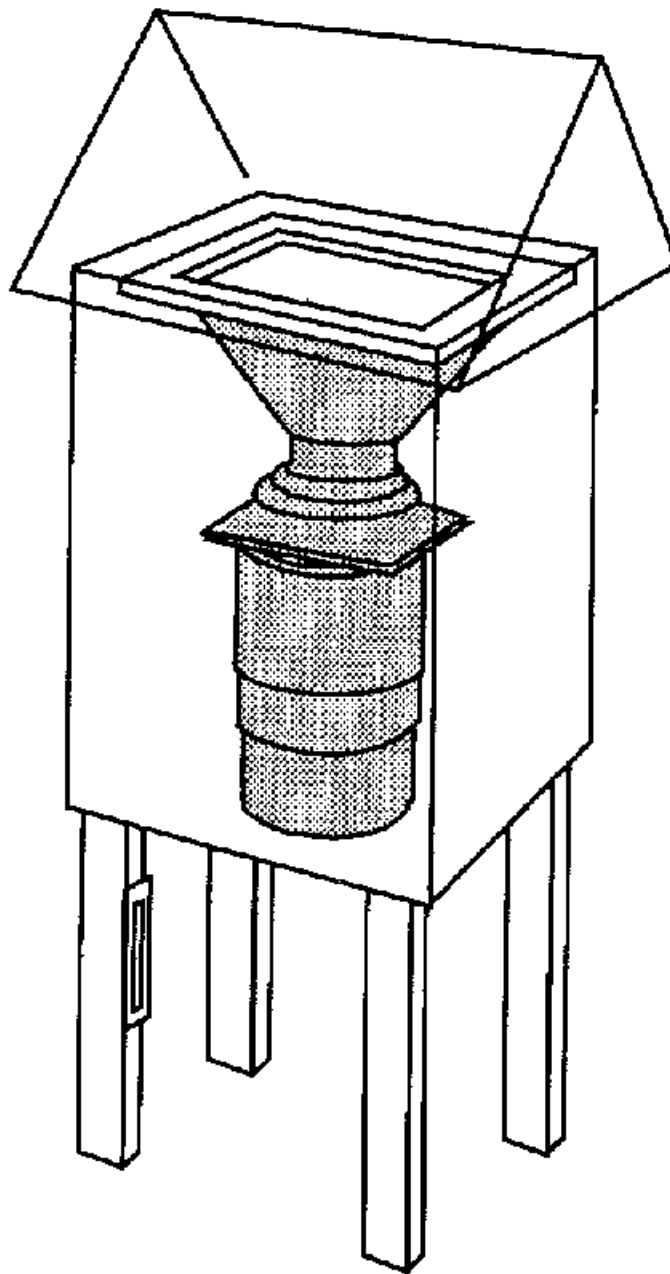


Figure 1. High-volume sampler with shelter.

Hi Volume Sampler in Shelter

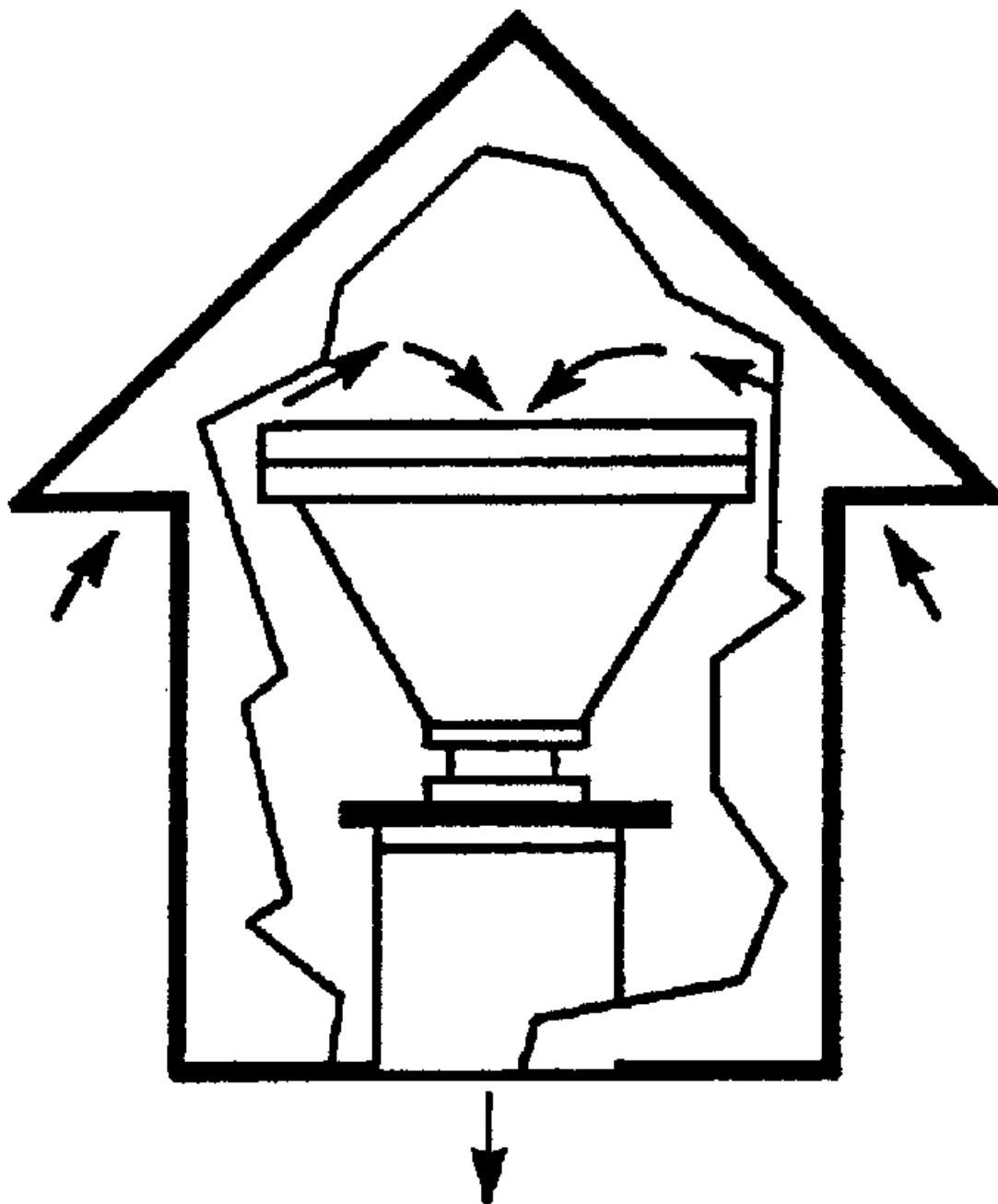


Figure 2. Inlet to EPA approved high volume sampler.

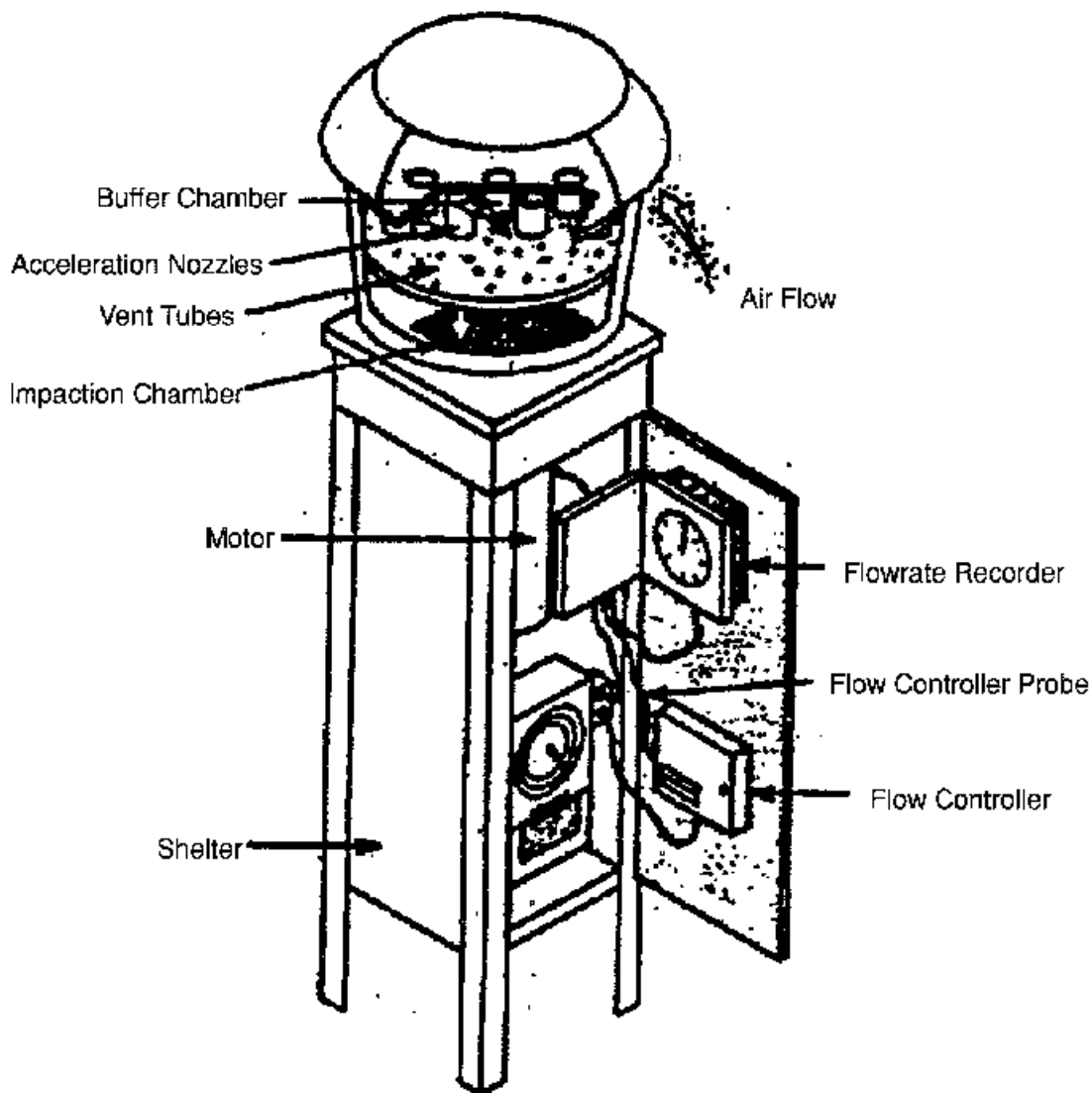


Figure 3. High-volume sampler with mass flow controller and impactor design size select inlet.

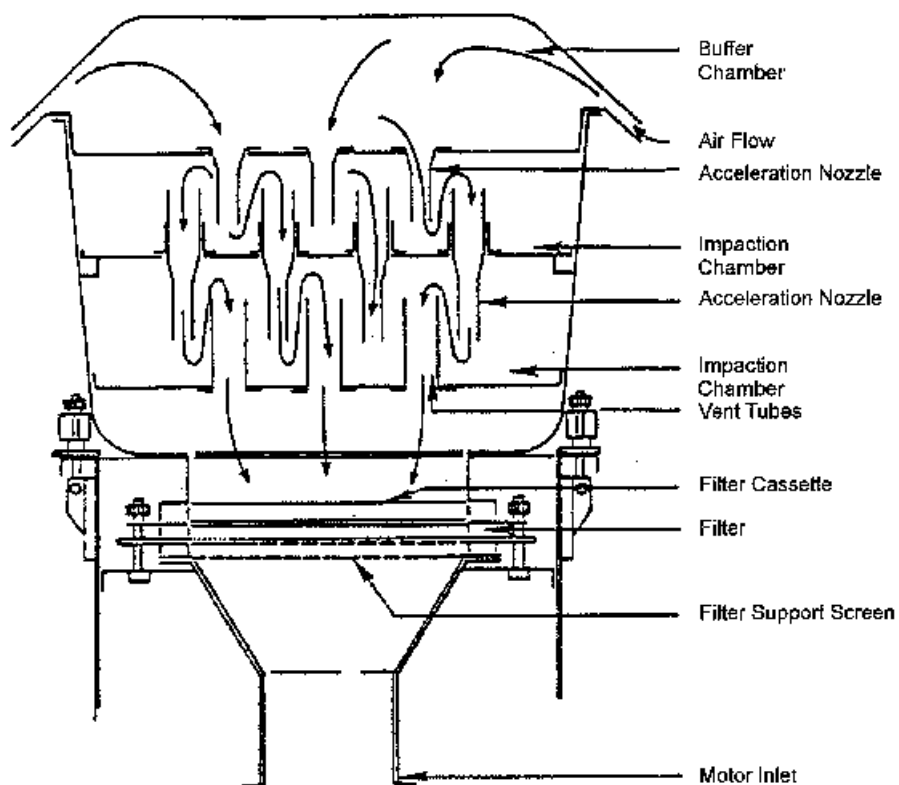


Figure 4. Schematic diagram of an impactor inlet for size select sampling for particulate matter.

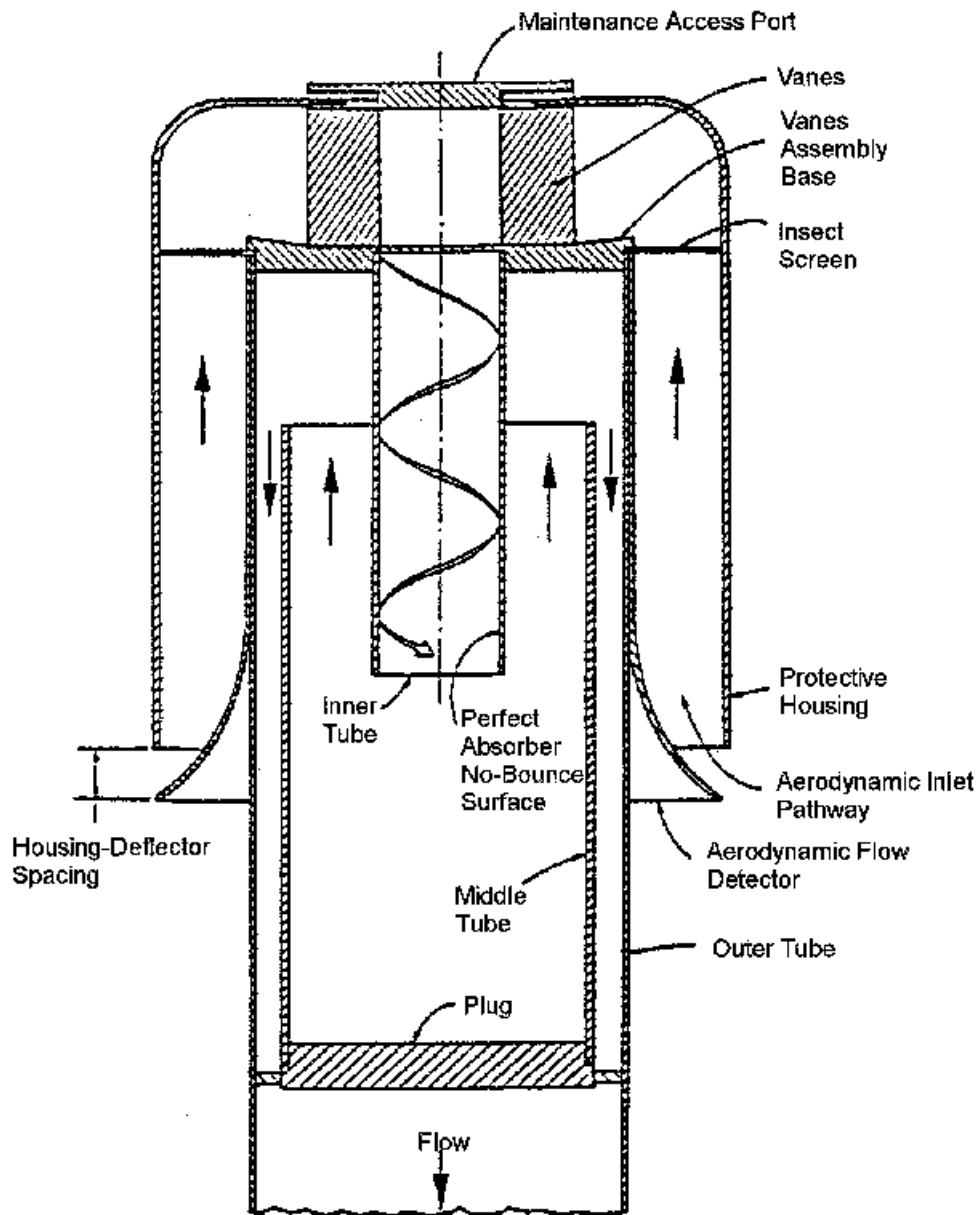


Figure 5. Schematic diagram of a cyclonic inlet for size select sampling for particulate matter.

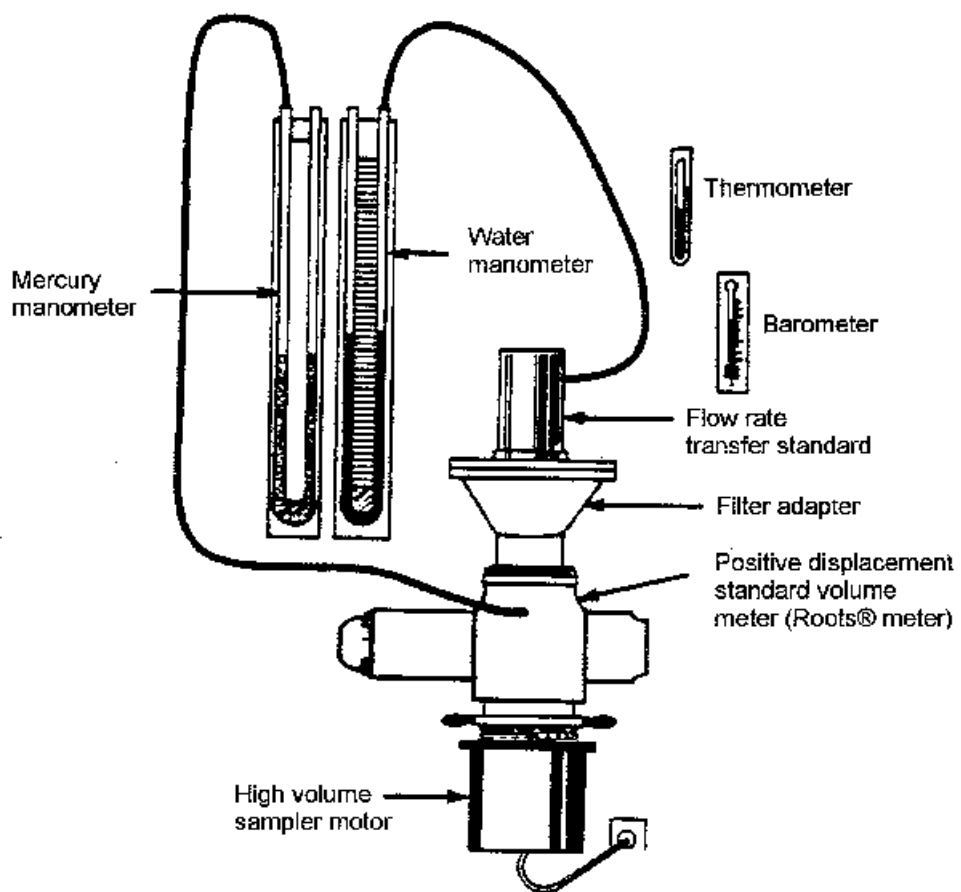


Figure 6. Flow rate transfer standard calibration setup.

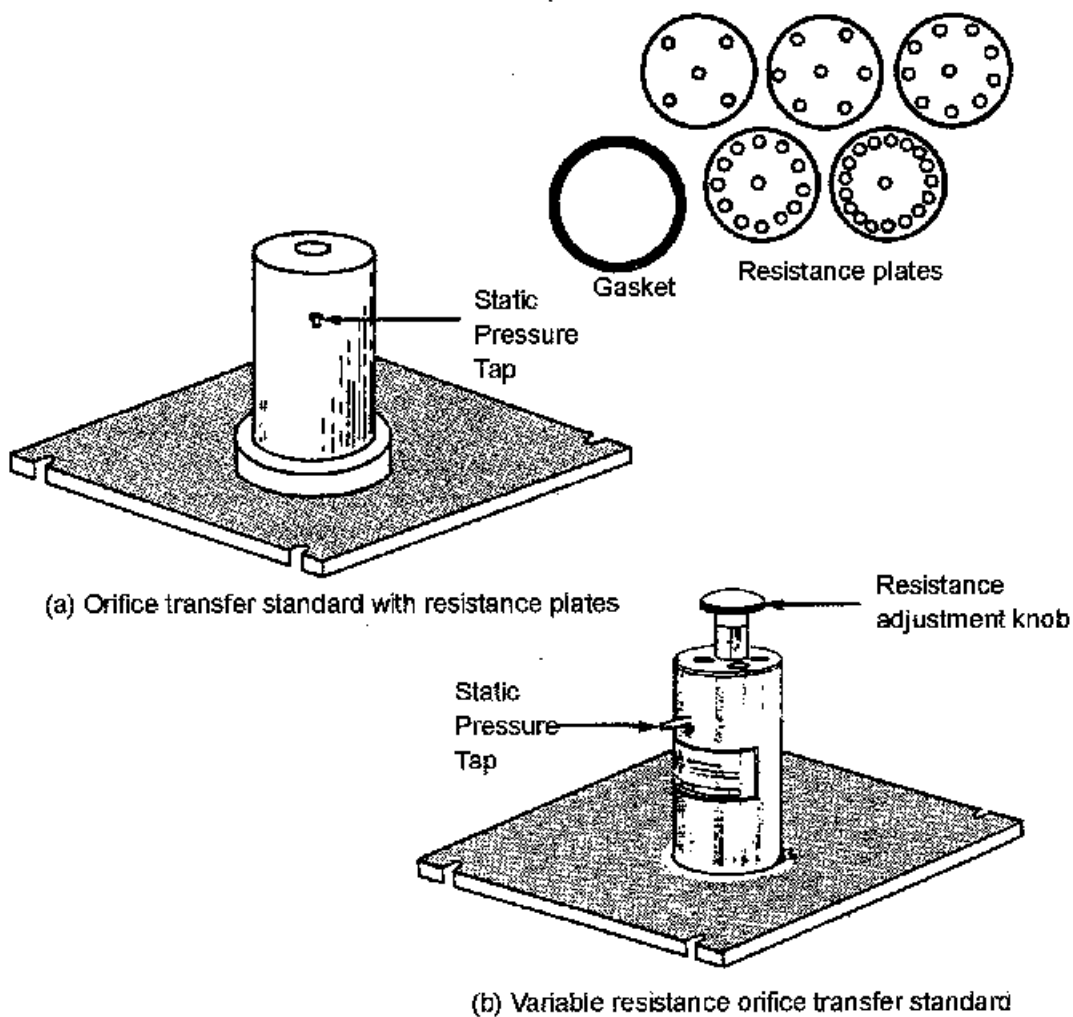


Figure 7. Typical orifice-type flow rate transfer standards.

ORIFICE TRANSFER STANDARD CERTIFICATION WORKSHEET						
Date:		Roots meter S/N:		Pa:		K
Operator:		Orifice S/N:		Pa:		mm Hg
Plate or Volts AC	Initial Volume	Final Volume	Δ Vol.	ΔTime (min)	ΔHg (mm)	ΔH ₂ O (in.)
DATA TABULATION						
Vstd	(x-axis) Qstd	(y-axis) [ΔH ₂ O (Pa/Pa)] ^{1/2}	Va	(x-axis) Qa	(y-axis) [ΔH ₂ O (Ta/Pa)] ^{1/2}	
	m =			m =		
	b =			b =		
	r =			r =		
CALCULATIONS						
Vstd = Δ Vol [(Pa - ΔHg)/760] (298/Ta) Qstd = Vstd/ΔTime y = mx + b			Va = ΔVol [(Pa - ΔHg)/Pa] Qa = Va/ΔTime y = mx + b			
For subsequent flow rate calculations:						
Qstd = { [ΔH ₂ O (Pa/Pa)] ^{1/2} - b } {1/m}			Qa = { [ΔH ₂ O (Ta/Pa)] ^{1/2} - b } {1/m}			
*NOTE: For PM10 monitoring, a calibration curve corrected to standard conditions is optional.						

Figure 8. Example orifice transfer standard certification worksheet.

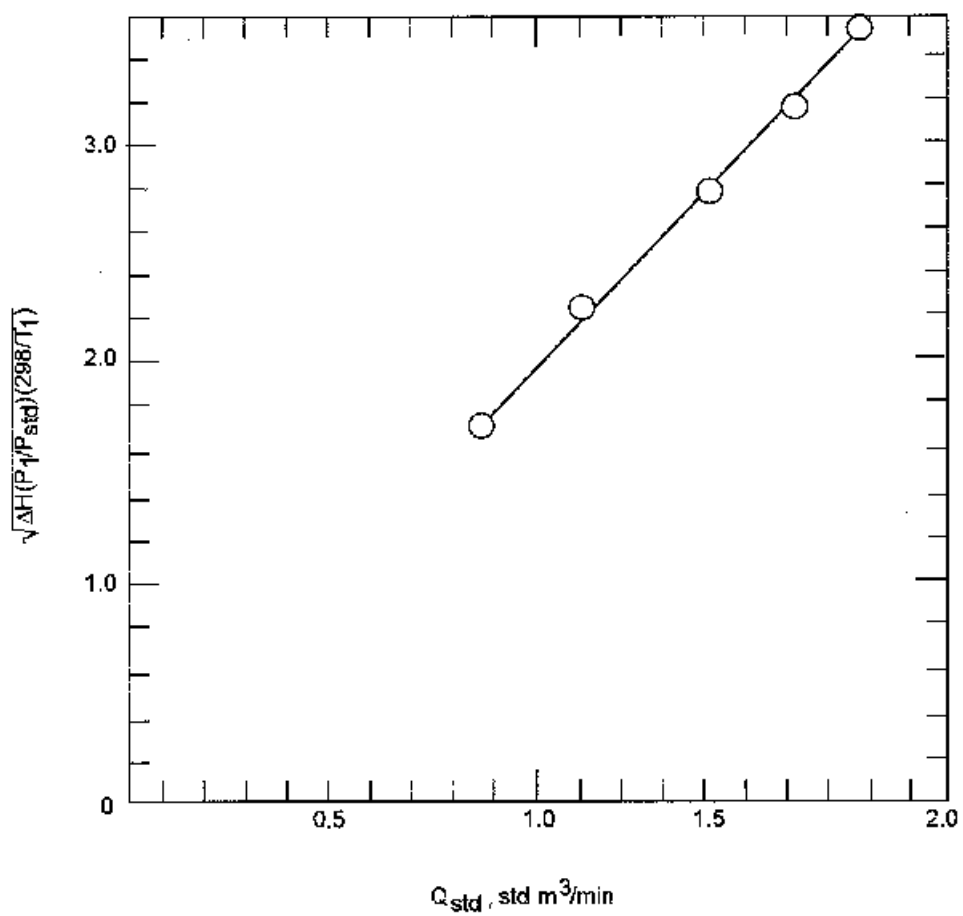


Figure 9. Typical calibration curve for a flow rate transfer standard.

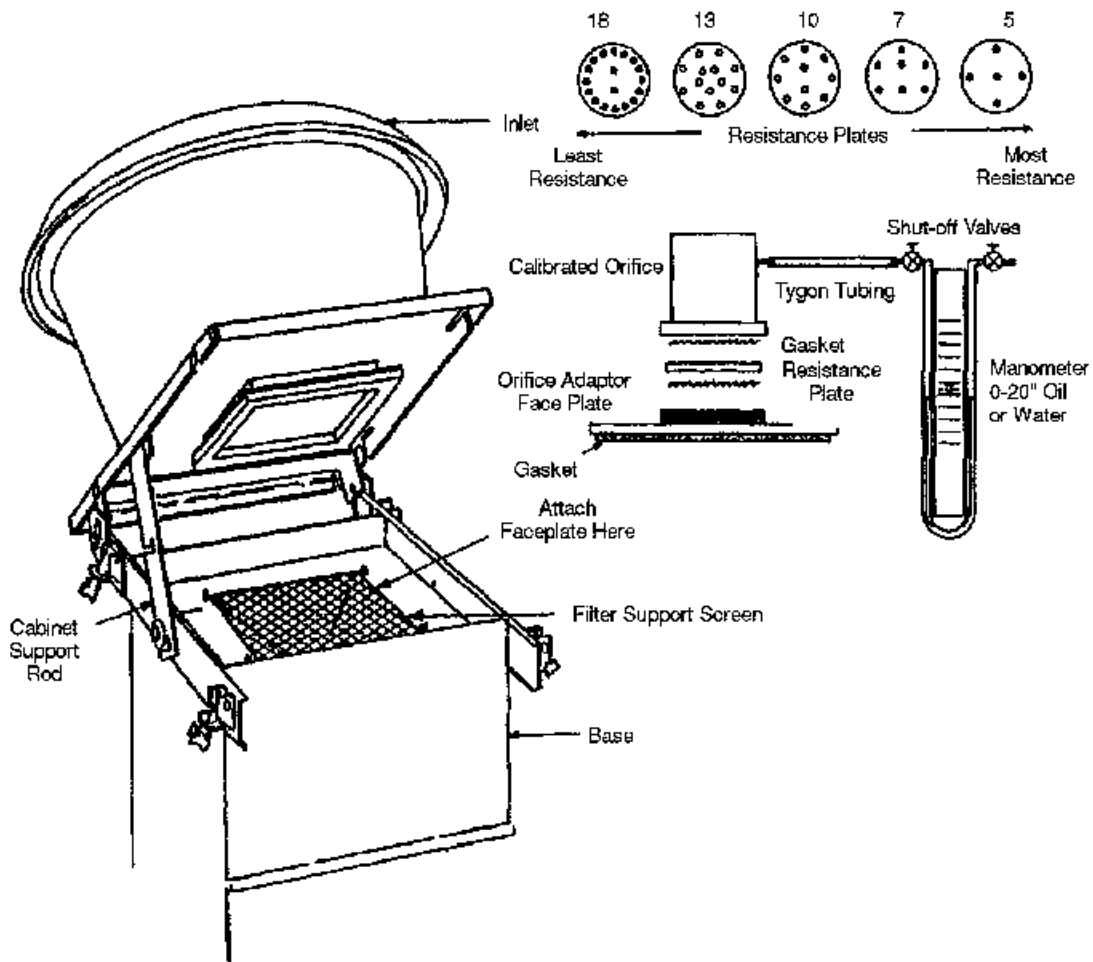


Figure 10. Typical calibration set-up for a mass flow controller (MFC).

MFC SAMPLER CALIBRATION DATA SHEET

Station Location _____ Date _____ Time _____

Sampler Model _____ S/N _____ Operator _____

Pa _____ mm Hg. Ta _____ °C _____ K. Unusual conditions: _____

Ps _____ mm Hg. Ts _____ °C _____ K. (*seasonal average Ta and Pa)

Orifice S/N _____ Orifice Calibration Date _____

Orifice calibration relationship: m = _____ b = _____ r = _____

Plate Number	Total ΔH ₂ O (in.)	X-Axis = Qa (orifice) flow rate ^a (m ³ /min)	Sampler ΔPex (in. H ₂ O) [or I for flow recorders]	Y-Axis = Sampler ΔPext ^b [or It for flow recorders] ^c

^aQa = $\{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\} (1/m)$
^bΔPext = $\{\Delta Pex(Ta + 30)/Pa\}^{1/2}$
^cIt = $\{I[(Ta + 30)/Pa]^{1/2}$ if a flow recorder is used

Sampler Calibration Relationship (Qa on x-axis; ΔPext or It on y-axis):
 ΔPext = m[Qa (Orifice)] + b or It = m[Qa(Orifice)] + b
 m = _____ b = _____ r = _____

For subsequent calculation of sampler flow rate:
 $\overline{Qa} = \{[(\text{mean } \Delta Pex)(Tav + 30)/Pav]^{1/2} - b\} (1/m)$
 or $\overline{Qa} = \{[\text{mean } I(Tav + 30)/Pav]^{1/2} - b\} (1/m)$

Set point flow rate (SFR) _____ Sampler set point (SSP) _____
 SFR = 1.13 (Ps/Pa) (Ts/Ts) SSP = $[Pa/(Ta + 30)] [m(SFR) + b]^2$
 or SSP = $[Pa/(Ta + 30)]^{1/2} [m(SFR) + b]$ for flow recorders

Figure 11. Example MFC sampler calibration data sheet.

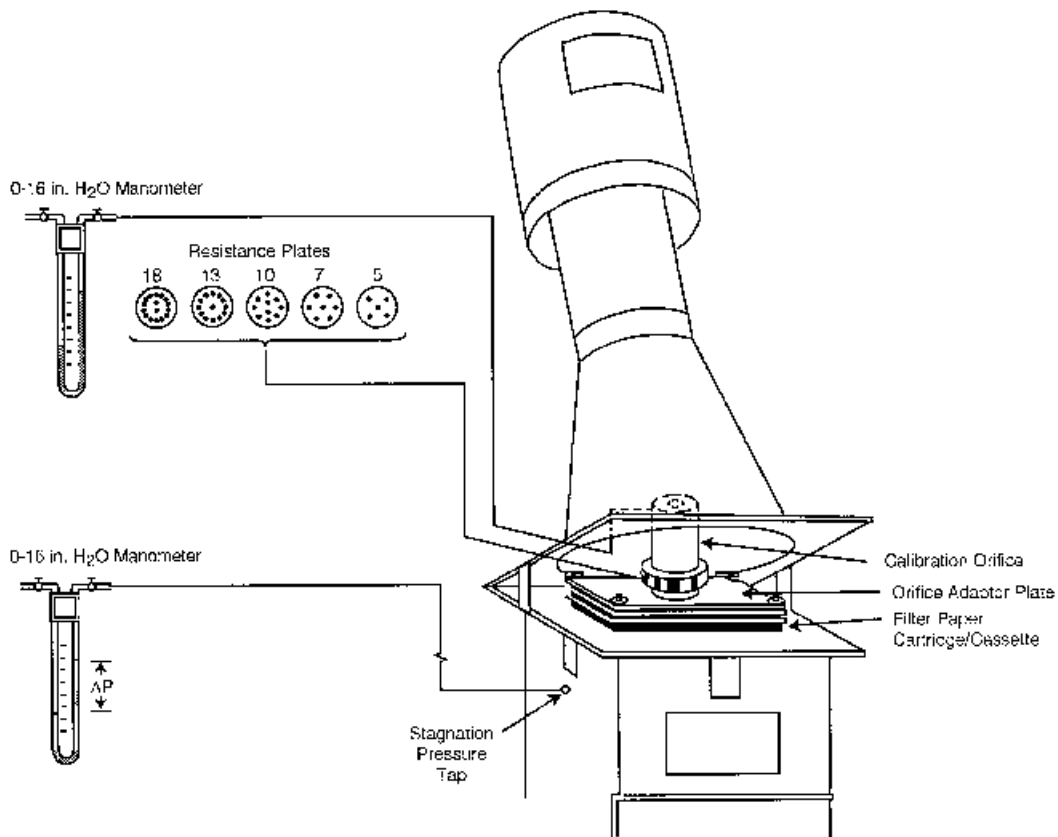


Figure 12. Calibration of a typical volumetric flow controller (VFC).

VFC SAMPLER CALIBRATION DATA SHEET						
Station Location _____		Date _____		Time _____		
Sampler Model _____		S/N _____		Operator _____		
Pa _____ mm Hg.		Ta _____ °C		K, Unusual Conditions _____		
Orifice S/N _____		Orifice Calibration Date _____				
Orifice Calibration Relationship: $m =$ _____ $b =$ _____ $r =$ _____						
Plate No.	ΔH_2O (in.)	ΔP_{stg} (mm Hg) ^a	$P1 = Pa - \Delta P_{stg}$ (mm Hg)	$P1/Pa$ (mm Hg)	Q_a (orifice) flow rate ^b (m ³ /min)	Q_s (orifice) $[Ta]^{1/2}$
Operational Flow Rate						

^amm Hg = 25.4 (in. H₂O/13.6)

^b Q_a (orifice) = $1/m \{[(\Delta H_2O) (Ta/Pa)]^{1/2} - b\}$

^c% Difference = $\left[\frac{Q_a \text{ (sampler)} - Q_a \text{ (orifice)}}{Q_a \text{ (orifice)}} \right] \{100\}$

Sampler Calibration Relationship

Lookup Table Validated (i.e., % difference < 4)

New calibration relationship:

$X = \frac{Q_a \text{ (orifice)}}{[Ta]^{1/2}}, Y = (P1/Pa)$

$m =$ _____ $b =$ _____ $r =$ _____

For subsequent calculation of sampler flow rate:

$Q_a = \{[P1/Pa - b][Ta]^{1/2}\} \{1/m\}$

Operational Flow Rate _____ m³/min

Q_s (Orifice)	Q_a (sampler) (Lookup Table)	% Difference ^c

Figure 13. Example VFC sampler calibration data sheet.

MFC SAMPLER FIELD DATA SHEET	
Station Location _____	Date _____ SAROAD# _____
Sampler Model _____	S/N _____
Filter ID No. _____	Pav _____ mm Hg. Tav _____ °C _____ K
Sampler Manometer Readings	Flow Recorder Readings
Initial ΔPex _____ in. H ₂ O	Mean I _____
Final ΔPex _____ in. H ₂ O	
Mean ΔPex _____ in. H ₂ O	
Sampler Calibration Relationship: m = _____ b = _____ r = _____	
\bar{Q}_a _____ m ³ /min	Elapsed Time _____ min
$\bar{Q}_a = \{[\text{mean } \Delta P_{ex} (T_{av} + 30)/P_{av}]^{1/2} - b\} \{1/m\}$	
$\bar{Q}_a = \{\text{mean } I [(T_{av} + 30)/P_{av}]^{1/2} - b\} \{1/m\}$ for flow recorders	
Operator _____	
Comments: _____	

Laboratory Calculations:	
\bar{Q}_{std} _____ std m ³ /min	Gross weight (Wg) _____ g
$\bar{Q}_{std} = \bar{Q}_a (P_{av}/P_{std}) (T_{std}/T_{av})$	Tare weight (Wt) _____ g
V_{std} _____ std m ³	Net Weight (Wn) _____ g
$V_{std} = (\bar{Q}_{std}) (\text{elapsed time})$	PM10 Concentration _____ μg/std m ³
	PM10 Concentration = (Wn) (10 ⁶)/Vstd

Figure 14. Example MFC sampler field data sheet.

VFC SAMPLER FIELD DATA SHEET	
Station Location _____	Date _____ SAROAD# _____
Sampler Model _____ S/N _____	
Filter ID No. _____ P _{av} _____ mm Hg, T _{av} _____ °C _____ K	
Relative Stagnation Pressure Readings	Absolute Stagnation Pressure
Initial ΔP _{stg} _____ mm Hg	P ₁ = _____ mm Hg
Final ΔP _{stg} _____ mm Hg	P ₁ = P _{av} - Average ΔP _{stg}
Average ΔP _{stg} = _____ mm Hg	
Average Stagnation Pressure Ratio (P ₁ /P _{av}) _____	
Average Flowrate (Q _a)* _____ m ³ /min	Elapsed Time _____ min
*Obtained from manufacturer's lookup table (or from alternate calibration relationship)	
Operator _____	
Comments: _____	

Laboratory Calculations:	
Q _{std} _____ Std m ³ /min	Gross Weight (W _g) _____ g
Q _{std} = Q _a (P _{av} /P _{std}) (T _{std} /T _{av})	Tare Weight (W _t) _____ g
V _{std} _____ std m ³	Net Weight (W _n) _____ g
V _{std} = (Q _{std}) (Elapsed Time)	PM10 Concentration _____ μg/std m ³
	PM10 Concentration = (W _n) (10 ⁶)/V _{std}

Figure 15. Example VFC sampler field data sheet.

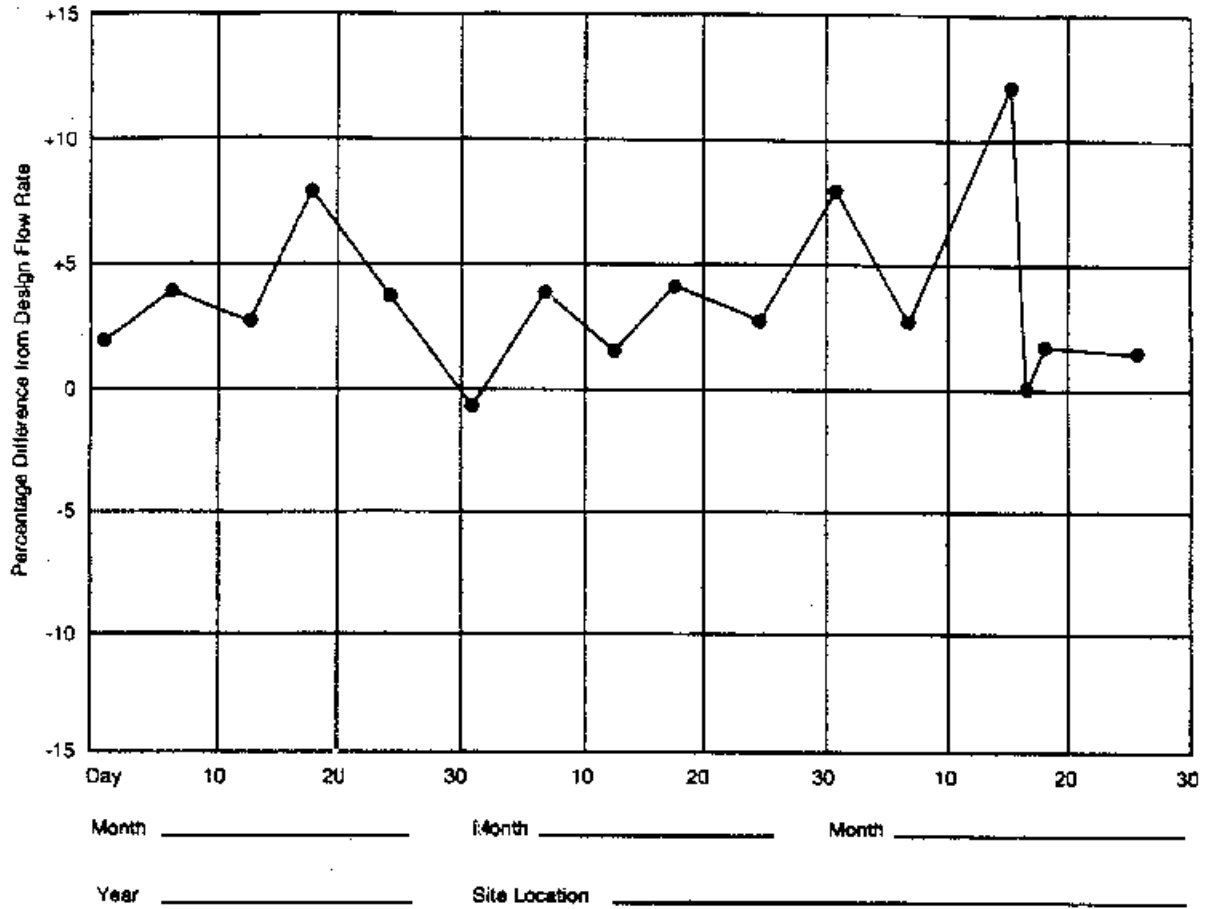


Figure 16. Field data control chart.

ATTACHMENT L-B

USEPA COMPENDIUM METHOD IO-3.1

**Compendium of Methods
for the Determination of
Inorganic Compounds
in Ambient Air**

Compendium Method IO-3.1

**SELECTION, PREPARATION
AND EXTRACTION OF
FILTER MATERIAL**

Center for Environmental Research Information
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268

June 1999

Method IO-3.1

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- James L. Cheney, U.S. Army Corps of Engineers, Omaha, NE
- Michael F. Davis, U.S. EPA, Region 7, KC, KS
- Joseph B. Elkins Jr., U.S. EPA, OAQPS, RTP, NC
- Robert G. Lewis, U.S. EPA, NERL, RTP, NC
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- William A. McClenny, U.S. EPA, NERL, RTP, NC
- Frank F. McElroy, U.S. EPA, NERL, RTP, NC
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

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Author(s)

- Avie Mainey, Midwest Research Institute, Kansas City, MO
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

Peer Reviewers

- David Brant, National Research Center for Coal and Energy, Morgantown, WV
- John Glass, SC Department of Health and Environmental Control, Columbia, SC
- Jim Cheney, U.S. Army Corps of Engineers, Omaha, NE
- Dewayne Ehman, Texas Natural Resource Conservation Committee, Austin, TX
- Gary Wester, Midwest Research Institute, Kansas City, MO
- Lauren Drees, U.S. EPA, NRMRL, Cincinnati, OH

DISCLAIMER

This Compendium has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Method IO-3.1
Selection, Preparation and Extraction of
Filter Material

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Chapter IO-3
CHEMICAL SPECIES ANALYSIS
OF
FILTER-COLLECTED SUSPENDED PARTICULATE MATTER (SPM)

Method IO-3.1
SELECTION, PREPARATION AND EXTRACTION OF
FILTER MATERIAL

1. Scope

1.1 This methodology consists of (1) filter media selection, (2) numbering and pre-field tare weighing of filters, (3) post-field final weighing of filters, (4) microwave or hot acid extraction, and (5) analysis for metal analysis by ICP, FAA, ICP/MS or GFAA.

1.2 Pre-field filters are conditioned in a room of constant humidity and temperature and are gravimetrically tared. After air samples have been collected, the filters are returned to the laboratory and conditioned as before and weighed. The final filter weight minus the tare weight is calculated. The procedure for the weighing of filters is based on 40 CFR 50, Appendix B, entitled "*Reference Method for the Determination of Suspended Matter in the Atmosphere (High-Volume Method)*."

1.3 After the post-field filter final weights have been obtained, the filter is subsampled by cutting a filter strip consisting of one-ninth of the overall filter and digested using a microwave or hot acid extraction technique; these extracts are then analyzed by one of many analytical techniques. The results are multiplied by a factor of 9 to obtain the actual total μg of each metal found on the entire 8" x 10" filter. Based upon the analysis of a blank filter, background metal concentration may be subtracted from the total metal concentration to get a net value. Therefore, the analytical results represent the total μg found on the 8" x 10" filter but do not represent the volume of air sampled.

1.4 Sectioning the filter for extraction is based on 40CFR50, Appendix B entitled "*Determination of Lead in Suspended Particle Matter Collected From Ambient Air*." The procedure for the microwave extraction is based on a method developed by EPA entitled *Microwave Extraction of Glass-Fiber Filters*, as identified in Section 2.2. This procedure has been modified for extracting quartz fiber filters.

2. Applicable Documents

2.1 ASTM Documents

- D4096 *Application of the High Volume Sample Method for Collection and Mass Determination of Airborne Particle Matter.*
- D1356 *Definition of Terms Related to Atmospheric Sampling and Analysis.*
- D1357 *Practice for Planning the Sampling of the Ambient Atmosphere.*
- D2986 *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test.*

2.2 Other Documents

- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: A Field Guide for Environmental Quality Assurance*, EPA-600/R-94/038a.
- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition)*, EPA-600/R-94/038b.
- *Reference Method for the Determination of Particulate Matter in the Atmosphere*, Code of Federal Regulations (40 CFR 50, Appendix J).
- *Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)*, Code of Federal Regulations (40 CFR 50, Appendix B).
- *Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air*, Federal Register 43 (194): 46258-46261.
- U. S. Environmental Protection Agency, *Microwave Extraction of Glass Fiber Filters*, Method Research and Development Division, RTP, NC 1989.

3. Apparatus

3.1 Equipment For Gravimetric Analysis

3.1.1 Controlled Temperature. Temperature between 15 and 30EC with less than ± 2 EC variation during equilibration period.

3.1.2 Controlled Humidity. Less than 50% relative humidity, constant within $\pm 5\%$

3.1.3 Analytical Balance. Sensitive to 0.1 mg, with weighing chamber designed to accept an unfolded 20.3 x 25.4 cm (8" x 10") filter.

3.1.4 Area Light Source. Similar to X-ray film viewer to backlight filters for visual inspection.

3.1.5 Numbering Device. Capable of printing identification numbers on the filters before they are placed in the filter conditioning environment if not numbered by the supplier.

3.1.6 Hygrothermograph. Capable of recording temperature and relative humidity in the weighing room.

3.2 Microwave Digestion Apparatus and Materials

3.2.1 Microwave Digestive System and Capping Station. With programmable power settings up to 600 watts, best source.

[Note: Commercial kitchen or home-use microwave should NOT be used for digesting samples. The oven cavity must be corrosion resistant and well ventilated. All electronics must be protected against corrosion for safe operation.]

3.2.2 PFA Teflon® Digestion Vessels. Capable of withstanding pressures of up to 120 psi. Pressure vessels capable of controlled pressure relief at pressures exceeding 120 psi (60-120 mL capacity), best source.

3.2.3 Teflon® PFA Overflow Vessel. Double ported (60-120 mL capacity), best source.

3.2.4 Rotating Table. Uniform exposure of samples within the oven.

3.2.5 Volumetric Glassware. 50-100 mL capacity (Class A borosilicate).

3.2.6 Bottles, Linear Polyethylene or Polypropylene with Leakproof Caps, for Storing Samples. Teflon® bottles for storing multielement standards (500 mL, 125 mL, and 30 mL), best source.

- 3.2.7 Centrifuge Tubes.** Oak Ridge polysulfone tubes with screw caps of polypropylene, 30 mL.
- 3.2.8 Nylon or Teflon® 0.45 µm Syringe Filters.** Acrodisc® No. 4438 or equivalent and syringes for rapid nonmetals contributing filtering.
- 3.2.9 Sterile Polypropylene Tubes with Screw Caps of Polypropylene, 15 mL Capacity.** Best source.
- 3.2.10 Pipette.** Automatic dispensing with an accuracy of setting 0.1 mL or better and repeatability of 20 FL, Grumman Automatic Dispensing Pipette, Model ADP-30DT or equivalent.
- 3.2.11 Particle Mask.** 3M, No. 8500, to be worn while cutting and handling glass-fiber filters.
- 3.2.12 Template.** Aid in sectioning the glass fiber filter. Federal Register 43 (194): 46258-46261.
- 3.2.13 Pizza Cutter, Thin Wheel.** Clean razor blade (< 1 mm).
- 3.2.14 Vortex Mixer.** VWR2 variable speed or equivalent.
- 3.2.15 Hydrochloric Acid.** Baker Instra-Analyzed, concentrated (sp.gr.1.19) or equivalent, for preparing samples.
- 3.2.16 Nitric Acid.** Baker Instra-Analyzed, concentrated (sp.gr.1.41) or equivalent, for preparing samples.
- 3.2.17 ASTM Type I Water.** ASTM D193.
- 3.2.18 Extraction Solution (5.55% HNO₃/16.75% HCl).** Prepare by adding in ~ 500 mL of Type I DI water, 55.5 mL of concentrated HNO₃, and 167.5 mL of concentrated HCl, dilute to one liter with Type I DI water.

3.3 Hot Acid Extraction Apparatus and Materials

3.3.1 Thermolyne Model 2200 Hot-Plate or Equivalent.

[Note: Temperature of the extracts may be monitored by the use of a beaker containing a thermometer and similar reagents as the samples.]

- 3.3.2 Volumetric Glassware.** 50-100 mL capacity (Class A borosilicate).
- 3.3.3 Bottles, Linear Polyethylene or Polypropylene with Leakproof Caps, for Storing Samples.** Teflon® bottles for storing multielement standards (500 mL, 125 mL, and 30 mL).
- 3.3.4 Centrifuge Tubes.** Polypropylene or Oak Ridge polysulfone tubes with screw caps of polypropylene, 30 mL (Nalgene 3119-0050/3115-0030 or equivalent).
- 3.3.5 Nylon or Teflon® 0.45 µm Syringe Filters.** Acrodisc® No. 4438 or equivalent and syringes for rapid nonmetals contributing filtering.
- 3.3.6 Sterile Polypropylene Tubes with Screw Caps of Polypropylene, 15 mL capacity.** Falcon Model No. 2099 or equivalent.
- 3.3.7 Pipette.** Automatic dispensing with an accuracy of setting 0.1 mL or better and repeatability of 20 FL. (Grumman Automatic Dispensing Pipette, Model ADP-30DT or equivalent).
- 3.3.8 Particle Mask.** 3M, No. 8500. To be worn while cutting and handling glass-fiber filters.
- 3.3.9 Vortex Mixer.** VWR2 variable speed or equivalent.
- 3.3.10 Hydrochloric Acid.** Baker Instra-Analyzed, concentrated (36.5%-38%/12.3 M) or equivalent, for preparing samples.
- 3.3.11 Nitric Acid.** Baker Instra-Analyzed, concentrated (70% 16M) or equivalent, for preparing samples.
- 3.3.12 ASTM Type I Water.** ASTM D193.

4. Filter Medium Selection

4.1 Introduction

4.1.1 In general, the filter medium depends on the purpose of the test. For any given standard test method, the appropriate medium will be specified. However, it is important to be aware of certain filter characteristics that can affect selection and use.

4.1.2 Selecting a filtration substrate for time-integrated SPM monitoring must be made with some knowledge of the expected characteristics and a pre-determined analytical protocol. For any given standard test method, the appropriate medium will normally be specified.

4.1.3 In high-volume sampling, four types of filter material to capture SPM are commonly used. They include cellulose fiber, quartz/glass fiber, mixed fiber, and membrane filter types. Selecting a filter depends upon variables such as background metal content, artifact formation, and affinity for moisture. The basic characteristics of the types of filter material used in high volume are outlined in Table 1 sampling. Useful filter properties are described in Table 2. Several characteristics are important in the selection of filter media. They are:

- **Particle Sampling Efficiency.** Filters should remove more than 99% of SPM drawn through them, regardless of particle size or flow rates.
- **Mechanical Stability.** Filters should be strong enough to minimize leaks during sampling and wear during handling.
- **Chemical Stability.** Filters should not chemically react with the trapped SPM.
- **Temperature Stability.** Filters should retain their porosity and structure during sampling.
- **Blank Correction.** Filters should not contain high concentrations of target compound analytes.

Quartz fiber filter medium is most widely used for determining mass loading. Weight stability with respect to moisture is an attractive feature. Quartz fiber filters provide high efficiency and collect airborne particles of practically every size and description. Typical characteristics of quartz fiber filters are (1) a fiber content of high purity quartz, (2) a binder of below 5% (zero for binderless types), (3) a thickness of approximately 0.5 mm, (4) a surface with no pinholes, and (5) an allowance of no more than 0.05% of smoke particles to pass through the filter at a pressure of 100mm of water with a flow rate of 8.53 m/min (28 ft/min), as determined by ASTM-D2986, *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test*.

Particulate matter collected on quartz fiber filters can be analyzed for many constituents. If chemical analysis is anticipated, binderless filters should be used. Glass is a commercial product generally containing test-contaminating materials; therefore, appropriate background corrections should be made. Background concentration of various metals associated with different grades of quartz fiber filters are documented in Table 3.

4.1.4 Silica fiber filters are used when it may be required or desirable to use a mineral fiber filter, which may later be extracted by strong reagents. These fibers are usually made by leaching glass fibers with strong mineral acids followed by washing with deionized water. The fibers are rather weak but can be formed into filter sheets using little or no binder. These filters have been recently developed and are commercially available.

4.1.5 For some purposes, airborne particles may be collected on cellulose fiber filters. Cellulose low-ash filters are especially useful when the filter is to be destroyed by ignition or chemical digestion. However, these filters have higher flow resistance (lower sampling rate) and have been reported to have much poorer

collection efficiency than the glass fiber media. Furthermore, cellulose is very sensitive to moisture conditions, and even with very careful conditioning before and after sampling, accurately weighing the collected particles is difficult. The filter should be enclosed in a lightweight metal can with a tight lid and weighed.

4.1.6 As documented in the 40 CFR Part 58, Appendices A and B, identify the filter specifications when used as part of the *Federal Reference Method for Particulate Matter in Ambient Air*. These specifications include (1) a quartz-fiber, nonhygroscopic filter, (2) a size of approximately 8" x 10", (3) an exposure area of approximately 63 in.², (4) a 99% collection efficiency as measured by ASTM-2986 (DOP test) for particles 0.3 μm diameter, (5) a pressure drop range of 42-54 mm Hg at a flow rate of 1.5 m³/min through the nominal exposed area, (6) a pH of 6 to 10, and (7) a maximum weight integrity of 2.4 mg.

4.2 Visual Filter Inspection

4.2.1 After purchased, all filters must be visually inspected for defects, and defective filters must be rejected if any are found. Batches of filters containing numerous defects should be returned to the supplier.

4.2.2 The following are specific defects to look for:

4.2.2.1 Pinhole. A small hole appearing as a distinct and obvious bright point of light when examined over a light table or screen, or as a dark spot when viewed over a black surface.

4.2.2.2 Loose material. Any extra loose material or dirt particles on the filter that must be brushed off before the filter is weighed.

4.2.2.3 Discoloration. Any obvious visible discoloration that might be evidence of a contaminant.

4.2.2.4 Filter nonuniformity. Any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity across the face of the filter.

4.2.2.5 Other. A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

4.2.3 Visually inspect each filter in front of an area light and observe for any specific defects listed above.

4.2.4 Use a renumbering stamp to code the filter on its noncollection side with a 7-digit code before tare weighing. The noncollection side of the filter is designated by the manufacturer printed number and by a mesh texture. The number code might be as follows:

Example: Filter Number Code = **9622001**

First 2 digits = yr, such as **96** for 1996

Third digit = project, such as **2**

Fourth digit = filter type, such as **2** for 8" x 10" quartz fiber, Whatman QMA type

Last 3 digits = filter number, such as **001**

5. Gravimetric Determination

5.1 Introduction

5.1.1 The filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain. The total volume of air sampled corrected to EPA standard conditions (25°C, 760 mm Hg) is determined from the measured flow rate and the sampling time. The concentration of TSP matter in the ambient air is computed as the mass of collected particles divided by the volume of air sampled (corrected to standard conditions) and expressed in μg/std m³ (see Inorganic Compendium Method IO-2.4). For samples collected at temperatures and pressures significantly different than standard conditions, the corrected

concentrations may differ substantially from actual concentrations Fg/m^3 , particularly at high elevations. The actual particulate matter concentration can be calculated from the corrected concentration using the actual temperature and pressure during the sampling period.

5.1.2 Verify that the weighing room conditions are within the limits. Filter equilibrium and weighing should be performed under controlled atmospheric conditions--a temperature of 25 ± 10 EC and a relative humidity $< 50\%$ (normally $50 \pm 5\%$ humidity).

5.1.3 Use the results from the motorized psychrometer to verify the temperature and relative humidity indicated by the hygrothermograph. Record the psychrometer values on the strip chart, along with the date, time, and your initials.

[Note: For traceability purposes, document your initials and full name in the front of the weighing room notebook.]

5.1.4 Record the room equilibration data on the Weighing Room Atmospheric Condition Form (see Table 4).

5.2 High Volume Filter Weighing Procedure

5.2.1 Filter Handling Procedure.

5.2.1.1 Filters should only be handled with finger cots or vinyl (nonpowdered) gloves. This procedure applies to filter handling in the field as well as in the weigh room.

5.2.1.2 Avoid using metal tweezers since the filters later will be used for metals analysis. When handling filter with gloved fingers or with any type of tweezers, avoid touching the sampled area.

5.2.2 Initial Weighing of High Volume Filter.

5.2.2.1 Upon receipt of new high volume filters (8" x 10" quartz fiber), take them to the climate controlled room, remove the paper and plastic envelope (wearing clean plastic gloves), place each on edge in a clean metal file rack, and cover with clean white paper towels.

5.2.2.2 Allow the filters to equilibrate in the metal file rack in the weighing room atmosphere for at least 24 h. Humidity and temperature must be within Federal Reference method specification, (i.e., $< 50\%$ and 15-35EC, respectively).

5.2.2.3 Zero the high volume balance before weighing.

5.2.2.4 Manually calibrate the balance. However, checks against two working NIST traceable weights (Class S) standards should be conducted before the daily weighing. If the difference between the traceable weights is more than 0.5 mg, do not use the balance until it has been repaired.

5.2.2.5 Record the results on the Weighing Balance Check Form (see Table 5).

5.2.2.6 Weigh each filter and record filter numbers and tare weights on the Filter Weighing Form (see Table 6).

5.2.2.7 Return the weighed filters to the plastic and paper envelopes.

5.2.2.8 Weigh filters in lots of approximately 100, if possible. After every tenth weighing, recheck the zero of the balance. The balance response should be ± 1 mg from 0. All differences should be corrected. Any difference exceeding 1 mg requires reweighing the previous ten filters. Any filter weight outside the normal range of 3.5-5.0 g requires immediate investigation. In addition, after every tenth filter weighing, the analyst should review at least one of the working standards. Once again, if this measurement disagrees from the verified value by more than 0.5 mg, reweigh the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both

working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 0.5 mg, then all weighings from the previous acceptable check must be repeated.

5.2.2.9 A second analyst should reweigh 10% of the filters. If the difference between the weights is less than 1.0 mg, the results are acceptable.

5.2.2.10 If the difference is greater than this limit, wait another 24 h and reweigh them.

5.2.2.11 If the results are still outside acceptable limits, wait another 24 h and reweigh them again. Then report the last reweigh values as the pre-field tare weights.

5.2.3 Final Weighing of High Volume Filter.

5.2.3.1 Exposed filters should be logged into the laboratory computer and received in individual manila folders, with computer printed identification labels affixed. No exposed filter should be touched until this label is affixed.

5.2.3.2 Condition all filters in the manner specified by the Federal Reference Method, as documented in Sections 5.1.2 and 5.2.2.

5.2.3.3 Weigh all filters according to the Tare Weighing Procedure in Section 5.2.2. Record final weights on the Filter Weighing Form (see Table 6).

5.2.3.4 For filters not to be analyzed, put an asterisk in the space preceding the four-letter code. Leave this space blank for samples to be analyzed. Sign and date the forms.

5.2.3.5 Archive asterisked high volume filters.

5.2.3.6 Have a second analyst reweigh 10% of the filters and verify that the weights have not changed.

- If the difference between the weights is less than 2.0 mg, the results are acceptable. Use the results from the first weighing.
- If the difference is greater than this limit, reweigh 100% of that lot and use the last reweigh weight.

5.2.3.7 Calculate and report the particulate matter concentrations as:

where:

$$\text{SPM} = \frac{(W_f - W_i) \times 10^6}{V_{\text{std}}}$$

SPM = mass concentration of suspended particulate matter (TSP or PM₁₀), µg/std m³.

W_i = initial weight of clean filter, g.

W_f = final weight of exposed filter, g.

V_{std} = air volume sampled, converted to standard conditions (25°C and 760 mm Hg), std m³.

10⁶ = conversion of g to µg.

5.3 Dichotomous and Partisol® Filter Weighing Procedure

5.3.1 Initial Weighing of Dichotomous or Partisol® Filters.

5.3.1.1 Fabric filters, 37-mm or 47 mm (as appropriate for the samples) in diameter, with a circumferential plastic reinforcing ring are usually supplied in small boxes. Open the boxes in the climate-controlled room under conditions suitable for high volume weighing. Cover with a clean paper towel and allow to equilibrate for 24 h.

5.3.1.2 Weigh filters on a Mettler microbalance; each balance is identified by a balance number.

5.3.1.3 Assign each balance a block of 7-digit sample numbers to be used sequentially. Assign a sample number to each filter when it is tared.

[Note: Inaccuracies in this aspect of the procedure will cause irremedial sample loss.]

5.3.1.4 Turn on the microbalance and allow it to warmup for at least 15 min. If the balance is used daily, leave it on at all times.

5.3.1.5 Manually calibrate the microbalance with two working NIST traceable weight (Class S) standards (for example, a 100-mg standard and a 200-mg standard). If the difference between the traceable weights is more than 3 µg, reweigh the working standards. Record the Results on the Weighing Balance Check Form (see Table 7).

5.3.1.6 If the values still disagree, troubleshoot and take appropriate corrective action, which may include (1) recertifying the working standards against the laboratory primary standards, and/or (2) having a service technician repair the microbalance.

5.3.1.7 Using clean nonserrated tweezers that will not damage the filter, remove the filter from the Lexan jig or filter cassette and place it on the weighing pan. Turn the release lever to "1" and dial in tare weights until a reading between 0.000 and 7.000 is obtained. Allow the reading to stabilize (which may require 2 to 4 min). Record the reading and the dialed-in tare weight on the Filter Weighing Form (see Table 8). Return the release lever to "0" and remove the filter from the weighing pan.

[Note: Do not use metal tweezers.]

5.3.1.8 Place a white label on a clean 50-mm diameter plastic petri dish (tight fitting lid type).

5.3.1.9 Assign a sample number to each filter (from those assigned to that balance), taking extreme care to avoid duplication or missed numbers.

5.3.1.10 Record the assigned sample number on the petri dish label, leaving sufficient room for one more letter to be written following the number. Do not record the balance number on this label.

5.3.1.11 Record the balance number, the assigned sample number, the dialed-in tare weight, and the digital-displayed tare weight on the sample form. Number each sheet of the form sequentially in the upper right-hand corner. Write "Tare Weight, Dichot or Partisol® Filters" on the top of each sheet. When bound, these forms may serve as the laboratory notebook.

5.3.1.12 Place the weighed filter in its numbered petri dish for future use.

5.3.1.13 After every tenth filter weighing, the analyst should check the "zero" and reweigh at least one of the working standard. Record the measurement on the Weighing Balance Check Form. Once again, if this measurement disagrees from the verified value by more than 3 µg, reweigh the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 3 µg then all weighings from the previous acceptable check must be repeated.

5.3.1.14 At the end of the weighing session, at least 10% of the filters should be reweighed by a second analyst. Record the replicate measurement on the Filter Weighing Form (see Table 6). If the replicate measurement disagrees from the original measurement by more than 15 µg, reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

5.3.1.15 Return the filter to the filter-handling container, replace the lid, and return it to conditioning chamber to protect it from contamination prior to sampling.

5.3.2 Final Weighing of Dichotomous or Partisol® Filter.

5.3.2.1 Filters should be returned from the field with a computer printed label affixed to the petri dish. The label should contain a five-character identification code that is different from the original sample number, a balance ID, the balance tare, and other information. All filters should be accompanied by extra labels. Some will have the words "To Be Analyzed" on the labels. The filter in each petri dish should rest in a Lexan jig or filter cassette.

5.3.2.2 Weigh each filter on the balance on which its tare weight was obtained. In the climate-controlled room, group the filters according to recorded balance numbers. Open the petri dishes, making certain that lids are placed under the bottoms and that no mixup occurs. Cover with a clean white paper towel and allow to equilibrate.

5.3.2.3 Repeat Section 5.3.1.4 to 5.3.1.6 of the filter tare weighing procedure.

5.3.2.4 Using clean, nonserrated tweezers that will not damage the filter, remove the filter from the Lexan jig or filter cassette and place it on the weighing pan. Dial in the tare weight recorded on the information label and turn the release lever to "1." Allow the reading to stabilize (which may require 2 to 4 min). Record the reading and the dialed-in tare weight. Return the release lever to "0" and remove the filter from the weighing pan.

5.3.2.5 After every tenth filter weighing, the analyst should check the "zero" and reweigh at least one of the working standards. Record the measurement on the Weighing Balance Check Form. Once again, if this measurement disagrees from the verified value by more than 3 ug, review the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 3 µg, then all weighings from the previous acceptable check must be repeated.

5.3.2.6 At the end of the post weighing session, at least 10% of the filters should be reweighed by a second analyst. Record the replicate measurement on the Filter Weighing Form (see Table 8). If the replicate measurement disagrees from the post measurement by more than 15 µg, reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, and/or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

5.3.2.7 If the dichotomous filter is not to be analyzed, use the tweezers to place it in a small glass envelope to which one of the extra labels has been affixed. Place an asterisk before the five-character code on the form. Deliver these filters to the filter bank for archiving.

5.3.2.8 If the filter is to be analyzed, use tweezers to carefully put it back into the petri dish. Place the petri dish **carefully** in a box.

5.3.2.9 Place a label on a sheet of 8 ½" x 11" paper for NAA, XRF, or other analysis as appropriate. Indicate the page number and balance number on each list. Keep the samples in the box in an order corresponding with the lists.

5.3.2.10 Without jostling the box, deliver it, the two lists, and the original Field Test Data Sheets with two copies of each to the sample custodian who will initial the original forms and return them upon receipt.

5.3.2.11 Calculate and report the particulate matter concentration for both fine and coarse samples utilizing the following equation:

$$PM = \frac{(W_f - W_i) \times 10^6}{V_{std}}$$

where:

PM = mass concentration of particulate matter (TSP, fine or coarse fraction), $\mu\text{g}/\text{std m}^3$.

W_i = average initial weight of clean filter, g.

W_f = average final weight of exposed filter, g.

V_{std} = air volume sampled, converted to standard conditions, std m^3 (see Inorganic Compendium Method IO-2.4).

10^6 = conversion of g to μg .

5.4 Transport of Filters

5.4.1 After collecting samples, transport the filters to the laboratory, taking care to minimize contamination and loss of the sample. Glass fiber filters should be transported or shipped in a shipping envelope. Cover the exposed surface of the membrane filters with an unexposed filter and seal the filter in plastic filter holders.

5.4.2 Assign numbers to the filters and log them into the data record form, ensuring that any necessary sampling information is included (Untreated filter samples may be stored indefinitely.)

5.4.3 Provide one blank sample with every 10 actual samples. No air is drawn through the blank filter, but it is subjected to the same handling and shipping manipulations as the actual samples.

6. Extraction of Glass Fiber Filters in Preparation for Metal Analysis

6.1 Introduction

This section describes both a microwave-extraction procedure and a hot-acid extraction procedure to extract inorganics from the particulate quartz glass-fiber filter. Following extraction, target analytes are analyzed by ICP, ICP/MS, FAA, or GFAA.

6.1.1 Ambient air quartz fiber filters should be received folded in half lengthwise with the particulate material inward and enclosed in protective envelopes. Store these protective envelopes approximately 15E-30EC until analysis.

6.1.2 The maximum sample holding times is usually 180 days. Analyze the samples within 180 days, even if these times are less than the maximum data submission times allowed.

6.2 Microwave Extraction Procedure

6.2.1 Filter Cutting Procedure.

6.2.1.1 Cut a 1" x 8" strip from the 8" x 10" filter using a template (see Figure 1) and cutting tool (see Figure 2) as described in the Federal Reference Method for lead. Use a laboratory microwave extraction system to extract the metals with a hydrochloric/nitric acid solution. After cooling, mix the digestate and use

Acrodisc® syringe filters to remove any insoluble material. Microwave extraction is used to prepare samples for ICP, ICP/MS, FAA, or GFAA.

6.2.1.2 Prior to use, acid wash the plexiglass filter template, the polysulfone centrifuge tubes and caps, and all other laboratory equipment that will come into contact with the filter samples to prevent contamination.

6.2.1.3 Using vinyl gloves, place the acid-cleaned filter template and cover inside a balance hood for cutting quartz fiber filters.

6.2.1.4 Wipe plexiglass template base, cover, and cutting blade with a clean, dry Kimwipe® to prevent sample cross-contamination.

6.2.1.5 Unfold the 8" x 10" quartz filter to be sectioned and carefully place sampled side up (numbered side down) within the plexiglass template filter margins.

6.2.1.6 Carefully (without disturbing sampled area of filter) place the grooved cover, notch side down, within the margins of the base template. Use a clear cutting blade to cut a 1" x 8" strip.

6.2.1.7 Using gloved fingers, accordion-fold or tightly roll the filter strip and transfer on edge to an acid cleaned polysulfone® tube, labeled with wax pencil. DO NOT use barcodes or tape in microwave.

6.2.1.8 Clean filter template between samples with dry Kimwipes®. (Gloves should be changed after 50 filters to minimize cross-contamination.)

6.2.1.9 Duplicate sample frequency is normally 1 per 20 field samples (see Table 9). Prepare a sample filter duplicate by moving the template cover to a second portion of the field collected filter. Cut an additional filter strip by moving the template cover to a second section of the filter and repeat Sections 6.2.1.6 through 6.2.1.8 above using a separate polysulfone tube.

6.2.1.10 Select a field collected filter for matrix spiking. In addition to the filter strip cut for determining metals, section a second portion of the filter, and fortify (spike) with target metals.

6.2.1.11 Prepare matrix spike samples at a frequency of 1 per 20 field samples or a minimum of 1 per extraction day (see Table 9). Move the template to a second section of the filter and repeat Sections 6.2.1.6 through 6.2.1.8, using a separate polysulfone tube and spike as shown in Table 9.

6.2.2 Microwave Calibration Procedure. Calibration of the microwave unit is a critical step prior to its use. In order that absolute power settings may be interchanged from one microwave unit to another, the actual delivered power must be determined, which allows the analyst to relate power in Watts to the partial power setting of the unit (% Power).

Calibration of a laboratory microwave unit (see Figure 3) depends on the type of electronic system used by the manufacturer. If the unit has a precise and accurate linear relationship between the output power and the scale used in controlling the microwave unit, the calibration can be a three-point calibration in the range of 50% to 100% power. If the unit does not prove linear (± 10 W) using the three-point technique, a multiple-point calibration is necessary. A bracketed calibration range of the digesting power to be used is recommended for determining the calibration points. If the unit power calibration needs multiple-point calibration, the point where the linearity begins must be identified. For example, a calibration at 100, 99, 98, 97, 95, 90, 80, 70, 60, and 50% power settings can be applied and the data plotted. The nonlinear portion of the calibration curve can be excluded or restricted. Each percent is equivalent to approximately 5.5-6.5 W and becomes the smallest unit of power that can be controlled. If 20-40 W are contained from 99-100%, that portion of the microwave calibration is not controllable by 3-7 times that of the linear portion of the control scale and will prevent duplication of precise power conditions specified in that portion of the power scale.

6.2.3 Microwave Power Evaluation. The equation in the following section evaluates the power available for heating in a microwave cavity. The variables are determined by measuring the temperature rise in 1 kilogram of water exposed to electromagnetic radiation for a fixed period of time. The following procedure is used for evaluating each calibration point, represented as % power output for each microwave.

6.2.3.1 Measure and record a 1 kilogram (1,000 g ± 0.1 g) sample of room temperature (23E± 2EC) distilled water in a thick-walled microwave transparent (Teflon®) beaker for each calibration point.

6.2.3.2 Measure and record the initial temperature of the water, (T_i), to within 0.1EC. The starting temperature should be between 22 and 26EC.

6.2.3.3 Place the Teflon® beaker in microwave and irradiate at full power (100% point) for 2 min (120 s). Each calibration point (i.e., 100%, 50% or multi-points) requires a separate clean beaker containing water at room temperature.

6.2.3.4 Remove beaker from the microwave and measure and record the maximum final temperature (T_f) to 0.1EC, within 30 s of the end of irradiation. This process should be done while stirring continuously (an electronic stirrer using a large stir bar works best).

Calculate the microwave power according to the following formula:

$$\text{Power} = \frac{K \times C_p \times M \times \Delta T}{t}$$

$$\frac{K \times C_p \times M}{t} = 34.87$$

$$\text{Power} = 34.87 \times \Delta T$$

where:

Power = The apparent power absorbed by the sample, watts (W = joule-s⁻²).

K = The conversion factor for thermochemical calories-s⁻¹ to W = 4.184.

C_p = The heat capacity, thermal capacity, or specific heat (cal-g⁻¹-EC⁻¹ = 1.0 for water).

M = The mass of the sample, grams.

ΔT = T_f - T_i, EC.

t = Time, s.

6.2.3.5 Derive an equation for the linear portion of the calibration range and determine the equivalent value in watts of the arbitrary setting scale. Use the actual power in watts to determine the appropriate setting of the particular microwave unit being used. Each microwave unit will have its own (% power) setting that corresponds to the actual power (in wattage) delivered to the samples.

6.2.3.6 An initial multipoint power evaluation should be performed for each microwave unit. If linear, the calibration should be checked on a regular basis, using the 3-point calibration verification routine. A single point verification may be appropriate when utilizing a single power output for digestion. If any part of the power source to the microwave has been serviced or altered, the entire calibration must be reevaluated.

6.2.4 Cleaning Procedure for PFA Vessels. All digestion vessels must be acid cleaned and rinsed with reagent water prior to use to prevent contamination.

6.2.4.1 Wash each PFA vessel with deionized detergent and rinse with reagent water.

6.2.4.2 Add 10 mL concentrated HNO₃ to each of 12 vessels, cap, and place in microwave.

6.2.4.3 Heat vessels at 100% power in microwave for 10 min as recommended by CEM (microwave manufacturer). Rinse the vessels with copious amounts of deionized, distilled water prior to use for any

analyses. If only 6 vessels are to be cleaned, 70% power may be utilized, which corresponds to approximately 5% per vessel.

6.2.5 Digestion Procedure for Microwave Extraction For Ambient Filter Samples.

[Note: Nitric and hydrochloric acid fumes are toxic. Prepare in a well-ventilated fume hood. Mixing results in an exothermic reaction. Stir slowly.]

6.2.5.1 Using vinyl gloves or plastic forceps, retrieve the filter strip from Section 6.2.1 and place on its edge in a labeled centrifuge tube. Using the plastic forceps, crush the filter strip down into the lower portion of the centrifuge tube to ensure acid volume will cover entire filter.

[Note: A breathing mask and vinyl gloves are required for safety of personnel handling dry glass-fiber filters. The breathing mask prevents the inhalation of minute glass shards and particulate material. The gloves protect the skin from the same materials and also prevent contamination of the sample by skin secretions. A recommended alternate to the use of a breathing mask would be performing cutting and transfer operations involving sample filters in a laminar flow hood, if available.]

[Note: More than one strip from a filter should be extracted to ensure adequate sample volume for sample and QC sample analysis. Blank filter samples should be extracted and analyzed, and digestion blanks should be run to ensure low levels of metals in the reagents used.]

6.2.5.2 Using a preset calibrated automatic dispensing pipette or Class A glass pipette, add 10.0 mL of the extraction solution to each of the centrifuge tubes. The acid should cover the strip completely. The sequence of adding the filter strip and acid to the centrifuge tube may be reversed, if more convenient, without affecting the results. Place the centrifuge tube in a Teflon® PFA vessel containing 31 mL of deionized water. Continue this process for a total of 12 samples to maximize microwave capacity.

6.2.5.3 Place the PFA vessel caps with the pressure release valves on the vessels hand-tight and tighten using the capping station to a constant torque of 12 ft-lb. Weigh and record the capped vessel assembly to the nearest 0.01 g. Place the vessels in the microwave carousel. Connect each sample vessel to the overflow vessel using the Teflon® PFA connecting tubes (see Figure 3).

6.2.5.4 Place the carousel containing the 12 vessels onto the turntable of the microwave unit. Any vessels containing 10 mL of acid solution for analytical blank purposes are counted as sample vessels. Irradiate the sample vessels at 486 W (power output) for 23 min. (Based on the calibration of the microwave as previously described). If fewer than 12 samples are to be digested, adjust the microwave system by reducing the power so that equivalent digesting power is delivered to the smaller sample batch. Generally, each vessel represents approximately 5% power. Therefore, a reduction in W would be reduced by 30% if only 6 vessels are digested. This reduction is only approximate, and each microwave unit will produce a different level of power output.

6.2.5.5 At the end of the microwave program, allow the pressure to dissipate (venting may be utilized with caution), then remove the carousel containing the vessels and cool in tap water for 10 min. Weigh the capped vessels assembly to the nearest 0.01 g and compare to the initial weight to verify no loss of sample. The initial and final weights should compare within 0.1 g. If the initial and final weights do not agree within 0.01 g, the appropriate action must be taken which may include rejecting the digested sample. Using the capping station uncapped the microwave vessels, remove the labeled centrifuge tubes containing samples and discard the water in the PFA vessels.

6.2.5.6 Using a calibrated automatic dispensing pipette or a Class A glass pipette, add 10 mL of deionized distilled water to each centrifuge tube. Cap the centrifuge tube tightly and vortex (mix) the contents thoroughly for 2-3 minutes to complete extraction. Using a nylon or teflon syringe pull-up a volume

of sample from the centrifuge tube, place Acrodisc filter on syringe and dispense into a pre-labeled sterile 15 mL centrifuge tube. Continue until centrifuge tube contains 10 mL of filtered digestate.

6.2.5.7 The final extraction volume is 20 mL based upon the above procedure. The final extraction solution concentration is 3% HNO₃/8% HCl. The filtered sample is now ready for analysis. Store for subsequent analysis by one or more of the Inorganic Compendium methods.

6.3 Hot Acid Extraction Procedure

6.3.1 Introduction. A hot extraction procedure to solubilize metals from the glass-fiber filter for subsequent analysis by ICP, ICP/MS, FAA, or GFAA is described in this method. An acid extraction solution is used to extract the metals from the quartz filter on a hot-plate.

6.3.2 Summary of Method.

6.3.2.1 Use the hot-acid extraction procedure as an alternate when microwave technology is not available.

6.3.2.2 Cut a 1" x 8" strip from the 8" x 10" filter as described in Federal Reference Method for lead. The inorganics are extracted from the filter strip by a HCl/HNO₃ acid solution using a hot acid extraction procedure. After cooling, pour the digestate rinses to a volumetric flask and dilute to volume. Filter to remove any insoluble material.

6.3.3 Hot Acid Extraction Procedure.

6.3.3.1 Using vinyl gloves or plastic forceps, retrieve the strip from Section 6.2.1 and place it in a labeled 150 mL Griffin beaker. Place the filter strip down into the lower portion of the beaker to ensure acid volume will cover entire filter.

[Note: More than one strip from a filter should be extracted to ensure adequate sample volume for sample and QC sample analysis. Blank filter samples should be extracted and analyzed and digestion blanks should be run to ensure low levels of metals in the reagents used.]

6.3.3.2 Using a preset calibrated automatic dispensing pipette or Class A glass pipette, add 10 mL of extracting acid (see Section 6.2.5.2) for analysis.

[Note: The acid should cover the strip completely.]

6.3.3.3 Place beaker on the hot-plate, contained in a fume hood, and reflux gently while covered with a watch glass for 30 min. Do not allow sample to dry. Remove the beakers from the hot-plate and allow to cool.

[Caution: Nitric acid fumes are toxic.]

6.3.3.4 Rinse the beaker walls and wash with D.I. water. Add approximately 10 mL reagent water to the remaining filter material in the beaker and allow to stand for at least 30 min. This critical step must not be deleted; it allows the acid to diffuse from the filter into the rinse. Transfer the extraction fluid in the beaker to a 20 mL volumetric flask or other graduated vessel. Rinse the beaker and any remaining solid material with Type I water and add the rinses to the flask. Some solids from the filter may be transferred to the flask with the rinses; this is acceptable. Dilute to the mark with Type I water and shake.

6.3.3.5 Using a nylon or Teflon[®] syringe, pull-up a volume of sample from the centrifuge tube, place disc filter on syringe, and dispense into a pre-labeled sterile 15 mL centrifuge tube. Continue until centrifuge tube contains 10 mL of filtered digestate.

6.3.3.6 The final extraction volume is 20 mL based upon the above procedure. The final extraction solution concentration is 3 % HNO₃/8% HCl. The filtered sample is now ready for analysis. Store for subsequent analysis by one or more of the Inorganic Compendium methods.

TABLE 1. CHARACTERISTICS OF FILTER MEDIUM

Cellulose Fiber (Cellulose Pulp)
<ul style="list-style-type: none"> • Low ash • Maximum temperature of 150EC • High affinity for water • Enhances artifact formation for $\text{SO}_4^{=}$ and NO_3^- • Good for x-ray/neutron activation analysis • Low metal content
Quartz Fiber (Quartz spun with/without organic binder)
<ul style="list-style-type: none"> • Maximum temperature up to 540EC • High collection efficiency • Non-hydroscopic • Good for corrosive atmospheres • Very fragile however • Difficult to ash; good with extraction
Synthetic Fiber (Teflon® and Nylon®)
<ul style="list-style-type: none"> • Collection efficiency > 99% for 0.01 μm particles • Low artifact formation • Low impurities • Excellent for X-ray analysis • Excellent for determining total mass due to non-hydroscopic nature • Nylon fiber good for HNO_3 collection
Membrane Fiber (Dry gel of cellulose esters)
<ul style="list-style-type: none"> • Fragile; requires support pad during sampling • High pressure drop • Low residue when ashed

TABLE 2. SUMMARY OF USEFUL PHYSICAL PROPERTIES OF VARIOUS FILTER MEDIUMS

Filter and Filter Composition	Density, mg/cm ²	pH	Filter Efficiency %
Teflon® (Membrane) (CF ₂) _n (2 µm Pore Size)	0.5	Neutral	99.95
Cellulose (Whatman 41) (C ₆ H ₁₀ O ₅) _n	8.7	Neutral (Reacts with HNO ₃)	58% at 0.3 µm
Glass Fiber (Whatman GF/C)	5.16	Basic pH - 9	99.0
"Quartz" Gelman Microquartz	6.51	pH - 7	98.5
Polycarbonate (Nuclepore) C ₁₅ H ₁₄ + CO ₃ (0.3 µm Pore Size)	0.8	Neutral	93.9
Cellulose Acetate/Nitrate Millipore (C ₉ H ₁₃ O ₇) _n (1.21 µm Pore Size)	5.0	Neutral (Reacts with HNO ₃)	99.6

TABLE 3. EXAMPLE OF TYPICAL BACKGROUND METAL CONCENTRATION (µg/FILTER) OF VARIOUS GRADES AND TYPES OF FILTERS

Metal	1	4I	42	542	Filter 17ch	Grades GF/A	EPM 2K	934QH	QMA	QMB
Al	1	2	2	1	--	4,300	170	2,950	4	--
Ag	--	--	--	--	--	< 1	< 1	--	< 1	--
As	< 0.02	< 0.02	< 0.02	< 0.02	--	< 6	< 6	--	< 6	--
B	1	< 2	2	2	--	6,100	42	--	42	--
Ba	< 1	< 1	< 1	< 1	--	8,500	50	< 1	10	--
Be	--	--	--	--	--	0.2	< 1	0.1	< 1	< 1
Bi	--	--	--	--	--	0.3	< 4	0.8	< 4	7
Ca	185	13	13	9	29	2,500	540	7,000	85	--
Cd	--	--	--	--	--	1	< 1	0.8	0.2	< 1
Co	--	--	--	--	--	11	< 1	5.5	1.1	< 1
Cr	0.3	0.3	0.3	0.7	--	0.2	4.5	3.0	1.6	0.7
Cu	1.2	0.1	0.4	0.2	0.6	56.0	1	7	3.4	3.4
Fe	5.0	6.0	6.0	3.0	5.0	100	15	265	23	29
Mg	< 0.005	--	< 0.005	< 0.005	--	--	--	--	--	--
K	4.0	1.5	1.5	0.6	7	775	573	125	--	--
Mg	7	1.8	1.8	1.0	6	1,090	238	2,800	--	--
Mn	0.1	< 0.05	< 0.05	< 0.05	--	8.0	< 1	2	0.5	< 1
Mo	--	--	--	--	--	2.0	< 2	4	< 2	< 1
N	28	--	12	260	--	--	--	--	--	--
Na	160	40	37	8	30	1,830	1,020	1,100	280	--
Ni	--	--	--	--	--	6.0	--	2.5	3.4	2.2
Pb	0.4	0.2	0.2	0.1	1	10	2.5	2.0	2.3	8.3
S	17	< 5	< 5	< 5	--	26	--	--	--	--
Sb	< 0.02	< 0.02	< 0.02	< 0.02	--	< 4	< 4	--	< 4	--
Si	15	< 2	< 2	< 2	--	--	--	--	--	--
Ti	--	--	--	--	--	0.1	< 1	< 30	< 1	< 4
V	--	--	--	--	--	0.1	--	10	--	< 4
Sn	2.5	0.6	0.6	0.3	--	5,500	15	34	10	13

TABLE 4. EXAMPLE WEIGHING ROOM ATMOSPHERIC CONDITION FORM

Equilibrium Period				Temperature Limits = 15EC to 35EC			Relative Humidity Limits = < 50%			Name
Begin Date	Begin Time	End Date	End Time	Max-Min	Avg	Limits met?	Max-Min	Avg	Limits met?	

TABLE 5. EXAMPLE WEIGHING BALANCE CHECK FORM

Date	Time	Balance Type	Balance ID	Class S Weights Serial No. or ID	mg			Limit = 0.5 mg Limits met?	Name
					Class S weight	Balance weight	Difference		

TABLE 6. EXAMPLE FILTER WEIGHING FORM

Filter No.	Pre-field Initial Weighing		10% Second Weighing by Second Analyst (Limit = 1.0 mg)				Post-field Final Weighing		10% Second Weighing by Second Analyst (Limit = 2.0 mg)			
	Weight (mg)	Name-Date	Weight (mg)	Diff. (mg)	Limit met?	Name-Date	Weight (mg)	Name-Date	Weight (mg)	Diff. (mg)	Limit met?	Name-Date

TABLE 7. EXAMPLE WEIGHING BALANCE CHECK FORM

Date	Time	Balance Type	Balance ID	Class S Weights Serial No. or ID	mg			Limit = 3 ug Limits met?	Name
					Class S weight	Balance weight	Difference		

TABLE 8. EXAMPLE FILTER WEIGHING FORM

Filter No.	Pre-field Initial Weighing		10% Second Weighing by Second Analyst (Limit = 15 Fg)				Post-field Final Weighing		10% Second Weighing by Second Analyst (Limit = 15 Fg)			
	Weight (mg)	Name-Date	Weight (mg)	Diff. (mg)	Limit met?	Name-Date	Weight (mg)	Name-Date	Weight (mg)	Diff. (mg)	Limit met?	Name-Date

TABLE 9. EXAMPLE QUALITY CONTROL SAMPLES

Type	Frequency	Contains 1"x8" filter strip	Comments ^a
Method Blank	1 per 20 samples	No	Containing reagents only, to evaluate background contributions from reagents.
Filter Lot Blank	1 per filter lot change	Yes	Analyzed prior to use of new filter lot.
Filter Duplicate	1 per 20 samples	Yes	This is a second 1"x8" filter strip cut from a single field sample.
Matrix Spike ^b	1 per 20 samples	Yes	See individual methods.
LCS ^c	1 per extraction day	Yes	See individual methods.

^aThese multimetal stock standards can be acquired from Spex Industries, Inc., Inorganic Ventures Inc., or equivalent.

^bThe matrix spike (MS) is a 1"x8". strip cut from a field sample filter and spiked at a target level.

^cThe Lab Control Sample (LCS) is a blank filter spiked with the same concentration of metals as the matrix spike.

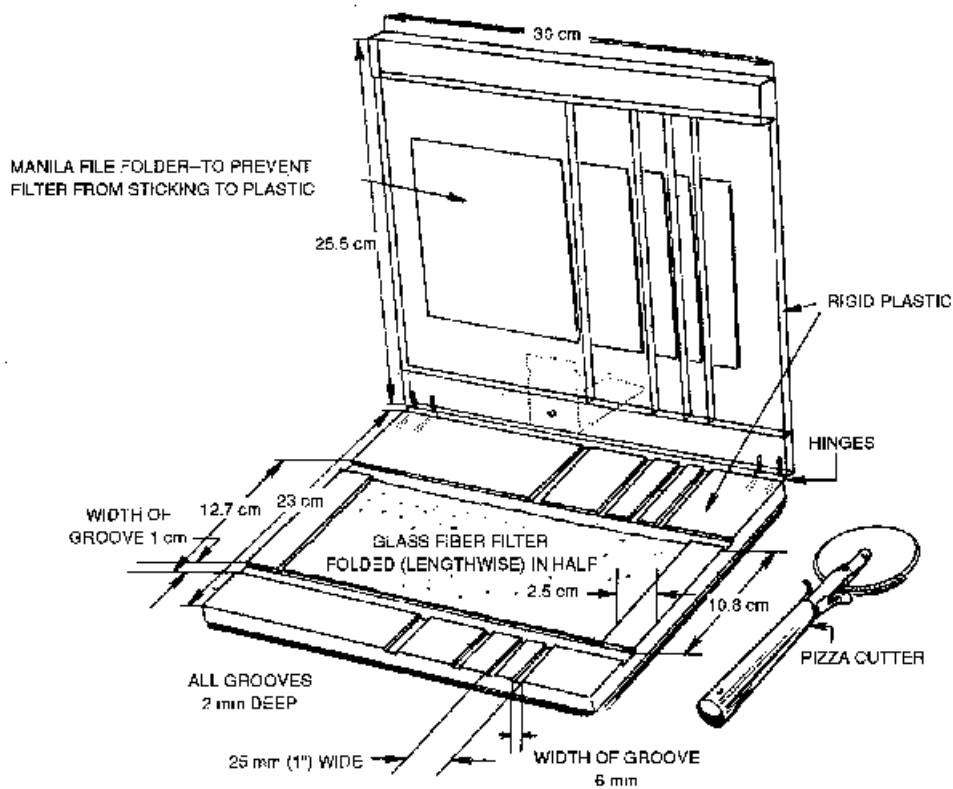


Figure 1. Example of templates for cutting filters.

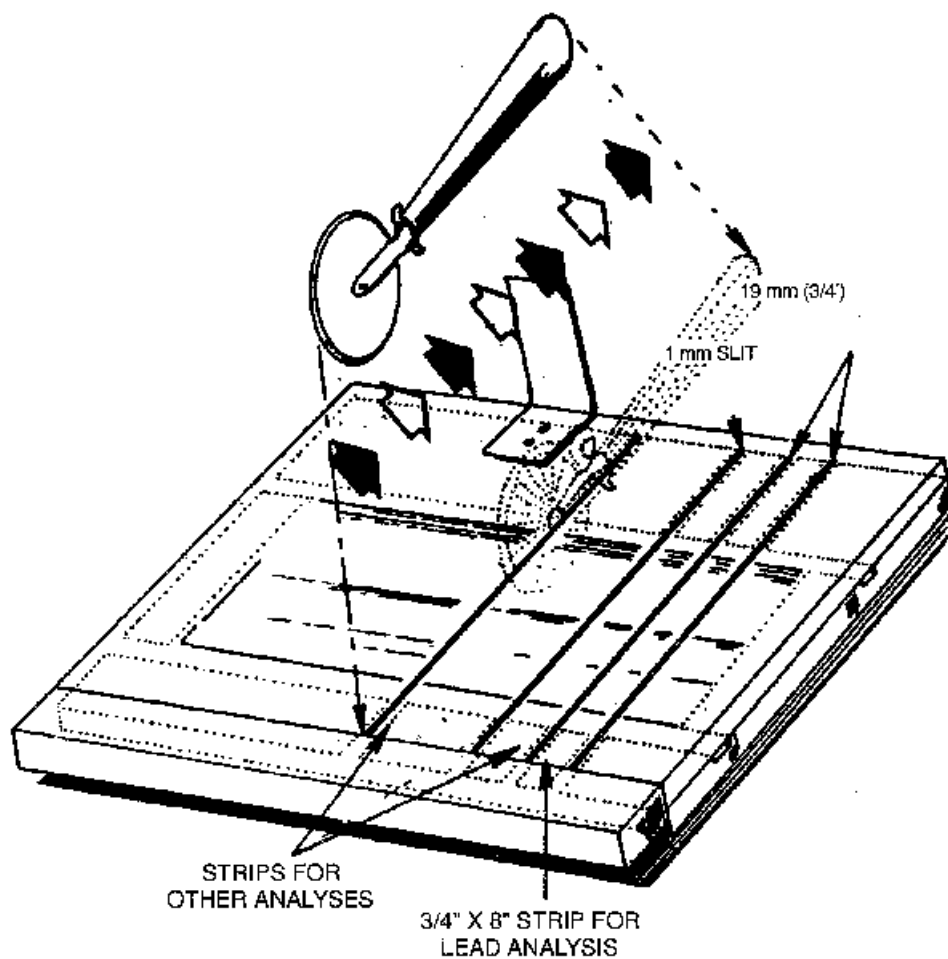


Figure 2. Diagram of filter cutting procedure.

MICROWAVE EXTRACTION

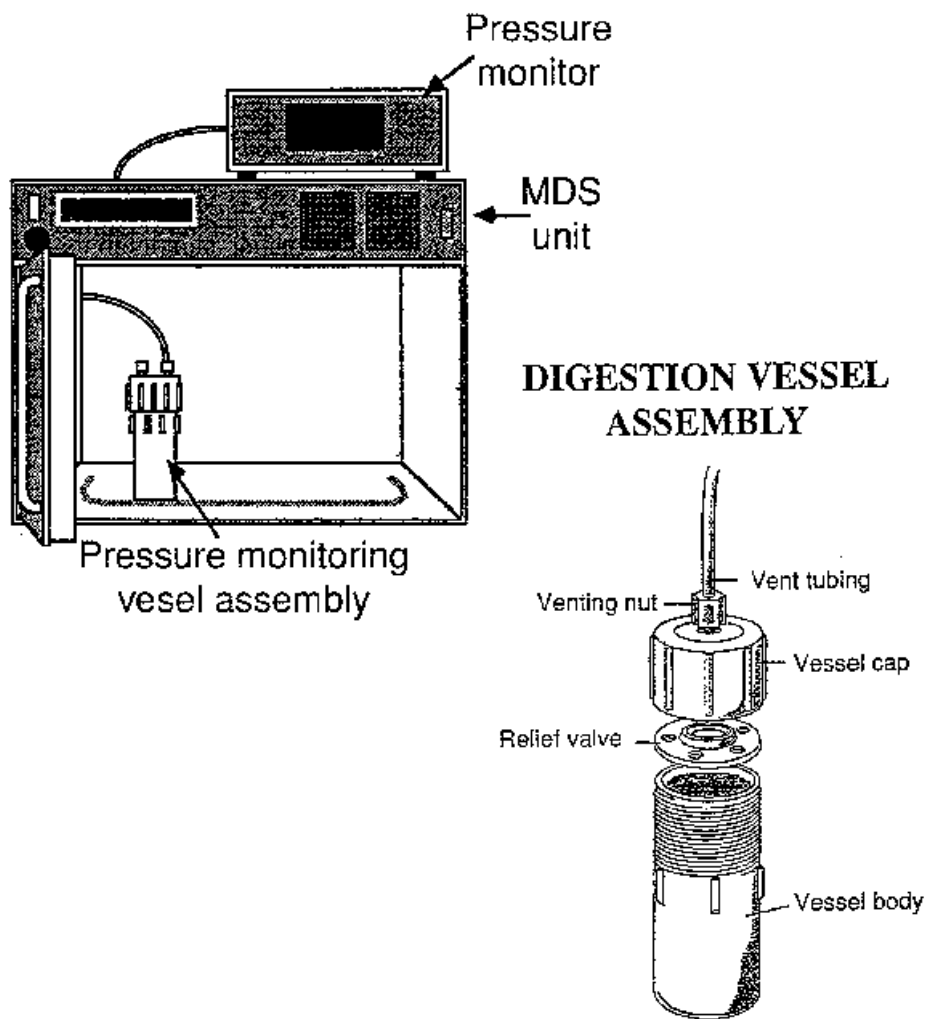


Figure 3. Example of microwave digestion system.

ATTACHMENT L-C

MACTEC STANDARD OPERATING PROCEDURE FOR
AIRMETRICS MINIVOL® PORTABLE AIR SAMPLER
MACTEC SOP-104

Harding ESE, Inc.
Standard Operating Procedure

90 Digital Dr.
Novato, CA 94949

Original Date Issued:	August 2002
Type:	Revision 0
Revision Date:	
Procedure #:	HESE SOP-104
Total Pages:	7

NAME	Standard Operating Procedure for AirMetrics MiniVol Portable Sampler
SCOPE	The scope of the SOP will detail the operating procedures and the procedures used to perform calibrations on AirMetrics MiniVol Portable Air Sampler
REFERENCE	EPA Compendium Method IO-2.1 AirMetrics MiniVol Portable Air Sampler User's Manual
EQUIPMENT	AirMetrics MiniVol Portable Air Sampler

INTRODUCTION MiniVol

Although accurate and precise, the monitors are bulky and immobile, causing difficulty in studying remote sites without a power source. The EPA and the Lane Regional Air Pollution Authority addressed the need for portable sampling devices with the production of the AirMetrics MiniVol Portable Air Samplers. These ambient air sampling machines are designed to collect either PM₁₀ or PM_{2.5}, depending on the attached impactor and filter head, and can also be used to measure non-reactive gases (CO & NO_x).

The MiniVols are not federal reference method (FRM) samplers, though their results closely approximate FRM air quality data. The MiniVol is a lightweight battery-operated sampler, which allows it to be used in remote areas. The low cost of the sampler allows for a network of MiniVols to be easily set up at a lower cost than a large machine network. Other features include a 7-day programmable timer, an elapsed time totalizer, rechargeable battery packs, and all-weather PVC construction.

The procedures that lie herein are to be used by the operator responsible for the collection of the PM₁₀ and PM_{2.5} filters. Refer to the manufacturer's operation manual for integrated gas sampling, hardware descriptions, and maintenance details.

2. FILTER HANDLING AND PRESAMPLING PREPARATION

2.1 Filter Handling

The current filter media approved by the U.S. EPA for sampling particulate matter is a 2µm pore-size Teflon material in a white 47mm diameter casing. Filters will be provided by the processing laboratory, and should be returned there for analysis with a copy of the filter's data

form. Touching the individual filters could void the sample and physically handling should be avoided. Storage of the filters should be in a dry, controlled environment. There are no control limits on humidity and temperature for unexposed filters, but exposed filters should be kept below ambient temperature. A cooler type container loaded with "blue ice" and transported in an air-conditioned vehicle is recommended.

2.2 Connecting the Sampler Body and the Battery Pack

When connecting the sampler body and battery pack, note that the body's pins are unevenly spaced and can only fit onto the battery pack in one way. The pin closest to a latch on the sampler body inserts into the odd-colored receptacle on the battery pack.

2.3 Sample Date and Time Programming

The Programmable Timer can be set to run for up to six cycles within 24 hours, in addition to separate time periods on different days within one week. The real-time clock must first be set to the correct day and time in order for sampling programming to be set. To set the day, hold down the "Clock" button until the correct day appears on the display. The time can be set by holding down the "Clock" button while depressing the "Hour" and "Min" buttons until the correct time is set.

To set the timer to sample, press the "Prog" button once to display the "Prog 1_{ON}" setting. Use the "Hour" and "Min" buttons to set the time of day for sampling to begin. Press the "Week" button until the correct sampling day appears on the display. Pressing the "Prog" button again will show the "Prog 1_{OFF}" setting. In this setting, program the monitor to stop sampling in the same manner as programming the monitor to turn on. Set the monitor to automatically run by pressing the "ON/AUTO/OFF" button until "AUTO" is selected.

2.3 Equipment Calibration

Calibration Apparatus A MiniFlo flow rate transfer standard device is used as the flow rate reference to calibrate the sampler's rotameter-. To be valid, the MiniFlo transfer standard must have been calibrated against a primary standard traceable to the NIST within the last year. A calibrated digital manometer, Magnehelic® gage, or water manometer is required to measure the pressure drop across the MiniFlo orifice element. The actual ambient temperature and barometric pressure must also be measured or obtained locally.

Precalibration System Check Procedures for the precalibration system check are as follows:

1. Place a filter into the MiniVol sampler filter holder and attach the filter holder assembly to the sampler. (Filters used for flow rate calibrations should not be used for subsequent sampling.)
2. Turn on the sampler and allow it to warm up to full operating temperature (at least 2 minutes).
3. While the sampler is running, depress and hold the reset button. (This allows the sampler to continue to run without tripping the low flow shut-off circuit.) Close off the inlet using the palm of your hand. Observe the rotameter, if there are no leaks the rotameter should drop to zero and remain there.

4. Verify that the transfer standard calibration equation is current and traceable to an acceptable primary standard.
5. Adjust manometer or Magnehelic® gage to read zero, and connect to the pressure tap on MiniFlo transfer standard. Note: Magnehelic® gage is sensitive to the position it is in when zeroed (vertical or horizontal). Maintain this position when taking readings.

Rotameter Calibration. Procedures for calibrating the sampler rotameter are as follows:

1. Install the MiniFlo transfer standard on the inlet tube of the MinVol sampler filter holder.
2. Turn on the sampler and allow it to warm up to normal operating temperature (at least 2 minutes). The transfer standard must also equilibrate before proceeding with the calibration.
6. Read and record the following parameters:
 - Ambient temperature (T_{act}), °K
 - Barometric pressure (P_{act}), mm Hg
4. Adjust the flow control until the rotameter displays 6.5 L/min.
5. Read and record the following parameters:
 - Transfer standard pressure drop (H), inches of water
 - Sampler rotameter indication (Q_{ind}), liters per minute
6. Repeat steps 4 and 5 for rotameter settings representing flow rates of 6.0, 5.5, 5.0, 4.5, and 4.0 L/min.

Calibration Calculations. Gather together all the calibration data, including the transfer standard calibration information and the MiniVol sampler calibration data sheet. The following calibration calculation procedures are recommended.

Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that has already been obtained.

Calculate Q_{act} for each calibration point as determined by the transfer standard calibration equation (**Eq.A** from MiniFlo calibration certificate).

Calculate and record the standard corrections ($Q_{@std}$) for each calibration point as:

Eq. 1

$$Q_{@std} = Q_{act} \times \sqrt{\frac{T_{std} P_{act}}{T_{act} P_{std}}}$$

where:

$Q_{@std}$ = standard correction flow rate, L/min

Q_{act} = flow rate at ambient conditions (transfer standard), L/min

T_{act} = ambient temperature, °K
 P_{act} = ambient barometric pressure, mm Hg
 T_{std} = standard temperature, 298 °K
 P_{std} = standard pressure, 760 mm Hg.

On a sheet of graph paper or computer spreadsheet plot the calculated standard correction flow rates, $Q_{@std}$ (y-axis) versus the corresponding rotameter indicated flow rate, Q_{ind} (x-axis). Using a programmable calculator or a spreadsheet and the following model, calculate the linear regression slope (m_{vol}), intercept (b_{vol}), and correlation coefficient (r) to obtain the MiniVol™ sampler flow rate calibration relationship.

For the regression model $y=mx+b$

Eq. 3

$$\text{let } y = Q_{@std} = Q_{act} \sqrt{\frac{T_{std} P_{act}}{T_{act} P_{std}}} \text{ and } x = Q_{ind}$$

So that the model is given by:

Eq. 4

$$Q_{@std} = m_{vol} Q_{ind} + b_{vol}$$

A six-point calibration should yield a regression equation with a correlation coefficient of $r > 0.990$, with no point deviating more than 2% from the value predicted by the regression equation.

Note: For actual sample periods, the sampler's average actual operational flow rate Q_{act} is calculated from the calibration slope and intercept using

Eq. 5

$$Q_{act} = (m_{vol} Q_{ind} + b_{vol}) \times \sqrt{\frac{T_{act} P_{std}}{T_{std} P_{act}}}$$

where

Q_{act} = sampler flow rate at ambient conditions, actual L/min
 Q_{ind} = rotameter response, L/min
 T_{act} = ambient temperature, °K
 P_{act} = ambient barometric pressure, mm Hg
 T_{std} = standard temperature, 298 °K
 P_{std} = standard pressure, 760 mm Hg
 m_{vol} = slope of the MiniVol™ flow rate calibration relationship
 b_{vol} = intercept of the MiniVol™ flow rate calibration relationship.

T_{act} and P_{act} readings may be measured on-site or obtained from a nearby U.S. National Weather Service or airport weather station. Barometric pressure readings obtained from remote stations must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the remote station. If ambient temperature and pressure readings are not available, a seasonal average temperature and barometric pressure can also be used. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. It is therefore recommended that seasonal values represent actual values within 20 EC and 40 mm Hg.

Rotameter Set Point Adjustment Procedure. Calculate and record the rotameter set point using temperatures and pressures expected on the day(s) sampling is conducted. These values can be seasonal set points or daily settings.

Eq. 6

$$I_{sp} = \frac{5.0 \times \sqrt{\frac{T_{std} P_{act}}{T_{act} P_{std}} - b_{vol}}}{m_{vol}}$$

where:

I_{sp} = sampler rotameter set point flow rate adjustment

5.0 = MiniVol design flow rate, L/min

P_{act} = ambient barometric pressure, mm Hg

T_{act} = ambient temperature, EK.

T_{std} = standard temperature, 298 EK

P_{std} = standard pressure, 760 mm Hg

m_{vol} = slope of the MiniVol flow rate calibration relationship

b_{vol} = intercept of the MiniVol flow rate calibration relationship.

Adjusting the sampler rotameter to seasonal average conditions will help minimize data loss caused by exceeding the MiniVol's design condition specifications.

Sampler Calibration Frequency

To ensure accurate measurement of the PM10/PM2.5 concentrations, calibrate the sampler upon installation and then recalibrate it as follows:

1. At least annually.
2. After any repairs that might affect sampler calibration.
3. If the field calibration flow check results exceed QC limits ($\pm 7\%$ from the sampler's indicated flow rate).

3. FIELD SAMPLING PROCEDURES

3.1 Sample Filter Cassette Recovery

1. Disconnect the AC cord from the battery pack and remove the MiniVol from its hanger. If possible, move indoors or under cover. Remove the handle by unscrewing a side cap, and removing the o-rings from the bar.
2. Lift the sampler out of the housing by holding the top 6" cap and lifting up gently. Support the sampler by either holding it above its housing or using the triangular mount stand on the bottom of the sampler. Use of the triangular stand should only be done when the battery pack is attached to the sampler. Do not pull the sampler completely out of the housing; the power cord will be torn.
3. Check for any light on the display board, such as "Low Flow" or "Low Battery" and record the findings in the Notes section of the data sheet.
4. Do a post flow check by turning on the pump and ensuring the Rotameter reads 5.0 LPM. Record this on the data sheet. If the pump does not start, press the reset button on the right side of the display board.
5. Record the hour meter value in the Hour Meter Stop field on the data sheet. Lower the sampler back into the housing.
6. Remove the filter head by pressing down on the quick-disconnect while pulling up on the filter assembly. Unscrew the base of the filter assembly and remove the filter with a gloved hand. Secure the filter cassette in the petri dish labeled with the correct filter number.
7. Return a copy of the data sheet with the exposed filter to the processing laboratory. If the PM₁₀ monitor is not to be used until the next sampling run, it should be disconnected from the battery pack and properly stored indoors.

3.2 Sample Filter Cassette Installation

1. The operator is to record the following information for the new filter on the AirMetrics MiniVol PM_{2.5} or PM₁₀ Field Data Form:
 - Filter Run Date
 - Site ID
 - Type of Impactor (PM_{2.5} or PM₁₀)
 - Model Number
 - Sample ID
 - Filter Number
 - Filter Date In
 - Start Date/Time
 - Stop Date/Time
 - Hour Meter Start
 - Start Flow
 - Impactor Run

2. Place a new filter in the opened (from directions above) filter assembly with a gloved hand and re-assemble the unit. Place filter assembly on the quick-disconnect and press firmly down to snap it in place. Pull up to insure connection. Do not pull up on the small rain hood cap.
3. Remove the sampler body from its housing and support it as defined above. On the Programmable Timer, check the current standard time. The reading should be correct during the fall and one hour ahead during the spring. To adjust the time, hold the clock button down while depressing the "Min" or "Hour" keys.
4. Press the "Prog" key to access the 6 available programmable times to turn the sampler on and off. Scroll through each program (1-6) by pressing "Prog" and reset any existing programmed times by using the "RST/RCL" button.
5. At "Prog 1_{ON}", set the time for the beginning of the sample run, which will be 12am of the sampling day on the calendar. At "Prog 1," set the day of the week using the "Week" key. The days will scroll through 10 different choices for day runs. Set the day according to the calendar date. At "Prog 1_{OFF}", set the time for the end of the sample run, which will be 12am the next day. Set the day of the week (same as above). Press the "Clock" key to exit the Program menus.
6. Select the Automatic mode by depressing the "ON/AUTO/OFF" button. The sampler will now start sampling at the programmed time automatically.
7. Reassemble the sampler's handle and place it on the hanger with the impactor away from the hanger stand. Plug in the AC power cord if applicable.

* After every five sampling runs, return the impactor assembly to the processing laboratory. A clean assembly will be supplied for the next run. Only impactors designed for the collection of PM_{2.5} will be supplied, so it is necessary to change the assembly if a new PM₁₀ impactor is needed. Disassemble the entire unit, and switch the PM_{2.5} impactor (with the small opening at its base) with the PM₁₀ impactor (with the larger opening at its base). The PM₁₀ impactor will be the only one present, and the entire unit will be half the size of the PM_{2.5}. Return the extra parts with the assembly after five runs.

3.3 Integrated Gas Sampling

For PM₁₀ Sampling, Follow the steps below:

1. Ensure that the PM₁₀ Impactor (silver) is installed in the preseparator assembly. The multiple impactor adapter is not needed, nor the PM_{2.5} Impactor (gold)
2. Clean and lubricate the target disk at least every 2-4 sampling periods, or more frequently depending on the degree of impaction stage soiling.
3. After the sampler has been assembled, After the sampler has been assembled, calibrated, verified to be in proper working order, and a filter loaded in the filter holder assembly, set flow rate adjustment in accordance with procedures as shown in the Operation Manual.
4. Follow all other procedures in the Operation Manual, noting that PM10 is being sampled.


Appendix L


Draft Final Prescribed Burn Air Sampling and Analysis Plan Munitions Response Site 16 Former Fort Ord, California

Prepared for

Shaw Environmental & Infrastructure, Inc.
Bldg 4522 Joe Lloyd Way
Monterey, California 93944

MACTEC Project No. 4088053164 04.3


Matthew H. Walraven
Project Environmental Scientist


Bruce L. Wilcer
Principal Geologist

March 24, 2006

Appendix L

Draft Final

Prescribed Burn Air Sampling and Analysis Plan

Munitions Response Site 16

Former Fort Ord, California

MACTEC Project No. 4088053164 04.3

This document was prepared by MACTEC Engineering and Consulting, Inc. (MACTEC) at the direction of Shaw Environmental for the sole use of Shaw, the United States Army Corps of Engineers (USACE), the U.S. Army, and the regulatory agencies, the only intended beneficiaries of this work. No other party should rely on the information contained herein without the prior written consent of Shaw, the USACE, and the Army. This report and the interpretations, conclusions, and recommendations contained within are based in part on information presented in other documents that are cited in the text and listed in the references. Therefore, this report is subject to the limitations and qualifications presented in the referenced documents.

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AIRMETRICS MINIVOL® PORTABLE AIR SAMPLER MACTEC SOP-104

LIST OF ACRONYMS AND ABBREVIATIONS

Army	U.S. Department of the Army
ATSDR	Agency for Toxic Substances and Disease Registry
CAAA	Clean Air Act Amendments
CARB	California Air Resources Board
CDQMP	Chemical Data Quality Management Plan
COC	Chain-of-Custody
COPC	Chemicals of Potential Concern
DQOs	Data quality objectives
DTSC	Department of Toxic Substances Control
EPA	Environmental Protection Agency
Fort Ord	The former Fort Ord
GPS	Global Positioning System
IDW	Investigation-derived waste
MACTEC	MACTEC Engineering and Consulting, Inc. (formerly Harding ESE, Inc.)
MBUAPCD	Monterey Bay Unified Air Pollution Control District
MEC	Munitions and Explosives of Concern
MRS	Munitions Response Site
PM ₁₀	Particulate Matter Less than 10 microns
POM	Presidio of Monterey
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
SAP	Sampling and Analysis Plan
Shaw	Shaw Environmental, Inc.
SMP	Smoke Management Plan
SOP	Standard Operating Procedure
SSHP	Site Safety and Health Plan
SSWP	Site Specific Work Plan
TWA	Time Weighted Average
µg/m ³	Micrograms per cubic meter
USACE	U.S. Department of the Army, Corps of Engineers
USEPA	U.S. Environmental Protection Agency

L1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) presents procedures for the collection and analysis of air samples during the prescribed burn event for Munitions Response Site (MRS) 16 at Former Fort Ord. The site history, background, historical use of munitions and explosives of concern (MEC), and details of the prescribed burn are presented in the site specific workplan (SSWP, main body of this report).

Two inter-related work activity plans, the *MRS 16 Prescribed Burn Plan, (Presidio of Monterey[POM] Fire Department, 2006)*, and *Munitions Response Site 16 Site Specific Work Plan* (main body of this report) will be implemented during the completion of the prescribed burn at MRS-16.

The Burn Plan describes the objectives of the prescribed burn; the burn area; the range of environmental conditions under which the burn will be conducted; the manpower and equipment resources required to ignite, manage, and execute the fire; and the smoke management plan. Procedures for conducting the burn within the window of environmental conditions established in the burn prescription are discussed in the Burn Plan.

The Site-Specific Work Plan (SSWP) describes the procedures, methods, and resources that Shaw and its subcontractors will use while performing MEC removal activities as well as other monitoring activities related to the program.

The Air Sampling and Analysis Plan (this document) is presented as appendix L to the SSWP, and outlines procedures for the collection and analysis of air samples during the prescribed burn. The purpose of collecting and analyzing air samples during the prescribed burn is to 1) provide data to assess the adequacy of the burn prescription relative to smoke dispersion and downwind impacts and 2) monitor and evaluate whether prescribed burns at the former Fort Ord result in downwind ambient concentrations of PM_{10} that exceed the applicable health-based screening level (California ambient air quality standard).

L2.0 PROJECT ORGANIZATION

The work described in this SAP will be performed by MACTEC Engineering and Consulting, Inc. (MACTEC) under subcontract to Shaw Environmental, Inc (Shaw). The overall project organization is presented in the SSWP (main body of this report). Project organization for key MACTEC personnel is described below.

L2.1 Project Manager

The MACTEC Project Manager for the former Fort Ord project, Mr. Bruce Wilcer, is responsible for the implementation of USACE and regulatory agency requirements for the project, and reports directly to the Shaw Task Manager. These responsibilities may include oversight of the development and completion of project documents and related activities as well as schedule and contract management, technical oversight, report generation, implementation of three-phase QC activities, and overall project quality.

L2.2 Task Manager

The MACTEC Task Manager for the sampling program described in this SAP, Mr. Matthew Walraven, is responsible for implementation and oversight of activities related to project performance, and reports directly to the MACTEC Project Manager. This responsibility includes development of project documents, implementation of project activities, management of the schedule and contract, oversight of technical issues, generation of reports, and implementation of the QC procedures specified in this SAP.

L2.3 Project Chemist

The Project Chemist, Mr. Scott Tucker, provides input for development of this SAP, as well as management of project tasks associated with sampling and preservation requirements, general oversight of field personnel in sampling activities, and coordination of sample collection and shipment to the analytical laboratory. The Project Chemist reports directly to the MACTEC Project Manager and provides oversight of and coordination with the analytical laboratory, review and evaluation of analytical

data, and implementation of three-phase QC activities and corrective actions (as necessary). The Project Chemist will also conduct a project kick-off meeting with the analytical laboratory prior to sample collection or analysis.

L3.0 PRESCRIBED BURN OPERATIONS

A description of the project background, including the location of the site, historical use, and description of MEC use at MRS-16 is provided in the SSWP. The following section summarizes the prescribed burn operations.

The Army recognizes that smoke produced by prescribed burn events has the potential to create short-term impacts on local air quality and potential temporary impacts on public health downwind. Fire and smoke management procedures are outlined in the *MRS-16 Prescribed Burn Plan*, (Prescribed Burn Plan) (POM Fire Department, 2006). Approximately 58 acres of vegetation is expected to be burned in approximately 3 hours of active ignition, and smoke will likely be in the air for several more hours as described in the Prescribed Burn Plan. As a result of information gained from the prescribed burn performed in Ranges 43-48 in October 2003 (described in the *Draft Final, Ranges 43-48 Prescribed Burn Air Monitoring Report [MACTEC, 2004]*), the following components will comprise the prescribed burn at MRS-16:

- Cutting of approximately 28 acres of vegetation comprising the MRS-16 fuel break
- Prescribed burning of approximately 58 acres of vegetation
- Implementation of the burn prescription which includes fuel moisture and atmospheric condition criteria as identified in the Prescribed Burn Plan. Procedures designed to enhance smoke management; including optimization of ignition patterns and modification of the burn prescription are also described in the Prescribed Burn Plan
- Notification to local communities prior to the prescribed burn event at MRS-16, using the procedures described in Appendix M - Notification Plan, in this document.

L4.0 PROJECT DESCRIPTION

This section provides a brief description of the project in terms of objective and scope. Details of the sampling program are provided in subsequent sections.

L4.1 Objective and Scope

This SAP outlines procedures for collection and analysis of air samples in areas potentially affected by air emissions from a prescribed burn at MRS-16. The results of other relevant studies and investigations have been considered during the development of this SAP to determine the chemicals of potential concern (COPCs) that will be monitored during the prescribed burn. In accordance with the California Clean Air Act (Title 17, CCR, 80100 et. seq.), a requirement for appropriate monitoring of burns that affect smoke sensitive areas is part of the Monterey Bay Unified Air Pollution Control District's (MBUAPCD) Smoke Management Program (SMP). Potential methods identified in the SMP for monitoring smoke impacts include visual observation and PM₁₀ analysis. Data collected from air monitoring will be used to plan future prescribed burns. As described earlier, the objectives of the sampling and analysis program described herein are to:

1. Provide data to assess the adequacy of the burn prescription relative to smoke dispersion and downwind impacts. The air sampling program is therefore focused on detection and quantification of particulate matter related to vegetation combustion (PM₁₀).
2. Evaluate whether prescribed burns at the former Fort Ord result in downwind ambient concentrations of PM₁₀ that exceed the applicable health-based screening level (California ambient air quality standard).

Air monitoring for PM₁₀ will be conducted starting at the active burn stage and continuing for a 24-hour period during the prescribed burn effort.

L5.0 PROJECT OVERVIEW AND DATA QUALITY OBJECTIVES

L5.1 Overview

During a prescribed burn, the smoke generated from the burning of vegetation may contain inhalable particulate matter (suspended particles less than 10 microns in diameter [PM_{10}]), which can cause temporary respiratory distress to sensitive populations during the burn event. The intense fire associated with prescribed burn conditions may also result in the incidental detonation of surface or near-surface MEC items. Detonation of MEC present at the site has the potential to release air pollutants to the atmosphere. These additional air emissions may potentially include combustion products, volatile or semivolatile organic compounds, unburned or incompletely burned energetic material, and particulate metals and metal compounds from chemical components of the MEC items. Although MEC-related emissions may also be generated during a prescribed burn, ground-level concentrations of these pollutants have been shown to remain well below human health-protective regulatory screening levels as described in the *Draft Final, Ranges 43-48 Prescribed Burn Air Monitoring Report (MACTEC, 2004)*. Data presented in the *Draft Prescribed Burn Supplemental Report, Ranges 43-48 (MACTEC, 2005)* also indicates that, with the exception of inhalable particulate matter, there is no sound technical reason for sampling air emissions resulting from combustion of vegetation such as acrolein and aldehydes. As a result, only samples for inhalable particulate matter (PM_{10}) will be collected during the MRS-16 prescribed burn. Smoke management techniques described in the SSWP will be employed by the Army to minimize public exposure to smoke from prescribed burns at the former Fort Ord. The purpose of the sampling and analysis program described herein is to:

1. Provide data to assess the adequacy of the burn prescription relative to smoke dispersion and downwind impacts.

2. Monitor and evaluate whether prescribed burns at the former Fort Ord result in downwind ambient concentrations of PM₁₀ that exceed the applicable health-based screening level (California ambient air quality standard).

Data quality objectives (DQOs) for the project are discussed in the sections below.

L5.2 Data Quality Objectives

L5.2.1 Statement of the Problem

Combustion of vegetation has potential temporary smoke impacts to downwind sensitive receptors.

These smoke impacts need to be evaluated relative to human health screening levels. The identity and quantity of products and residues emitted to the air from prescribed burn activities at the former Fort Ord has been documented in previous studies, including a previous ambient air sampling investigation conducted at Ranges 43-48 (*MACTEC, 2004*). This previous investigation has concluded that MEC-related air contaminants downwind of that prescribed burn at the former Fort Ord did not exceed health-based screening levels, and that the best indicator of smoke impacts at public receptors is PM₁₀ (*MACTEC, 2005a*).

L5.2.2 Identification of Decisions

The primary decisions related to this project are to (1) evaluate whether prescribed burns at the former Fort Ord result in downwind ambient concentrations of PM₁₀ that exceed the applicable health-based screening level (California ambient air quality standard), and (2) provide data to support the evaluation of the burn prescription relative to smoke dispersion and downwind impacts to the public.

L5.2.3 Identification of Inputs to Decisions

Inputs to decisions necessary for evaluating downwind impacts from prescribed burn activities at former Fort Ord are as follows:

- Identify target list of COPCs

The target list of COPCs for the MRS-16 prescribed burn has been identified through evaluation of data presented in previous technical publications, including the *Draft Final Summary After-Action Report: Ranges 43-48 Prescribed Burn, Former Fort Ord, California (Army, 2004) Draft Final, Prescribed Burn Supplemental Report, Ranges 43-48 (MACTEC 2006)*, and *Health Consultation, Former Fort Ord Site (Agency for Toxic Substances and Disease Registry, 2005)*. Based on the information provided in these documents, PM₁₀ was selected as the COPC for the MRS-16 prescribed burn.

- Identify appropriate screening levels for COPCs (i.e., PM₁₀) in air.
- For PM₁₀, the California Ambient Air Quality Standard for was selected as the most appropriate screening level for the project. This decision is based on previous coordination between the Army, USACE, U.S. Environmental Protection Agency, Region 9 (EPA), California Environmental Protection Agency (Cal EPA) Department of Toxic Substances Control, (DTSC), California Air Resources Board (CARB), and Monterey Bay Unified Air Pollution Control District (MBUAPCD) during the development of the sampling and analysis plan for the prescribed burn at Ranges 43-48 (*MACTEC, 2003*).
- Measure downwind concentrations of COPCs in air during a prescribed burn event.
- Record field observations, including wind speed and direction during the event, and visually identify the downwind area that received smoke impacts.

L5.2.4 Definition of Study Boundaries

The study boundary is defined as the area downwind of the prescribed burn event that includes adjacent populations that could receive smoke impacts. The maximum extent of placement of samplers (study boundary) will be based on predictions of air flow patterns from air modeling studies and information collected from previous burn events. Air samples will be collected on the day of the prescribed burn event, over a 24-hour period.

L5.2.5 Development of Decision Rules

If measured concentrations of PM₁₀ in air are less than established screening levels, then no modifications will be made to future prescribed burn operations.

If measured concentrations of PM₁₀ in air are greater than or equal to established screening levels, then modifications to future prescribed burn operations will be evaluated.

L5.2.6 Specification of Limits on Decision Errors

The null hypothesis is that, following this investigation, no modifications to future prescribed burn operations will be necessary ("future prescribed burn operations" in this context includes burn tactics for separate burns at later dates).

A false positive decision error would be to conclude that modifications are necessary when, in fact, they are not. The consequence of this error would be that unnecessary modifications or limitations to future prescribed burn operations would be made, resulting in unnecessary cost to the government.

A false negative decision error would be to conclude that modifications are not necessary when, in fact, they are. The consequence of this error would be that future prescribed burn operations with unacceptable adverse impacts to human health would occur.

This investigation employs a biased sampling strategy designed to characterize areas of maximum impact. Consequently, confidence limits on decision errors are not applicable to this investigation. The expected COPC distribution is not random; hence, the judgmental sampling strategy proposed does not lend itself to statistically derived confidence levels for decision errors. However, decision errors will be controlled by adhering to the procedures specified in this SAP, including specific QA/QC procedures as defined in the sampling and analytical methods. In particular, analytical laboratory QA/QC acceptance criteria will be used to verify that measurements are capable of supporting project decisions.

L5.2.7 Optimization of Investigation Design for Obtaining Data

The sampling investigation design includes the following factors:

- Number of air samples
- Location of air samples
- Sampling and analytical methods
- QC samples.

Rationale for the sampling investigation design includes the following factors and objectives:

- To obtain samples that characterize the degree of smoke impact to downwind public receptors
- To obtain samples that characterize the spatial extent of smoke impact
- Because the downwind COPC concentration distribution is nonrandom within the study area, a judgmental sampling design is appropriate. Also, because decisions will not be made using mean concentrations compared to the screening level, probabilistic sampling design is not useful.

Factors to consider to optimize sampling investigation design include the following:

- The location of the highest concentrations of PM₁₀ in air may vary during the event as meteorological conditions evolve throughout the day
- Field sampling methods should facilitate rapid mobilization of sampling units to locations of predicted smoke impact the day of or day before the prescribed burn event.

The sampling strategy for PM₁₀ in air will be to conduct the following activities:

- Collect air samples in multiple areas most likely to experience smoke impacts (based on observations from previous prescribed burn events, wind direction predictions for the specific burn day, and/or pre-burn smoke dispersion modeling).,
- Collect time weighted average (TWA) air samples from onset of the prescribed burn and lasting until active burning is completed (called “burn event” sample; estimated to be about 8 to 10 hours)
- After the conclusion of the burn event sample, collect a second TWA sample to complete a 24-hour sample interval (e.g., if the burn event is 8 to 10 hours, then the second sample will be 14 to 16 hours in duration)
- Samples shall be measured/analyzed for PM₁₀ as shown in Table 1.

L6.0 SAMPLING PROCESS DESIGN

Results of the DQO process described in Section 5.0 were used to develop the sampling process design for this project. This section describes the details of the planned sampling, such as the number and location of samples. The rationale and assumptions used for sample process design are presented in Section 5.7. Section 7.0 provides a description of the sampling and analytical methods.

L6.1 Objective and Basic Design

The primary objectives of this field investigation are to:

1. Determine if ground-level concentrations of PM₁₀ downwind of a burn event are above or below the applicable health-based screening level (California ambient air quality standard), and
2. Assist in assessing the adequacy of the burn prescription for minimizing downwind smoke impacts to the public.

The California Ambient Air Quality Standard human health standard for PM₁₀ is based on a time-weighted average (TWA) for a 24-hour period. In order to collect a TWA 24-hour sample, and have the ability to assess the highest impact periods during the burn, two sampling intervals will be used. The first set of samples will be collected during the burn event, which is expected to last approximately 8 to 10 hours. The burn event includes approximately 3 hours of active ignition and several subsequent hours during which visible smoke may continue to be present. A second set of samples will be collected following the burn event to comprise the TWA sample for the remainder of the 24 hour period.

This air sampling program may include support and participation from both Army and regulatory agency resources. Most sampling equipment and field personnel will be provided by MACTEC under subcontract to Shaw, who is contracted directly with the USACE.

The sampling locations and procedures described in this SAP will be followed to the maximum practicable extent during this field investigation. However, deviations from the SAP may be required because of unforeseen conditions in the field. Any deviations from the SAP must be approved by the Task Manager prior to implementation, and will be documented in writing in a Field Change Notification.

L6.2 Air Sampling Locations

A total of seven sampling locations (five that are pre-selected and two that will be located the day of, or day before the burn) are proposed for the MRS-16 Prescribed Burn. The proposed five pre-selected sampling stations are located in residential or otherwise public areas that, based on the data collected during past burn events, may be affected by smoke impacts (Plate 1). Because the actual areas of smoke impact cannot be known in advance of a burn event, the five pre-selected sampling stations will be supplemented with two sampling stations that will be deployed in one of six proposed areas, based on the smoke dispersion modeling the day before the prescribed burn. The sampling program described here for MRS-16 requires only PM₁₀ analysis, which does not require commercial electrical power to run the equipment, allowing the selected stations to be located quickly to areas of predicted smoke impacts the day before or on the day of the burn, as appropriate.

The decision on where to place the selected sampling stations on the day of the prescribed burn will be made by the Task Manager in the field based on modeling results and will focus on areas between pre-selected sampling units that may receive smoke impacts. Plate 1 presents six general candidate locations for additional sampling stations. Two of these six locations will be selected by the task manager on or before the day of the burn based on the above observations. The actual final locations of the stations will be recorded using Global Positioning System (GPS) technology.

L7.0 SAMPLING AND ANALYTICAL METHOD REQUIREMENTS

The proposed sampling program includes collecting PM₁₀ samples as summarized in Table 1 as follows:

- TWA air samples during the active burning (burn event sample) approximately 8-10 hours
- Second set of TWA samples collected over a duration that will complete the 24-hour monitoring period.
- One trip blank sample will be submitted to the laboratory for each sampling event per day
- Duplicate (QC) air samples (one duplicate for each sampling event, per day).

Air samples will be collected at seven locations for a 24 hour period during the burn day events:

Air samples for PM₁₀ will be integrated TWA samples where samples are collected on or in a specific media for subsequent laboratory analysis. Each 24-hour TWA sample will be comprised of approximate 8- and 14-hour samples beginning at the initiation of the burn and terminating approximately 24 hours later. All samples will be collected at approximately two (2) meters above ground level (agl), which is at or near human adult breathing zone and within the probe siting criteria recommended by the USEPA (USEPA, 1987). USEPA guidance for spacing from obstructions will also be followed.

The air sampling and analytical methods that will be used to monitor PM₁₀ in this investigation are summarized in Table 1 and described in the following sections. The analytical laboratory for this project will be certified by the State of California Environmental Accreditation Program, as applicable, and analyses will be performed in accordance with the requirements set forth in USEPA Compendium Method IO-3.1. All sampling media will be maintained under strict chain-of-custody (COC) control. Sampling media will be uniquely identified and labeled, and the time, location, and duration of exposure will be recorded on COC forms. All COC forms will be completed and signed by the sampling technician

before relinquishing the exposed media for shipment to the analytical laboratory. Duplicate samples will be handled identically to exposed media, and will be submitted “blind” to the laboratory for analysis.

L7.1 Particulate Matter Less than 10 Microns (PM₁₀)

Sampling Method

To maximize the flexibility of the sampling program, battery-operated low-volume sampling equipment (e.g., Airmetrics MiniVol® Sampler or equivalent) will be used to collect integrated TWA air samples for PM₁₀ analysis at the seven sampling locations. Samples will be collected using Teflon® filter media. Sampling operations will be in accordance with the Standard Operating Procedure number MACTEC SOP 104 (Appendix C) that meets the requirements established in Reference 1, USEPA Compendium Method IO-2.1 (Appendix A, modified for low volume sampling).

Analysis Method

Filter mass concentration of the low-volume air sampler will be determined gravimetrically. Pre- and post-exposure filter weights will be assessed with a microbalance. The total volume of air sampled, corrected to USEPA reference condition, is determined from the measured air flow rate and the sample time. The mass concentration of PM₁₀ in the ambient air is computed as the total mass of captured particles in the PM₁₀ size range divided by the volume of air sampled, and is expressed in micrograms per cubic meter (µg/m³). Filter weighing procedures will be conducted by an analytical laboratory and will meet requirements specified in Reference 2, USEPA Compendium Method IO-3.1 (Appendix B).

Performance Criteria and Quality Control

One field duplicate sample will be collected and analyzed per day of sampling, and field (trip) blanks will be submitted on a frequency of 10% of total samples (the field blank filter will be shipped to the field and returned without drawing air through the sampler). Laboratory QA/QC samples, at a minimum, will be performed at the frequency specified in the analytical method. Analytical parameters such as initial

calibration and instrument conditions will be in compliance with the acceptance criteria as specified in the analytical method.

References

1. USEPA Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Compendium Method IO-2.1. Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM₁₀ Using High-Volume (HV) Sampler (modified for low volume and less than 24 hour sampling). EPA/625/R-96/010a, Center for Environmental Research Information, Cincinnati, Ohio. June 1999.
2. USEPA Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. Compendium Method IO-3.1. Selection, Preparation and Extraction of Filter Material. EPA/625/R-96/010a, Center for Environmental Research Information, Cincinnati, Ohio, June 1999.
3. MACTEC. Standard Operating Procedure for Airmetrics MiniVol® Sampler. MACTEC SOP 104. March 2006.

L7.2 Decontamination Procedures

Each air sample will be collected on individual sampling media as supplied by the laboratory.

Decontamination procedures will not be required because all sample tubing and pumps will be downstream of the collection media, and no sampling media will be re-used.

L8.0 INVESTIGATION-DERIVED WASTE

Investigation-derived waste (IDW) consists of tools, personal protective equipment, soil cuttings, wash water, and other disposable items that may have come into contact with site contaminants. IDW is not expected as part of this investigation.

L9.0 QUALITY CONTROL

Contractor QC activities relative to the acquisition and reporting of chemical data are described in the CDQMP (*MACTEC, 2002*). The project and task managers and the project chemist for this project are described in Section 2.0 of this SAP. This section describes the QC procedures specific to the MRS-16 prescribed burn air sampling.

L9.1 Three-Phase Quality Control Process

The three-phase QC process, as described below, will be implemented for the prescribed burn air sampling activities. Each phase of QC is important for obtaining a quality product, however since the prescribed burn is anticipated to be a one day event, the preparatory and initial inspections are most applicable to the scope of the project. Production work is not to be performed on a definable feature of work until a successful preparatory and initial phase inspection have been completed.

During these inspections, the Project Manager will verify implementation of the requirements of this SAP and the relevant requirements of the CDQMP.

L9.1.1 Preparatory Phase Inspection

Before conducting a feature of work, the Project Manager shall check that technical requirements have been planned for and that work prerequisites have been identified and met. The Project Manager's responsibility is to check that lessons learned during previous similar work have been incorporated as appropriate into the project procedures to prevent recurrence of past problems. Minimum review criteria for a preparatory phase inspection are contained in Section 4.1.3 of the CDQMP (*MACTEC, 2002*).

Discrepancies among existing conditions and approved plans/procedures are to be resolved before the day of the prescribed burn, and completion of corrective actions for unsatisfactory and nonconforming conditions identified during a preparatory inspection is to be verified by the Project or Task Manager prior to granting approval to begin work. In addition, the Project Chemist will hold a project kick-off

meeting with the analytical laboratory to discuss the analytical and deliverable requirements for this project as described in this SAP. Minutes of this meeting will be documented and reported to Shaw in a letter report. Laboratory kick off meetings and necessary technical systems audits will be completed prior to the submittal of samples for analysis.

L9.1.2 Initial Phase Inspection

The second QC phase consists of checks performed during the initial field activities. On the day of the prescribed burn, the Task Manager will monitor the work and verify compliance with the specifications and requirements of the contract, delivery order, and approved plan and procedures. Minimum review criteria for the initial phase inspection are provided in Section 4.1.3 of the CDQMP (*MACTEC, 2002*). The Task Manager is also responsible for verifying that a daily health and safety (H&S) inspection is performed and documented as prescribed in the site safety and health plan (SSHP). The Project Manager or designee is responsible for ensuring that discrepancies between site practices and approved specifications are identified and resolved. Discrepancies between site practices and approved plans/procedures are to be resolved and corrective actions for unsatisfactory and nonconforming conditions or practices are to be verified by the Project Manager or designee prior to granting approval to proceed. .

L9.1.3 Follow-Up Phase Inspection

The follow up phase of the three phase quality control process is designed for projects of multiple day duration, and do not apply as described in Section 4.1.3 of the CDQMP. The follow up phase inspection for this project will consist of a review of the samples collected and documentation of sample collection for compliance with the specifications described in this SAP by the Task Manager. Documentation of any deviation from the project plans will be verified by the Project Manager and provided in writing to Shaw as a non-conformance report.

L9.2 Additional Inspections

Additional inspections performed on the same definable feature of work may be required at the discretion of the USACE or the Project Manager. Additional preparatory and initial inspections are generally warranted under any of the following conditions: unsatisfactory work, as determined by program or project management or USACE; changes in key personnel; resumption of work after a substantial period of inactivity (e.g., 2 weeks or more); and changes to the project scope of work/specifications.

L9.3 Deliverables

The following deliverables will be prepared:

- Laboratory kick off meeting minutes
- Comprehensive certificate of analysis (from analytical laboratory).
- Non-Conformance Report (as needed)

L9.4 Data Validation

Data validation will consist of review and re-calculation of the laboratory raw data to verify accuracy of concentrations reported. The laboratory will provide the equivalent of an EPA Level 4 data package for each data set submitted for analysis. The Project Chemist will be responsible for coordinating or performing data validation efforts.

L10.0 REFERENCES

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TABLES

**Table L1. Screening Level, Sampling and Analytical Methods
and QA/QC Requirements for PM₁₀
Prescribed Burn Air Sampling and Analysis Plan
Former Fort Ord, California**

Analyte Class	Analyte	Air Screening Level ($\mu\text{g}/\text{m}^3$)	Screening Level Reference
Particulate Matter	Particulate Matter less than 10 microns (PM ₁₀)	50(24-hour)	California AAQS ¹

¹ California Ambient Air Quality Standard

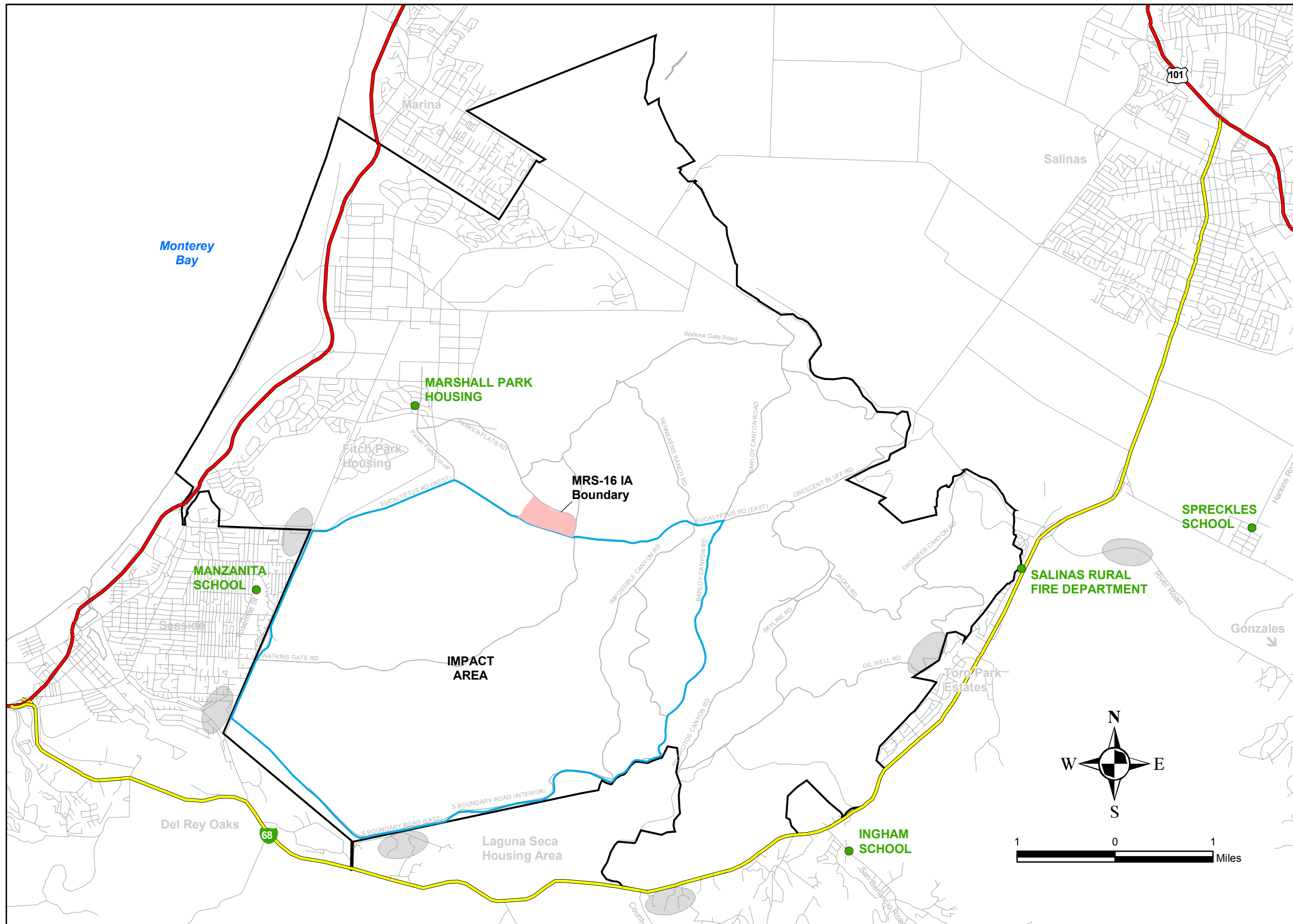
Pollutant	Sampling Equipment	Sampling Method	Analytical Method	Quality Assurance / Quality Control		
				Field Duplicates	Field Blanks	Lab QA/QC
Particulate Matter < 10 microns (PM ₁₀)	Low Volume Sampler with Size-Selective Inlet equipped with Teflon filter (Airmetrics MiniVol®)	USEPA Compendium Method IO-2.1, modified for low volume and less than 24 hour sampling	USEPA Compendium Method IO-3.1	One per day of sampling	10%	See Note 1

Note 1: Laboratory QA/QC samples, at a minimum, will be performed at the frequency specified in the analytical method. Analytical parameters such as initial calibrations and instrument conditions will be in compliance with the acceptance criteria as specified in the analytical method.

Checked:  _____

Approved:  _____

PLATES



- EXPLANATION**
- Proposed Monitoring Station
 - Potential Monitoring Station*
 - Impact Area
 - Munitions Response Site 16 Interim Action Boundary
 - Fort Ord Boundary
 - Road Classification Limited Access Highway
 - Highway
 - Roads

* Specific locations will be selected the day before or the day of the burn based on meteorological modeling results.

DRAWN: TJH	PROJECT NO: 4088053164 04.3
ENGINEER:	SCALE: AS SHOWN
CHECKED:	APPROVED:
DATE: 05/2006	DATE:



Prescribed Burn
Air Sampling and Analysis Plan
Former Fort Ord, California

Munition Response Site 16 and
Proposed Monitoring Locations

PLATE

1

ATTACHMENT L-A

USEPA COMPENDIUM METHOD IO-2.1

**Compendium of Methods
for the Determination of
Inorganic Compounds
in Ambient Air**

Compendium Method IO-2.1

**SAMPLING OF AMBIENT AIR
FOR TOTAL SUSPENDED
PARTICULATE MATTER (SPM)
AND PM₁₀ USING
HIGH VOLUME (HV) SAMPLER**

Center for Environmental Research Information
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268

June 1999

Method IO-2.1

Acknowledgments

This Method is a part of *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air* (EPA/625/R-96/010a), which was prepared under Contract No. 68-C3-0315, WA No. 2-10, by Midwest Research Institute (MRI), as a subcontractor to Eastern Research Group, Inc. (ERG), and under the sponsorship of the U.S. Environmental Protection Agency (EPA). Justice A. Manning, John O. Burckle, Scott Hedges, Center for Environmental Research Information (CERI), and Frank F. McElroy, National Exposure Research Laboratory (NERL), all in the EPA's Office of Research and Development, were responsible for overseeing the preparation of this method. Other support was provided by the following members of the Compendia Workgroup:

- James L. Cheney, U.S. Army Corps of Engineers, Omaha, NE
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- Robert G. Lewis, U.S. EPA, NERL, RTP, NC
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- William A. McClenny, U.S. EPA, NERL, RTP, NC
- Frank F. McElroy, U.S. EPA, NERL, RTP, NC
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

This Method is the result of the efforts of many individuals. Gratitude goes to each person involved in the preparation and review of this methodology.

Author(s)

- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

Peer Reviewers

- David Brant, National Research Center for Coal and Energy, Morgantown, WV
- John Glass, SC Department of Health and Environmental Control, Columbia, SC
- Jim Cheney, U.S. Army Corps of Engineers, Omaha, NE
- Margaret Zimmerman, Texas Natural Resource Conservation Commission, Austin, TX
- Lauren Drees, U.S. EPA, NRMRL, Cincinnati, OH

DISCLAIMER

This Compendium has been subjected to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Method IO-2.1
Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM₁₀ Using High Volume (HV) Sampler

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Chapter IO-2 Integrated Sampling of Suspended Particulate Matter (SPM)

Method IO-2.1 SAMPLING OF AMBIENT AIR FOR TOTAL SUSPENDED PARTICULATE MATTER (SPM) AND PM₁₀ USING HIGH VOLUME (HV) SAMPLER

1. Scope

1.1 Suspended particulate matter (SPM) in air generally is a complex, multi-phase system of all airborne solid and low vapor pressure liquid particles having aerodynamic particle sizes from below 0.01-100 μm and larger. Historically, SPM measurement has concentrated on total suspended particulates (TSP), with no preference to size selection.

1.2 The U. S. Environmental Protection Agency (EPA) reference method for TSP is codified at 40 CFR 50, Appendix B. This method uses a high-volume sampler to collect particles with aerodynamic diameters of approximately 100 μm or less. The high-volume samples 40-60 ft^3/min of air with the sampling rate held constant over the sampling period. The high-volume design causes the TSP to be deposited uniformly across the surface of a filter located downstream of the sampler inlet. The TSP high-volume can be used to determine the average ambient TSP concentration over the sampling period, and the collected material subsequently can be analyzed to determine the identity and quantity of inorganic metals present in the TSP.

1.3 Research on the health effects of TSP in ambient air has focused increasingly on particles that can be inhaled into the respiratory system, i.e., particles of aerodynamic diameter less than 10 μm . The health community generally recognizes that these particles may cause significant adverse health effects. Recent studies involving particle transport and transformation strongly suggest that atmospheric particles commonly occur in two distinct modes: the fine (< 2.5 μm) mode and the coarse (2.5-10.0 μm) mode. The fine or accumulation mode (also termed the respirable particulate matter) is attributed to growth of particles from the gas phase and subsequent agglomeration, while the coarse mode is made of mechanically abraded or ground particles. Particles that have grown from the gas phase (either because of condensation, transformation, or combustion) occur initially as very fine nuclei--0.05 μm . These particles tend to grow rapidly to accumulation mode particles around 0.5 μm which are relatively stable in the air. Because of their initially gaseous origin, particle sizes in this range include inorganic ions such as sulfate, nitrate, ammonia, combustion-form carbon, organic aerosols, metals, and other combustion products. Coarse particles, on the other hand, are produced mainly by mechanical forces such as crushing and abrasion. Coarse particles, therefore, normally consist of finely divided minerals such as oxides of aluminum, silicon, iron, calcium, and potassium. Coarse particles of soil or dust mostly result from entrainment by the motion of air or from other mechanical action within their area. Since the size of these particles is normally > 2.5 μm , their retention time in the air parcel is shorter than the fine particle fraction.

1.4 On July 1, 1987, the U. S. Environmental Protection Agency (EPA) promulgated a new size-specific air quality standard for ambient particulate matter. This new primary standard applies only to particles with aerodynamic diameters \leq 10 micrometers (PM₁₀) and replaces the original standard for TSP. To measure concentrations of these particles, the EPA also promulgated a new federal reference method (FRM). This method is based on the separation and removal of non-PM₁₀ particles from an air sample, followed by filtration and gravimetric analysis of PM₁₀ mass on the filter substrate.

1.5 The new primary standard (adopted to protect human health) limits PM_{10} concentrations to $150 \mu\text{g}/\text{std. m}^3$ averaged over a 24-h period. These smaller particles are able to reach the lower regions of the human respiratory tract and, therefore, are responsible for most of the adverse health effects associated with suspended particulate pollution. The secondary standard, used to assess the impact of pollution on public welfare, has also been established at $150 \mu\text{g}/\text{std. m}^3$.

1.6 Ambient air SPM measurements are used (among other purposes) to determine whether defined geographical areas are in attainment or non-attainment with the national ambient air quality standards (NAAQS) for PM_{10} . These measurements are obtained by the States in their State and local air monitoring station (SLAMS) networks as required under 40 CFR Part 58. Further, Appendix C of Part 58 requires that the ambient air monitoring methods used in these EPA-required SLAMS networks must be methods that have been designated by the EPA as either reference or equivalent methods.

1.7 Monitoring methods for particulate matter are designated by the EPA as reference or equivalent methods under the provisions of 40 CFR Part 53, which was amended in 1987 to add specific requirements for PM_{10} methods. Part 53 sets forth functional specifications and other requirements that reference and equivalent methods for each criteria pollutant must meet and explicit test procedures by which candidate methods or samplers are to be tested against those specifications. General requirements and provisions for reference and equivalent methods are also given in Part 53, as are the requirements for submitting an application to the EPA for a reference or equivalent method determination.

1.8 Several methods are available for measuring SPM in ambient air. As mentioned earlier, the most commonly used device is the high-volume sampler, which consists essentially of a blower and a filter, and which is usually operated in a standard shelter to collect a 24-h sample. The sample is weighed to determine concentration and may be analyzed chemically. The high volume sampler is considered a reliable instrument for measuring the mass concentration of TSP in ambient air. When EPA first regulated TSP, the NAAQS was stated in terms of SPM captured on a filter with aerodynamic particle size of $< 100 \mu\text{m}$ as defined by the TSP sampler; therefore, the TSP sampler was used as the reference method.

1.9 Under Part 53 requirements, reference methods for PM_{10} must be shown to use the measurement principle and meet other specifications set forth in 40 CFR 50, Appendix J. They must also include a PM_{10} sampler that meets the requirements specified in Subpart D of 40 CFR 53. Appendix J specifies a measurement principle based on extracting an air sample from the atmosphere with a powered sampler that incorporates the inertial separation of PM_{10} size range particles followed by the collection of PM_{10} particles on a filter over a 24 h period. The average PM_{10} concentration for the sample period is determined by dividing the net weight gain of the filter over the sample period by the total volume of air sampled, corrected to EPA's standard temperature (25EC) and standard pressure (760 mm Hg). Other specifications for flow rate control and measurement, flow rate measurement device calibration, filter media characteristics and performance, filter conditioning before and after sampling, filter weighing, sampler operation, and correction of sample volume to EPA reference temperature and pressure are prescribed in Appendix J. In addition, sampler performance requirements in Subpart D of Part 53 include sampling effectiveness (the accuracy of the PM_{10} particle size separation capability) at each of the three wind speeds and at "50% cutpoint" (the primary measure of 10- μm particle size separation). Field tests for sampling precision and flow rate stability are also specified. In spite of the instrumental nature of the sampler, this method is basically a manual procedure, and all designated reference methods for PM_{10} are therefore defined as manual methods.

1.10 This document describes the procedures for sampling SPM in ambient air for both TSP and PM₁₀ based upon active sampling using a high volume air sampler. The ambient particles are collected on quartz fiber filters. The sampler collects TSP or PM₁₀ ambient particles depending upon the type of inlet selected.

2. Applicable Documents

2.1 ASTM Documents

- D4096 *Application of the High Volume Sample Method for Collection and Mass Determination of Airborne Particulate Matter.*
- D1356 *Definition of Terms Related to Atmospheric Sampling and Analysis.*
- D1357 *Practice for Planning the Sampling of the Ambient Atmosphere.*
- D2986 *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test.*

2.2 Other Documents

- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: A Field Guide for Environmental Quality Assurance*, EPA/600/R-94/038a.
- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition)*, EPA/600/R-94/038b.
- *Reference Method for the Determination of Particulate Matter in the Atmosphere*, 40 CFR 50, Appendix J.
- *Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)*, 40 CFR 50, Appendix B.
- *Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air*, 40 CFR 50, Appendix G.
- *Reference Method for this Determination of Suspended Particulates in the Atmosphere (PM₁₀ Method)*, 40 CFR 50, Appendix J.

3. Summary of Method

3.1 The sampling of a large volume of atmosphere, 1,600-2,400 m³ (57,000-86,000 ft³), with a high-volume blower, typically at a rate of 1.13-1.70 m³/min (40-60 ft³/min), is described in this method. The calibration and operation of typical equipment used in this sampling is also described.

3.2 Air is drawn into the sampler and through a glass fiber or quartz filter by means of a blower, so that particulate material collects on the filter surface. Without a 10 µm size-selective inlet, particles of 100 µm size and less enter the sampling inlet and are collected on the downstream filter. The collection efficiencies for particles larger than 20 µm decreases with increasing particle size, and it varies widely with the angle of the wind with respect to the roof ridge of the sampler shelter. When glass fiber filters are used, particles 100-0.1 µm or less in diameters are ordinarily collected. With a size-select inlet, particles 10 µm diameter or less are collected on the quartz filter.

3.3 The upper limit of mass loading is determined by plugging the filter medium with sample material, which causes a significant decrease in flow rate. For very dusty atmospheres, shorter sampling periods will be necessary.

3.4 The volume of air sampled is determined by a flow-rate indicator. The instrument flow-rate indicator is calibrated against a reference orifice meter. The latter is a working standard which, in turn, has been calibrated against a master flow meter certified by the National Institute of Standards and Technology (NIST).

3.5 Airborne particulate matter retained on the filter may be examined or analyzed chemically by a variety of methods (ICP, ICP/MS, AA, GFAA, and NAA) as delineated in Inorganic Compendium Methods IO-3.2 through IO-3.7.

4. Significance

4.1 The area of toxic air pollutants has been the subject of interest and concern for many years. Recently the use of receptor models has resolved the elemental composition of atmospheric aerosol into components related to emission sources. The assessment of human health impacts resulting in major decisions on control actions by federal, state and local governments is based on these data. Accurate measures of toxic air pollutants at trace levels is essential to proper assessments.

4.2 The high volume sampler is commonly used to collect the airborne particulate component of the atmosphere. A variety of options available for the sampler provides broad versatility and allows the user to develop information about the size and quantity of airborne particulate material and, using subsequent chemical analytical techniques, information about the chemical properties of the particulate matter. The advent of inductively coupled plasma spectroscopy has improved the speed and performance of metals analysis in many applications.

5. Definitions

[Note: Definitions used in this document are consistent with those used in ASTM Methods. All pertinent abbreviations and symbols are defined within this document at point of use.]

5.1 High-Volume Air Sampler (HV). A device for sampling large volumes of an atmosphere for collecting the contained particulate matter by filtration. Consists of a high-capacity blower, a filter to collect suspended particles, and a means for measuring the flow rate.

5.2 Working Flow-Rate Standard. A flow-rate measuring device, such as a standard orifice meter, that has been calibrated against a master flow-rate standard. The working flow-rate standard is used to calibrate a flow measuring or flow rate indicating instrument.

5.3 Master Flow-Rate Standard. A flow-rate measuring device, such as a standard orifice meter, that has been calibrated against a primary standard.

5.4 Primary Flow-Rate Standard. A device or means of measuring flow rate based on direct primary observations such as time and physical dimensions.

5.5 Spirometer. A displacement gasometer consisting of an inverted bell resting upon or sealed by liquid (or other means) and capable of showing the amount of gas added to or withdrawn from the bell by the displacement (rise or fall) of the bell.

5.6 Absolute Filter. A filter or filter medium of ultra-high collection efficiency for very small particles (submicrometer size) so that essentially all particles of interest or of concern are collected. Commonly, the efficiency is 99.95% or higher for a standard aerosol of 0.3 μm diameter.

5.7 Calibration. The process of comparing a standard or instrument with one of greater accuracy (small uncertainty) to obtain quantitative estimates of the actual values of the standard being calibrated, the deviation of the actual value from a nominal value, or the difference between the value indicated by an instrument and the actual value.

5.8 Differential Pressure Meter. Any flow measuring device that operates by restricting air flow and measuring the pressure drop across the restriction.

5.9 Emissions. The total of substances discharged into the air from a stack, vent, or other discrete source.

5.10 Flowmeter. An instrument for measuring the rate of flow of a fluid moving through a pipe or duct system. The instrument is calibrated to give volume or mass rate of flow.

5.11 Impaction. A forcible contact of particles of matter. A term often used synonymously with impingement.

5.12 Impactor. A sampling device that employs the principle of impaction (impingement).

5.13 Impingement. The act of bringing matter forcibly in contact. As used in air sampling, refers to a process for the collection of particulate matter in which the gas being sampled is directed forcibly against a surface.

5.14 Inhalable Particles. Particles with aerodynamic diameters of $< 10 \mu\text{m}$ that are capable of being inhaled into the human lung.

5.15 Interference. An undesired positive or negative output caused by a substance other than the one being measured.

5.16 Mass Flowmeter. Device that measures the mass flow rate of air passing a point, usually using the rate of cooling or heat transfer from a heated probe.

5.17 Matter. The substance of which a physical object is composed.

5.18 Orifice Meter. A flowmeter, employing as the measure of flow rate the difference between the pressures measured on the upstream and downstream sides of the orifice (that is, the pressure differential across the orifice) in the conveying pipe or duct.

5.19 Aerodynamic Diameter (a.d.). The diameter of a unit density sphere having the same terminal settling velocity as the particle in question. Operationally, the size of a particle as measured by an inertial device.

5.20 Particle. A small discrete mass of solid or liquid matter.

5.21 Particulate. Solids or liquids existing in the form of separate particles.

5.22 Precision. The degree of mutual agreement between individual measurements, namely repeatability and reproducibility.

5.23 Pressure Gage. The difference in pressure existing within a system and that of the atmosphere. Zero gage pressure is equal to atmospheric pressure.

5.24 Rotameter. A device, based on the principle of Stoke's law, for measuring rate of fluid flow. It consists of a tapered vertical tube having a circular cross section, and containing a float that is free to move in a vertical path to a height dependent upon the rate of fluid flow upward through the tube.

5.25 Sampling. A process consisting of the withdrawal or isolation of a fractional part of a whole. In air or gas analysis, the separation of a portion of an ambient atmosphere with or without the simultaneous isolation of selected components.

5.26 Standard. A concept that has been established by authority, custom, or agreement to serve as a model or rule in the measurement of quantity of the establishment of a practice or procedure.

5.27 Traceability to NIST. Documented procedure by which a standard is related to a more reliable standard verified by the National Institute of Standards Technology (NIST).

5.28 Uncertainty. An allowance assigned to a measured value to take into account two major components of error: The systematic error and the random error attributed to the imprecision of the measurement process.

6. Apparatus Description

6.1 General Description

6.1.1 The essential features of a typical non size-specific TSP high-volume sampler are shown in Figure 1. The high volume sampler is a compact unit consisting of a protective housing; an electric motor driven; a high-speed, high-volume blower; a filter holder capable of supporting a 203 x 254-mm (8 in. by 10 in.) filter; and a flow-controller for controlling the air-flow rate through the instrument at 40-60 ft³/min.

6.1.2 In operation, this traditional TSP sampler draws ambient air into the sampler through the air inlet gap between the cover and the sampler housing walls (see Figure 2). The air inlet is uniform on all sides of the sampler to provide an effective particle capture air velocity between 20-35 cm/sec. at the recommended flow rate between 40-60 ft³/min. The gable roof design of the sampler allows the sampled air to be evenly distributed over the surface of a downstream filter, where TSP is collected.

6.1.3 For PM₁₀ measurement, the traditional gable roof of the TSP sampler is replaced with an impactor design size-select inlet, as illustrated in Figure 3. For the impactation design, an air sample enters a symmetrical (therefore wind-direction insensitive) hood and is deflected upward into a buffer chamber. The buffer chamber is evacuated at a rate of 68 m³/h (40 cfm) through multiple circular nozzles. Particles are accelerated as they pass through the nozzles to an impactation chamber (see Figure 4). Because of their momentum, particles having diameters larger than the inlet's 10- μ m cut design impact the surface of the

impaction chamber. Smaller particles rise through the impaction chamber at speeds slow enough to minimize reentrainment of the impacted particles and then pass through multiple vent tubes to the high-volume sampler's filter where they are collected.

6.1.4 The second size-select design for PM₁₀ measurement is the cyclone inlet, as illustrated in Figure 5. The omnidirectional cyclone used for fractionation in this inlet allows particles to enter from all angles of approach. An angular velocity component is imparted to the sample air stream and the particles contained in it by a series of evenly spaced vanes. Larger particle removal occurs in an inner collection tube. This tube incorporates a "perfect absorber," which is an oil-coated surface to eliminate particle bounce and reentrainment. The sample flow (with the unremoved smaller particles) then enters an intermediate tube, where the trajectory is altered to an upward direction. An additional turn is then made to alter the flow to a downward trajectory to allow the remaining particles (i.e., PM₁₀ fraction) to deposit on a filter for subsequent analysis. As with the impaction inlet, control of air velocities in the cyclonic inlet is critical to maintain the correct particle size cutpoint. Maintaining the correct design volumetric flow rate through the inlet is important. This design flow rate is specified by the manufacturer in the instruction manual. For example, a popular cyclonic impaction inlet has a design flow rate of 1.13 m³/min.

6.2 Filter Medium

6.2.1 Selecting a filtration substrate for time-integrated SPM monitoring must be made with some knowledge of the expected characteristics and a pre-determined analytical protocol. For any given standard test method, the appropriate medium will normally be specified.

6.2.2 Of the various types of filters listed in Table 1 of Chapter IO-2 Overview, four general types of filter material have been used to capture SPM. They include cellulose fiber, quartz/glass fiber, mixed fiber and membrane filter types. Selecting a filter depends upon variables such as background metal content, artifact formation, and affinity for moisture. The basic characteristics of the types of filter material used in air monitoring are outlined in Table 1, while useful filter properties are identified in Table 2. Several characteristics are important in selection of filter media. They are:

- **Particle Sampling Efficiency.** Filters should remove more than 99% of SPM from the air drawn through them, regardless of particle size or flow rate.
- **Mechanical Stability.** Filters should be strong enough to minimize leaks and wear during handling.
- **Chemical Stability.** Filters should not chemically react with the trapped SPM.
- **Temperature Stability.** Filters should retain their porosity and structure during sampling.
- **Blank Correction.** Filters should not contain high concentrations of target compound analytes.

6.2.3 Quartz fiber filters are the most commonly used filters for SPM sampling for determining mass loading. Typical characteristics of quartz fiber filters are (1) a fiber content of high purity quartz, (2) a binder of below 5% (zero for binderless types), (3) a thickness of approximately 0.5 mm, (4) a surface with no pinholes, and (5) an allowance of no more than 0.05% of smoke particles to pass through the filter at a pressure of 100 mm of water with a flow rate of 8.53 m/min (28 ft/min), as determined by a DOP smoke test (see ASTM Method D2986).

6.2.4 Quartz fiber filters are made from finely spun glass fiber by combining the fiber with an organic binder and compressing this material in a paper machine. These filters are increasingly used in air sampling. These filters have the ability to withstand high temperatures (up to 540EC). They are further typified by high-collection efficiency. In some cases, the organic binder may interfere with subsequent analysis, so the filter is flash-fired to remove the binder material. This action causes some loss in tensile strength and usually requires that a backing material be used during sampling. The quartz filters are nonhygroscopic, thus allowing them to be used in areas where humidity is high. Because they are glass, they are the filter choice for most corrosive atmospheres. All the filters in this category are fragile and must be handled with care. Quartz fiber filters, because of the high silicate content, are extremely difficult to ash by chemicals or heat.

Therefore, extraction procedures are performed on these filters to remove the sample for subsequent chemical analysis. For this reason, flash-fired quartz filters are the major atmospheric sampling filters.

6.3 Flow Control System

The high-volume sampler employs two basic types of flow control systems. One is a mass-flow-control (MFC) system; the other is a volumetric-flow-control (VFC) system. Because the calibration and standard operating procedures differ considerably between these two types of flow-control systems, this method presents procedures that are control-system-specific. PM₁₀ inlets can be used with either the MFC and VFC systems.

6.3.1 Mass-flow-control (MFC) system. The flow rate in a MFC system is actively sensed and controlled at a predetermined set point. Air is pulled through the filter into the intake of a blower and subsequently exits the sampler through an exit orifice, which facilitates measurement of the flow with a manometer or pressure recorder. The flow rate is controlled by an electronic mass-flow controller, which uses a flow sensor installed below the filter holder to monitor the mass flow rate and related electronic circuitry to control the speed of the motor accordingly to provide a constant sampling rate. The controlled flow rate can be changed by an adjustment knob on the flow controller.

6.3.2 Volumetric-flow-control (VFC) system. A VFC system maintains a constant volumetric flow rate through the inlet, rather than a constant mass flow rate as in the MFC system. In a popular commercial VFC system, a choked-flow venturi is operated such that the air attains sonic velocity in the throat of the device. In this "choked" mode, the flow rate is unaffected by downstream conditions, such as motor speed or exit pressure and is a predictable function of upstream conditions, such as the stagnation pressure ratio and temperature. Thus, the volumetric flow is controlled without any moving parts or electronic components. In this type of flow control system, no means is provided for adjusting the controlled flow rate. The controlled flow rate is set by the manufacturer through engineering design of the venturi.

7. Calibration

7.1 Introduction

[Note: All sampling equipment must be properly calibrated. Calibration is the relationship between an instrumental output and the input of a known reference standard. The objective of this section is to provide technically sound flow-rate calibration procedures for the MFC and VFC HV samplers.]

[Note: Consistency of temperature and barometric pressure is required. All temperatures should be expressed in kelvin ($K = EC + 273$). All barometric pressures should be expressed in mm Hg. Avoid calibrating an HV sampler using one set of units and then performing sample calculations using another set.]

7.1.1 HV sampler inlet. Two types of size-selective inlets available are impaction and cyclonic for monitoring inhalable particles ($< 10 \mu\text{m}$). The particle size discrimination characteristics of both the impaction and cyclonic type inlets depend critically on maintaining certain air velocities within the inlet; a change in velocity will result in a change in the nominal particle size collected. For this reason, the flow rate through the inlet must be maintained at a constant value that is as close as possible to the inlet's design flow rate. The design flow rate for a given sampler is specified in the sampler's instruction manual. The manual may also provide tolerance limits (or upper and lower limits) within which the sampler flow must be maintained. If the tolerance is not specified by the manufacturer, it should be assumed to be $\pm 10\%$.

7.1.1.1 The symmetrical design of the impaction inlet (see Figure 4) ensures wind-direction insensitivity. Ambient air that is drawn into the inlet is evacuated from the buffer chamber through nine acceleration nozzles into the first impaction chamber, where initial particle separation occurs. The air is then accelerated through an additional 16 jets into a second impaction chamber. The acceleration jets have critical diameters calculated by the manufacturer to provide the necessary changes in velocity to effect correct particle size fractionation within the impaction chambers. The air flow finally exits the inlet through nine vent tubes onto a sample filter. Because air velocities are critical to maintain the correct particle size cutpoint within the inlet, maintaining the correct design flow rate through the inlet is important. This design flow rate is specified by the manufacturer in the instruction manual. For example, the design flow rate for one popular impaction inlet is 1.13 m³/min.

7.1.1.2 The omnidirectional cyclone inlet (see Figure 5) used for fractionation allows particles to enter from all angles of approach. A angular velocity component is imparted to the sample air stream and the particles contained in it by a series of evenly spaced vanes. Larger particle removal occurs in an inner collection tube. This tube incorporates a "perfect absorber," an oil-coated surface to eliminate particle bounce and reentrainment. The sample flow (with the unremoved smaller particles) then enters an intermediate tube, where the trajectory is altered to an upward direction. An additional turn is then made to alter the flow to a downward trajectory to allow the remaining particles (i.e., PM₁₀ fraction) ultimately to deposit on a filter for subsequent analysis. As with the impaction inlet, control of air velocities in the cyclonic inlet is critical to maintain the correct particle size cutpoint. Maintaining the correct design volumetric flow rate through the inlet is important. This design flow rate is specified by the manufacturer in the instruction manual. For example, as in the case of the impaction inlet, a popular cyclonic inlet also has a design flow rate of 1.13 m³/min.

7.1.2 Total suspended particulate (TSP). As illustrated in Figure 2, particles of less than 100 µm are collected at a flow rate of 1.13-1.70 m³/min (40-60 ft³/min) using the conventional high-volume sampler, without size selection.

7.2 Summary of Calibration Procedures

[Note: During calibration, a closure plate perforated with a number of circular orifices is connected to the inlet of the sampler. The pressure drop across this orifice plate provides a measure of instrument air flow rate at any time. This pressure drop may be indicated by a rotameter, manometer, or other pressure-responsive device traceable to an NIST certified standard.]

7.2.1 A simple and sufficiently accurate method of calibrating is to compare the sampler meter with an orifice meter (working standard) that has been calibrated against a primary or master standard such as a Roots meter.

7.2.2 The preferable primary standard is a Roots meter of sufficient capacity to allow an accurate time-volume reading, which would be at least 30 s.

7.2.3 A positive displacement pump or blower may be used as a master flow-rate standard. In this case, the delivery rate of the master standard must be known accurately and the equipment must be in sound mechanical condition (no bypass leakage).

7.3 Certification of an Orifice Transfer Standard

[Note: The following certification procedure is applicable to an orifice transfer standard such as those that have been used previously in the calibration of both the traditional HV sampler and the PM₁₀ samplers. Two common types of orifice devices are available: one equipped with a set of fixed resistance plates (e.g., a reference flow [Ref] device or a top-hat orifice) and one with an externally variable resistance valve. The

series of plates normally provided by the orifice manufacturer include an 18-, 13-, 10-, 7-, and 5-hole plate. Unfortunately, the 5-hole plate provides too low a flow rate to be useful for HV calibration, and other plates may produce flow rates substantially outside the design flow-rate range of the commercially available HV inlets. One may opt to fabricate or procure a different series of resistance ranges or use the variable-resistance type orifice device.]

7.3.1 Orifice Calibration Procedure.

7.3.1.1 Assemble the following equipment (see Figure 6):

- Orifice transfer standard (i.e., top-hat orifice, variable orifice, or ReF device) to be calibrated.
- Water or oil manometer with a 0-400 mm (0-16") range and minimum scale divisions of 1 mm (0.1"). This manometer should be permanently associated with the orifice transfer standard.
- Variable voltage transformer, a set of resistance plates, or available flow orifice (see Figure 7).
- Calibrated positive displacement, standard volume meter (such as a Roots Meter®) traceable to National Institute of Standards and Technology (NIST).

[Note: As they are sold, standard volume meters may not be traceable to NIST. Traceability can be established directly through NIST or indirectly through the meter manufacturer's repair department. Periodic recertification is not normally required under clean service conditions unless the meter has been damaged and must be repaired. In general, damage will be indicated by a substantial (e.g., 50%) increase in the pressure drop across the meter. The meter's traceability certificate should contain a graph of the pressure drop as a function of flow rate.]

- High-volume air mover (e.g., a blower motor from a HV sampler).
- Accurate stopwatch.
- Mercury manometer, with a 0-200 mm (0-8") range and minimum scale divisions of 1 mm (0.1").
- Thermometer, capable of accurately measuring temperatures over the range of 0-50EC (273-323 K) to the nearest ± 1 EC and referenced to an NIST or ASTM thermometer within ± 2 EC at least annually.
- Barometer, capable of accurately measuring ambient barometric pressure over the range of 500-800 mm Hg (66-106 kPa) to the nearest mm Hg and reference within ± 5 mm Hg of a barometer of known accuracy at least annually.
- Orifice transfer standard certification worksheet (see Figure 8).

7.3.1.2 Record on the orifice transfer standard certification worksheet the standard volume meter's serial number; orifice transfer standard's type, model, and serial number; the person performing the certification; and the date.

7.3.1.3 Observe the barometric pressure and record it as Pa. Read the ambient temperature in the vicinity of the standard volume meter and record it as T_a ($K = EC + 273$).

7.3.1.4 Connect the orifice transfer standard to the inlet of the standard volume meter. Connect the mercury manometer to measure the pressure at the inlet of the standard volume meter. Connect the orifice (water or oil) manometer to the pressure tap on the orifice transfer standard. Connect a high-volume air mover to the outlet side of the standard volume meter. Make sure that all gaskets are present and are in good condition.

7.3.1.5 Check that the standard volume meter table is level and adjust its legs if necessary.

7.3.1.6 Check for leaks by temporarily clamping both manometer lines (to avoid fluid loss) and blocking the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Start the high-volume air mover and note any change in the standard volume meter's reading. The reading should

remain constant. If the reading changes, locate any leaks by listening for a whistling sound and/or retightening all connections, making sure that all gaskets are properly installed.

[Note: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime; it can raise temperatures to the point of defeating the electrical insulation which could result in fire or electric shock to the user.]

7.3.1.7 After satisfactorily completing the leak check, turn off the high-volume air sampler, unblock the orifice, and unclamp both manometer lines. Zero the water and mercury manometers by sliding their scales so that their zero lines are even with the bottom of the menisci.

7.3.1.8 Turn on the high-volume air sampler. Adjust the variable voltage transformer to achieve an appropriate flow rate (i.e., within the approximate range of 0.9-1.3 m³/min (32-46 ft³/min)). If necessary, use fixed resistance plates or the variable resistance valve to achieve the appropriate flow rate (see Figure 7). The use of fixed resistance plates is discouraged (but not prohibited) because the leak check must be repeated each time that a plate is installed.

7.3.1.9 After setting a flow rate, allow the system to run for at least 1 min to attain a constant motor speed. Observe the standard volume meter dial reading and simultaneously start the stopwatch. Error in reading the meter dial can be minimized by starting and stopping the stopwatch on whole number dial readings (e.g., 4091.00).

7.3.1.10 Record the initial volume that the meter dial indicated when the stopwatch was started. Maintain this constant flow rate until at least 3 m³ of air have passed through the standard volume meter. Record the standard volume meter's inlet pressure manometer reading as) Hg and the orifice manometer reading as) H₂O. If) H₂O changes significantly during the run, abort the run and start again.

7.3.1.11 When at least 3 m³ of air have passed through the system, note the standard volume meter reading and simultaneously stop the stopwatch. Record the final volume that the meter dial was indicating when the stopwatch was stopped. Record the elapsed time (Time) indicated on the stopwatch.

7.3.1.12 Calculate the volume measured by the standard volume meter (Vol.) using the following equation:

$$\text{) Vol.} = \text{Final Volume} - \text{Initial Volume}$$

7.3.1.13 Correct this volume to ambient atmosphere pressure.

$$V_a = \text{) Vol.} (\text{Pa} - \text{) Hg}) / \text{Pa}$$

where:

- V_a = actual volume at ambient barometric pressure, m³.
-) Vol. = actual volume measured by the standard volume meter, m³.
- Pa = ambient barometric pressure during calibration, mm Hg.
-) Hg = differential pressure at inlet to volume meter, mm Hg.

7.3.1.14 Calculate the actual volumetric flow rate (m³/min).

$$Q_a = V_a / \text{Time}$$

where:

- Qa = actual volumetric flow rate through the orifice, m³/min.
-) time = elapsed time, min.

7.3.1.15 Repeat Sections 7.3.1.8 through 7.3.1.14 for at least four additional flow rates within the approximate range of 0.9-1.3 m³/min (32-46 ft³/min). At least five evenly distributed different flow rates are required, and at least three flow rates must be in the specified inlet flow-rate interval (1.02-1.24 m³/min [36-44 ft³/min]). Better calibration precision may be obtained by running additional flow rates or repeating the flow rates.

7.3.1.16 For each flow, compute $[(\text{H}_2\text{O})(\text{Ta}/\text{Pa})]^{1/2}$, and plot these values against the corresponding values of Qa. Draw the orifice transfer standard's certification curve. For the model $[(\text{H}_2\text{O})(\text{Ta}/\text{Pa})]^{1/2} = m(Qa) + b$, calculate the linear least squares regression's slope (m), intercept (b), and correlation coefficient (r) of the certification relationship. Plot the regression line on the same graph as the calibration data, as illustrated in Figure 9. A certification graph should be readable to 0.02 m³/min.

7.3.1.17 If any calibration point does not fall within $\pm 2\%$ of the line, rerun the point, recalculate, and replot.

7.3.1.18 For subsequent use of the orifice transfer standard, calculate Qa from the calibration relationship as:

$$Qa(\text{orifice}) = \{[(\text{H}_2\text{O})(\text{Ta}/\text{Pa})]^{1/2} - b\} \{1/m\}$$

where:

- Qa(orifice) = actual volumetric flow rate as indicated by the orifice transfer standard, m³/min
-) H₂O = pressure drop across the orifice, mm H₂O.
- Ta = ambient temperature during use, K (K = EC + 273).
- b = intercept of the orifice calibration relationship.
- m = slope of the orifice calibration relationship.

7.3.2 Orifice Transfer Standard Calibration Frequency. Upon receipt and at 1-yr intervals, the calibration of the orifice transfer standard should be certified with a standard volume meter (such as a Roots Meter®) traceable to NIST. An orifice transfer standard should be visually inspected for signs of damage before each use and should be recalibrated if the inspection reveals any nicks or dents.

7.4 Procedure for a Mass-Flow-Controlled (MFC) High Volume Sampler

The MFC sampler calibration procedure presented in this section relates known flow rates to the pressure in the exit orifice plenum. The known flow rates are determined by an orifice transfer standard that has been certified according to the procedure presented in Section 7.3.1. The exit orifice plenum is the area within the motor housing (below the motor unit) that contains the air flow just before it is exhausted to the atmosphere through the exit orifice. This exit orifice plenum pressure should be measured with a 25-cm (10") water or oil manometer. Also, each sampler should have its own dedicated manometer, which can be conveniently mounted to the side of the sampler housing. Other types of pressure measurement devices may be used provided they have comparable accuracy. The 4" continuous pressure (flow) recorders of the type often supplied with high volume PM₁₀ samplers are generally not sufficiently accurate and are not recommended for quantitative sampler pressure or flow measurements. These flow recorders should be used only for nonquantitative determination that the flow was approximately constant and uninterrupted over the sample period. The flow recorder may be connected in parallel with the manometer or other pressure

measuring device, using a tee or "y" tubing connection. For this MFC calibration procedure, the following conditions are assumed:

- The high volume PM₁₀ sampler is equipped with a mass flow controller to control its sample flow rate.
- The sampler flow rate is measured by measuring the exit orifice plenum pressure, using a water or oil manometer [or, if necessary, a continuous-flow recording device using square-root-scale chart paper].
- The transfer standard for the flow-rate calibration is an orifice device equipped with either a series of resistance plates or an integral variable-resistance valve. The pressure drop across the orifice is measured by an associated water or oil manometer.

[Note: Because flow recorders are still widely used for quantitative flow measurements, the calibration procedure includes specific instructions for quantitatively calibrating a flow recorder. These flow recorder instructions are enclosed in brackets [] and should be used only when a manometer or other pressure measurement device cannot be used.]

7.4.1 Calibration Equipment.

7.4.1.1 Orifice transfer standard with calibration traceable to NIST (see Section 7.3).

7.4.1.2 An associated water or oil manometer, with a 0-400 mm (0-16") range and an minimum scale division of 2 mm (0.1")

[Note: Digital manometers may also be used in place of water or oil manometers, especially in cold/frigate climates. Ensure the battery in the manometer is new before use.]

7.4.1.3 A water or oil manometer, with a 0-400 mm (0-16") range and a minimum scale division of 2 mm (0.1") for measurement of the sampler exit orifice plenum pressure. This manometer should be associated with the sampler.

[Note: Manometers used for field calibration may be subject to damage or malfunction and should thus be checked frequently.]

7.4.1.4 Thermometer, capable of accurately measuring temperature over the range of 0-50EC (273-323 K) to the nearest ± 1 EC and referenced to an NIST or ASTM thermometer within ± 2 EC at least annually.

7.4.1.5 A portable aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient barometric pressure over the range of 500-800 mm Hg (66-106 kPa) to the nearest mm Hg and referenced within ± 5 mm Hg of a barometer of known accuracy at least annually.

7.4.1.6 Miscellaneous handtools, calibration data sheets or station log book, and 51 mm (2") duct tape.

7.4.2 Multipoint Flow-Rate Calibration. The procedure presented here is basic and generic, given the assumptions listed in Section 7.4. More detailed calibration procedures, variations, or alternative procedures may be presented in the manufacturer's instruction manual. The manual should be reviewed carefully and the various calibration variations or alternative procedures should be evaluated. In-house equipment and personnel, procedural simplicity and uniformity, and subsequent data applications should be considered in establishing the specific, detailed calibration procedure to be implemented.

[Note: Do not attempt to calibrate the MFC sampler under windy conditions. Short-term wind velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The calibration will be less precise because of pressure variations.]

7.4.2.1 Set up the calibration system as recommended by the manufacturer. A typical MFC PM₁₀ sampler calibration configuration is illustrated in Figure 10. MFC samplers are calibrated without a filter or filter cassette installed.

7.4.2.2 Disconnect the motor from the flow controller and plug it directly into a stable line voltage source (i.e., the sampler's on-off timer, if so equipped, or other source of the line voltage).

7.4.2.3 Install the orifice transfer standard and its adapter faceplate on the sampler. Check all gaskets and replace any questionable ones.

[Note: Tighten the faceplate nuts evenly on alternate corners to properly align and seat the gaskets. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.]

7.4.2.4 Select the first calibration flow rate and install the appropriate resistance plate or adjust the variable orifice valve. At least four flow rates are required to define the calibration relationship. For resistance plate orifices, make sure that the orifice and resistance plate gaskets are in place and the orifice is not cross-threaded on the faceplate.

7.4.2.5 To leak check, block the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Seal the pressure port with a rubber cap or similar device. Turn on the sampler. Gently rock the orifice transfer standard and listen for a whistling sound that would indicate a leak in the system. A leak-free system will not produce an upscale response in the sampler's exit orifice manometer or flow recorder. Leaks are usually caused either by a damaged or missing gasket between the orifice transfer standard and the faceplate or by cross-threading of the orifice transfer standard on the faceplate. All leaks must be eliminated before proceeding with the calibration. When the system is determined to be leak-free, turn off the sampler and unblock the orifice.

[Note: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime and can raise temperatures to the point of defeating the electrical insulation, which could result in fire or electric shock to the user.]

7.4.2.6 Inspect the connecting tubing of both manometers for crimps or cracks. Open the manometer valves (if present) and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometers' sliding scales so that their zero lines are at the bottom of the menisci. Connect the orifice transfer standard manometer to the orifice transfer standard. Connect the sampler's exit orifice manometer [and the continuous-flow recorder, if used] to the exit orifice plenum port. Ensure that one side of each manometer is open to atmospheric pressure. Make sure that the tubing fits snugly on the pressure ports and on the manometer.

7.4.2.7 If a continuous flow recorder is to be used quantitatively in lieu of a manometer, record the site location, sampler S/N, date, and the operator's initials on the blank side of a clean recorder chart. Make sure the chart has a square-root scale. Open the front door of the sampler and install the clean recorder chart.

7.4.2.8 Read and record the following parameters on the HV data sheet. An example calibration data sheet for the MFC sampler is illustrated in Figure 11.

- Date, location, and operator's signature.
- Sampler S/N and model.
- Ambient Pa, mm Hg.
- Ambient temperature (T_a), K ($K = EC + 273$).
- Orifice S/N and calibration relationship.

[Note: Consistency of temperature and barometric pressure units is required. All temperatures should be expressed in kelvin ($K = EC + 273$). Also, all barometric pressures should be expressed in mm Hg. Avoid calibrating a sampler using one set of units and then performing sampler calculations using another set.]

[Note: Ideally, the temperature of the air in the exit orifice plenum should be measured because it will be somewhat higher than ambient temperature. However, an adequate approximation of this temperature may be obtained by adding 30 K to the ambient temperature. This addition is incorporated in the calculations given in Section 7.4.3.]

7.4.2.9 Turn on the sampler and allow it to warm up to operating temperature (3-5 min). Then read and record the orifice transfer standard's manometer deflection, ΔH_2O (in. H₂O), and the corresponding sampler's manometer deflection, ΔP_{ex} [or flow recorder chart reading, I].

[Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2" of clearance at the bottom for air flow and for the manometer tubing.]

7.4.2.10 Install the other resistance plates or adjust the variable orifice value to obtain each of the other calibration flow rates and repeat Section 7.4.2.9 for each. At least four calibration flow rates are required.

7.4.2.11 Plot the calibration data on a sheet of graph paper as specified in Section 7.4.3.4.

[Note: The data should be plotted in the field as the calibration is occurring, rather than afterwards back at the laboratory.]

Repeat Section 7.4.2.9 for any data that are questionable on the plot.

[Note: Running additional calibration points at differing flow rates or repeating the calibration points at the same flow rates is encouraged to improve the precision of the calibration.]

7.4.2.12 Turn off the sampler and remove the orifice transfer standard.

7.4.2.13 Reconnect the sampler motor to the flow controller.

7.4.2.14 Perform the calibration calculations presented in the following section. The data generated will be used to set the mass flow controller (see Section 7.4.4) to a value that will result in optimal volumetric flow based on the seasonal average temperature and barometric pressure at the monitoring site.

7.4.3 Calibration Calculations. Gather all calibration data, including the orifice calibration information and the sampler calibration data sheet (and, if used, the flow recorder chart, which should graphically display the various calibration flow rates).

[Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that have already been obtained.]

7.4.3.1 Verify that the orifice transfer standard calibration relationship is current and traceable to an acceptable primary standard.

7.4.3.2 Calculate and record Q_a for each calibration point from the orifice calibration information using the following equation.

$$Q_a(\text{orifice}) = \{ \} H_2O(T_a/P_a)^{1/2} - b \} \{1/m\} \text{ where:}$$

- Q_a(orifice) = actual volumetric flow rate as indicated by the transfer standard orifice, m³/min
-) H₂O = pressure drop across the orifice, in. H₂O.
- T_a = ambient temperature during use, K (K = EC + 273).
- P_a = ambient barometric pressure during use, mm Hg.
- b = intercept of the orifice calibration relationship.
- m = slope of the orifice calibration relationship.

7.4.3.3 Calculate and record the quantity for each calibration point as:

$$) P_{ext} = \{ \} P_{ex}(T_a + 30)/P_a)^{1/2}$$

where:

-) P_{ext} = transformed manometer reading.
-) P_{ex} = sampler manometer reading, in. H₂O
- T_a = ambient temperature, K (K = EC + 273).
- P_a = ambient barometric pressure, mm Hg.

[If a continuous-flow recorder is used quantitatively, calculate and record the quantity [It] as follows:

$$[It] = I(T_a + 30)/P_a)^{1/2}$$

where:

- [It] = transformed flow recorder chart reading.
- I = flow recorder chart reading, arbitrary units on square root scale.

[Note: If recorder charts with linear scales are used, substitute (I)^{1/2} for I in the above equation.]

7.4.3.4 On a sheet of graph paper, plot the calculated Q_a(orifice) flow rates on the x-axis and the transformed sampler manometer response,) P_{ext} [or the transformed flow recorder reading, It], on the y-axis.

Because determining the sampler's average operational flow rate (Q_a) during a sample period depends on the ambient average temperature and pressure, using a graphic plot of the calibration relationship is not recommended for subsequent data reduction. This plot is used only to visually assess the calibration points to see if any should be rerun. Plot the regression line on the same graph paper as the calibration data. For the regression model y = mx + b, let y + 2) P_{ext} and x = Q_a(Orifice) so that the model is given by:

$$) P_{ext} = m[Q_a(\text{orifice})] + b$$

For the flow recorder, the model is:

$$It = m[Q_a(\text{orifice})] + b]$$

Using a programmable calculator or a calculation data form, determine the linear regression slope (m), intercept (b), and correlation coefficient (r) and record them on the data sheet. A five-point calibration should yield a regression equation with a correlation coefficient of r > 0.990, with no point deviating more

than $\pm 0.04 \text{ m}^3/\text{min}$ from the value predicted by the regression equation. Plot the regression line on the same graph paper that has the individual calibration points.

7.4.3.5 For subsequent sample periods, the sampler's average actual operational flow rate, \overline{Q}_a , is calculated from the calibration slope and intercept using the equation.

$$\overline{Q}_a = \{ \overline{P}_{ex} (T_{av} + 30) / P_{av} \}^{1/2} - b \} \{ l/m \}$$

where:

- \overline{Q}_a = the sampler's average actual flow rate, m^3/min .
- \overline{P}_{ex} = average of initial and final sampler manometer readings $(P_{ex_i} + P_{ex_f}) / 2$, mm Hg.
- T_{av} = average ambient temperature for the sample period, K ($K = EC + 273$).
- P_{av} = average ambient pressure for the sampling period, mm Hg.
- b = intercept of the sampler calibration relationship.
- m = slope of the sampler calibration relationship.

[For the flow controller,

$$\overline{Q}_a = \{ \overline{I} (T_{av} + 30) / P_{av} \}^{1/2} - b \} \{ l/m \}$$

where:

- \overline{I} = average flow recorder reading for the sample period.]

[*Note: If recorder charts with linear scales are used, substitute $(I)^{1/2}$ for (I) in the above equation.*]

7.4.4 Mass Flow Controller Adjustment Procedure. The controlled flow rate of an MFC sampler is adjustable and must be set to the proper design flow rate. The constant mass flow maintained by the MFC causes the actual volumetric flow rate through the inlet to fluctuate as the ambient temperature and barometric pressure change at the monitoring site. Normally, the range of these fluctuations is within the allowable tolerance limits for the inlet. However, the flow-rate set point of the mass flow controller must be correctly adjusted so that the deviations are "centered" with respect to the seasonal average temperature and barometric pressure at the site, not the temperature and pressure prevailing at the time of setting. The correct seasonal volumetric setpoint flow rate (SFR) at T_a and P_a has had the same mass flow rate as the inlet design volumetric flow rate at T_s and P_s .

[*Note: The correct SFR may differ from day to day and may be somewhat higher or lower than the inlet design flow rate on any particular day.*]

7.4.4.1 Determine the seasonal average temperature (T_s) and seasonal average pressure (P_s) at the site and record them on the calibration data sheet. (Determination of the number of "seasons," i.e., the number of different seasonal average temperatures needed for the year, is left to the discretion of the operator.)

7.4.4.2 Calculate SFR and record on the calibration data sheet:

$$\text{SFR} = (1.13) (P_s/P_a)(T_a/T_s)$$

where:

- SFR = set-point actual volumetric flow rate for adjustment of the mass flow controller, based on seasonal average temperature and average pressure at site, m³/min.
1.13 = inlet design flow rate (as specified by the manufacturer), m³/min.
P_s, P_a = seasonal average and current ambient barometric pressure at the site, respectively, mm Hg.
T_s, T_a = seasonal average and current ambient temperature, respectively, K (K = EC + 273).

7.4.4.3 Calculate and record on the sampler's calibration data sheet the sampler set-point manometer reading [or flow recorder reading] that corresponds to the SFR calculated in Section 7.4.4.2.

$$\text{SSP} = [P_a/(T_a + 30)][m(\text{SFR}) + b]^2$$

where:

- SSP = sampler set-point manometer reading, in H₂O.
P_a = ambient barometric pressure, mm Hg.
T_a = ambient temperature, K (K = EC + 273).
m = slope of the sampler's calibration relationship.
SFR = set-point flow rate from 7.4.4.2, m³/min.
b = intercept of the sampler's calibration relationship.
[For the flow recorder,

$$\text{SSP} = [m(\text{SFR}) + b] [P_a/(T_a + 30)]^{1/2}$$

7.4.4.4 Visually check to make sure the motor is connected to the mass flow controller and the manometer is properly connected.

7.4.4.5 Install a clean filter (in a filter cassette) in the sampler according to the manufacturer's instructions. [If the continuous flow recorder is used quantitatively, install a clean chart and verify that the recorder is zeroed (i.e., the pen rests on the innermost circle of the chart).]

7.4.4.6 Turn on the sampler and allow it to warm up to operating temperature (3-5 min).

7.4.4.7 Following the manufacturer's instructions, adjust the mass flow controller until the manometer reading [or flow recorder response] indicates the sampler set point (SSP) as calculated in Section 7.4.4.3.

7.4.4.8 Verify that the flow controller will maintain this flow rate for at least 10 min. Turn off the sampler.

7.4.4.9 The sampler can now be prepared for the next sample run day.

7.5 Procedure for a Volumetric-Flow-Controlled (VFC) Sampler

The VFC sampler calibration procedure presented in this section relates known flow rates (Q_a, as determined by an orifice transfer standard) to the ratio of the stagnation pressure to the ambient barometric pressure (P_I/P_a). The stagnation pressure (P_I) is the air pressure inside the sampler in the area just under the filter. VFC samplers have a stagnation pressure tap or port through which the stagnation pressure can be measured. A VFC sampler may also have an exit orifice below the motor similar to those in MFC samplers. In this case, the sampler flow rate can be measured and calibrated using the exit orifice plenum pressure generally described in Section 7.4. However, using the stagnation pressure generally provides a more accurate

indication of sampler flow rate. Additionally, a continuous-flow recorder may be connected to the exit orifice pressure tap for nonquantitative determination that the flow rate was constant and uninterrupted over the sample period.

The stagnation pressure should be measured with a 0-1000 mm (0-36") oil, water, or digital manometer. Also, each sampler should have its own dedicated manometer, which can be conveniently mounted to the side of the sampler housing. Other types of pressure measurement instruments may be used provided they have comparable accuracy. However, the 4" continuous pressure (i.e., flow) recorders often supplied with HV samplers are generally not sufficiently accurate and are **not recommended** for quantitative sampler pressure or flow rate measurements.

The VFC sampler's flow control system is a choke-flow venturi. This system must be precisely sized for a given average annual temperature and pressure because no means is provided for the user to adjust the operational flow rate. Therefore, the purchasing agency should notify the manufacturer of the **operational** location of the sampler; differences in temperature and pressure between the shipping address and the monitoring site may result in an incorrect operational flow rate. As with the MFC sampler, both the ambient temperature and barometric pressure readings must be determined or estimated during the sampling period for the subsequent calculation of total sampler volume in standard volume units.

For this VFC calibration procedure, the following conditions are assumed:

- The VFC sampler uses a choked-flow venturi to control the actual volumetric flow rate.
- The sampler flow rate is measured by measuring the stagnation pressure ratio, and the sampler is not equipped with a continuous flow recorder.
- The sampler inlet is designed to operate at a constant actual volumetric flow rate of 1.13 m³/min.
- The transfer standard for the flow-rate calibration is an orifice device equipped with either a series of resistance plates or an integral variable-resistance valve. The pressure drop across the orifice is measured by an associated water or oil manometer.
- The sampler will be calibrated in actual volumetric flow-rate units (Q_a), and the orifice transfer standard is also calibrated in Q_a, as specified in Section 7.3.

7.5.1 Calibration Equipment.

7.5.1.1 Orifice transfer standard with proper calibration traceable to NIST (see Section 7.3).

7.5.1.2 An associated water, oil, or digital manometer, with a 0-400 mm (0-16") range and minimum scale divisions of 2 mm (0.1").

7.5.1.3 An oil, water, or digital manometer, with a 0-1000 mm (0-36") range and minimum scale divisions of 2 mm (0.1") or other pressure measurement device for measurement of the sampler stagnation pressure. Ideally, this manometer (or other pressure instrument) should be associated with the sampler.

[Note: Manometers used for field calibration may be subject to damage or malfunction and should thus be checked frequently.]

7.5.1.4 Thermometer, capable of accurately measuring temperature over the range of 0-50EC (273-323 K) to the nearest ± 1EC and referenced to an NIST or ASTM thermometer within ± 2EC at least annually.

7.5.1.5 A portable, aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient barometric pressure over the range of 500-800 mm Hg to the nearest mm Hg and referenced within ± 5 mm Hg to a barometer of known accuracy at least annually.

7.5.1.6 Calibration data sheets or the station log book and 51 mm (2")-wide duct tape.

7.5.1.7 A clean filter.

7.5.2 Multipoint Flow-Rate Calibration Procedure - VFC Sampler. The procedure presented here is basic and intended to be generic, given the assumptions listed in Section 7.5. More detailed calibration procedures, variations, or alternative procedures may be presented in the manufacturer's instruction manual. The manual should be reviewed carefully and that the various calibration variations or alternative procedures be evaluated. In-house equipment and personnel, procedural simplicity and uniformity, and subsequent data applications should be considered in establishing the specific, detailed calibration procedure to be implemented.

[Note: The calibration of some VFC samplers may be affected by changes in line voltage, particularly if the line voltage is below normal (normal is about 115 V). For this reason, VFC samplers should always be calibrated at the monitoring site. Further, if the line voltage at the site is low and likely to fluctuate significantly, a line voltage booster or regulator may be advisable. Also, be sure that replacement blower motors are of the correct type.]

[Note: Do not attempt to calibrate the VFC sampler under windy conditions. Short-term velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The calibration will be less precise because of the pressure variations.]

7.5.2.1 Set up the calibration system as recommended by the manufacturer. A typical VFC sampler calibration configuration is illustrated in Figure 12. The VFC sampler manufacturer may specify that the sampler be calibrated with a filter installed, which generally precludes calibration flow rates higher than normal operating flow rate. Additional calibration flow rates obtained without a filter may be appropriate, as discussed in Section 7.5.2.8.

7.5.2.2 Install the orifice transfer standard and its adapter faceplate on the sampler. First inspect all gaskets and seals and replace any doubtful ones.

[Note: Tighten the faceplate nuts evenly on alternate corners to properly align and uniformly seat the gaskets. The nuts should be hand-tightened only; too much compression can damage the sealing gasket.]

7.5.2.3 Select a calibration flow rate and install the appropriate resistance plate (or no plate) or adjust the variable resistance valve. At least four flow rates are required to define the calibration relationship. At least three flow rates should be within the acceptable flow-rate range (i.e., 1.02-1.24 m³/min) for the sampler inlet. For resistance plate orifices, make sure the orifice and resistance plate gaskets are in place and the orifice is not cross-threaded on the faceplate.

7.5.2.4 Leak check the system by blocking the orifice with a large-diameter rubber stopper, wide duct tape, or other suitable means. Seal the pressure port with a rubber cap or similar device. Turn on the sampler. Gently rock the orifice transfer standard and listen for a whistling sound that would indicate a leak in the system. Leaks are usually caused either by a damaged or missing gasket between the orifice transfer standard and the faceplate or by crossthreading of the orifice transfer standard on the faceplate. All leaks must be eliminated before proceeding with the calibration. When the system is determined to be leak-free, turn off the sampler and unblock the orifice.

[Note: Avoid running the sampler for longer than 30 s at a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the motor's lifetime. It can raise temperatures to the point of defeating the electrical insulation, which could result in fire or electric shock to the user.]

7.5.2.5 Inspect the connecting tubing of the manometers for crimps or cracks. Open the manometer valves (if present) and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometers' sliding scales so that their zero lines are at the bottom of the menisci. Connect the transfer standard manometer to the transfer standard and the sampler stagnation pressure manometer (or other pressure instrument) to the stagnation pressure port. Ensure that one side of each manometer is open to atmospheric pressure. Make sure the tubing fits snugly on the pressure ports and on the manometers.

7.5.2.6 Read and record the following parameters on the VFC Sampler Data Sheet. An example calibration data sheet for the VFC sampler is illustrated in Figure 13.

- Date, location, and operator's signature.
- Sampler S/N and model.
- Ambient barometric pressure (Pa), mm Hg.
- Ambient temperature (Ta), EC and K (K = EC + 273).
- Orifice S/N and calibration relationship.

[Note: Consistency of temperature and barometric pressure units is required. All temperatures should be expressed in kelvin (K = EC + 273). Also, all barometric pressures should be expressed in mm Hg. Avoid calibrating a HV sampler using one set of units and then performing sampler calculations using another set.]

7.5.2.7 Turn on the sampler and allow it to warm to operating temperature (3-5 min). Read and record the orifice transfer standard's manometer reading, H₂O, and the corresponding sampler relative stagnation pressure manometer reading, Pstg, on the data sheet. (Relative stagnation pressure is a negative pressure [i.g., a vacuum] relative to atmospheric pressure as measured by a manometer with one leg open to the atmosphere.) Be sure to convert the manometer reading to mm Hg using the following equation before recording the reading on the calibration data sheet:

$$\text{mm Hg} = 25.4 (\text{in. H}_2\text{O}/13.6)$$

[Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2" of clearance at the bottom of air flow and for the manometer tubing.]

7.5.2.8 Install the other resistance plates or adjust the variable orifice value to obtain each of the other calibration flow rates and repeat Section 7.5.2.7 for each. At least four calibration flow rates are required with at least three in the acceptable flow-rate range. Difficulties may be encountered in obtaining flow rates in the acceptable range. Even with modified resistance plates (or with no plates) installed, it may be impossible to obtain three acceptable flow rates with a filter mounted on the sampler. Lower flow rate calibration points may be used by extrapolation into the acceptable range without a filter installed in the sampler. If additional calibration points are obtained without a filter, they should be examined carefully to make sure they are consistent with the calibration points obtained with a filter (i.e., they fall on a smooth curve through all the calibration points).

7.5.2.9 Plot the calibration data on a sheet of graph paper as specified in Section 7.5.3.5 of the next section. Repeat Section 7.5.2.7 for any data that are questionable on the plot. Running additional calibration points at differing flow rates or repeating the calibration points at the same flow rates is encouraged to improve the precision of the calibration.

[Note: The data should be plotted in the field as the calibration is occurring, rather than afterwards back at the laboratory.]

7.5.2.10 Turn off the sampler and remove the orifice transfer standard.

7.5.2.11 Install a clean filter on the sampler in the normal sampling mode (use a filter cassette if one is normally used). Turn on the sampler and allow it to warm up to operating temperature.

7.5.2.12 Read the relative stagnation pressure as in Section 7.5.2.7 and record it on the data sheet in the row for the operational flow rate.

7.5.2.13 Perform the calibration calculations presented in the following sections.

7.5.3 Calibration Calculations. Gather together all the calibration data, including the orifice transfer standard's calibration information and the sampler calibration data sheet.

[Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that have already been obtained.]

7.5.3.1 Verify that the orifice transfer standard calibration relationship is current and traceable to an acceptable primary standard.

7.5.3.2 Calculate the record $Q_a(\text{orifice})$ for each calibration point from the orifice calibration information and the equation.

$$Q_a(\text{orifice}) = \{ [\text{H}_2\text{O}(T_a/P_a)]^{1/2} - b \} \{ l/m \}$$

where:

$Q_a(\text{orifice})$ = actual volumetric flow rate as indicated by the transfer standard orifice, m^3/min .

H_2O = pressure drop across the orifice, in. H_2O .

T_a = ambient temperature during use, K ($K = \text{EC} + 273$).

P_a = ambient barometric pressure during use, mm Hg.

b = intercept of the orifice transfer standard's calibration relationship.

m = slope of the orifice transfer standard's calibration relationship.

7.5.3.3 Calculate and record the value of the absolute stagnation pressure ratio, $[PI]$, for each calibration point:

$$[PI] = P_a - P_{\text{stg}}$$

where:

$[PI]$ = absolute stagnation pressure, mm Hg.

P_a = ambient barometric pressure, mm Hg.

P_{stg} = relative stagnation pressure, mm Hg.

7.5.3.4 Calculate and record the stagnation pressure ratio:

$$\text{Stagnation pressure ratio} = PI/P_a$$

7.5.3.5 On a sheet of graph paper, plot the calculated orifice transfer standard's flow rates, $Q_a(\text{orifice})$, on the x-axis vs. the corresponding stagnation pressure ratios, PI/P_a , on the y-axis. Draw a smooth curve through the plotted data. If necessary, extrapolate the curve to include the acceptable flow-rate range.

7.5.3.6 If the sampler manufacturer has provided a factory calibration table (i.e., the lookup table) for the sampler, compare $Q_a(\text{orifice})$ for several points on the calibration plot with $Q_a(\text{sampler})$ determined from the factory calibration. Calculate the percentage difference between $Q_a(\text{orifice})$ and $Q_a(\text{sampler})$ using the following equation.

$$\% \text{ Difference} = \frac{Q_a(\text{sampler}) - Q_a(\text{orifice})}{Q_a(\text{orifice})} \cdot 100$$

If the agreement is within a few (i.e., 2 or 4) percent, the factory calibration is validated and may be used for subsequent sample periods. Proceed to Section 7.5.5.

7.5.3.7 If the agreement is not within a few percentage points, recheck the accuracy of the orifice transfer standard and recheck the calibration procedure. Look for leaks, manometer reading errors, incorrect temperature or pressure data, or miscalculations. Also check for abnormally low line voltage at the site (it should be at least 110 V ac), for the correct blower motor, and for the presence of a gasket between the motor and the choked-flow venturi. A factory calibration is not likely to be substantially incorrect, and any discrepancy of more than a few percent is probably due to some problem with the sampler or with the calibration procedure. However, if no errors or problems with the sampler or with the calibration can be found, or if no factory calibration is provided by the manufacturer, proceed as described in Section 7.5.4.

7.5.4 Generation of Calibration Relationship - VFC Sampler.

7.5.4.1 For each calibration point, calculate and record the quantity,

$$[(P/P_a)T_a]^{1/2}$$

where:

P/P_a = stagnation pressure ratio from the equation in Section 7.5.3.

T_a = ambient temperature during sampler calibration, K ($K = EC + 273$).

7.5.4.2 For the general linear regression model, $y = mx + b$, let $y = [(P/P_a)T_a]^{1/2}$ and let $x = Q_a(\text{orifice})$, such that the model is given by:

$$[(P/P_a)T_a]^{1/2} = m[Q_a(\text{orifice})] + b$$

Calculate the linear regression slope (m), intercept (b), and correlation coefficient (r).

[Note: Inspect the plotted calibration curve to determine whether any of the calibration points that are substantially outside of the acceptable flow-rate range need to be eliminated so that they do not result in an inappropriate linear regression line.]

7.5.4.3 For subsequent sample periods, the sampler's average actual operating flow rate, Q_a , is calculated from the calibration slope and intercept using the following equation.

$$\overline{Q_a}(\text{sampler}) = \{[\overline{P/P_a}]T_{av}\}^{1/2} - b \} \{1/m\}$$

where:

$\overline{Q_a}(\text{sampler})$ = the sampler's average actual flow rate, m^3/min .

$\overline{P/P_a}$ = average stagnation pressure ratio for the sampling period.

T_{av} = average ambient temperature for the sampling period, K ($K = EC + 273$).

b = intercept of the sampler calibration relationship.

m = slope of the sampler calibration relationship.

[Note: The average value for P_I should be calculated from stagnation pressure measurements taken before and after the sampling period. P_{av} should be estimated from barometric pressure for the sampling period. See also Section 9.4 for additional information.]

7.5.4.4 If a calibration (Lookup) table is desired, evaluate the above equation for various appropriate values of P_I/P_a and T_a and list the corresponding values of $Q_a(\text{sampler})$ in tabular form.

7.5.5 Single-Point Operational Flowrate Ventilation. This procedure compares the VFC sampler's normal operating flow rate to the design flow rate of the inlet (e.g., 1.13 m³/min).

7.5.5.1 Determine the value of P_I/P_a for the operational flow rate obtained with only the filter cassette installed (see Section 7.5.2.11 and Section 7.5.2.12).

7.5.5.2 Determine the new sampler flow rate, $Q_a(\text{sampler})$ from the lookup table that corresponds to this value of P_I/P_a . Use the manufacturer's calibration table if it has been validated in 7.5.3.6; otherwise, use the equation in Section 7.5.4.3.

7.5.5.3 Compare $Q_a(\text{sampler})$ with the inlet design flow rate (e.g., 1.13 m³/min) using the following equation:

$$\text{Design flow rate\% difference} = \frac{Q_a(\text{sampler}) \& 1.13}{1.13} \times 100$$

This design flow rate percentage difference must be less than the allowable flow rate tolerance (i.e., ± 10 , if not otherwise specified by the manufacturer). However, this value should be well within ± 7 to allow for some variation with ambient temperature. If this value is not within ± 7 , recheck the calibration procedure and data for errors. Check the sampler for leaks, bad motor brushes, missing gaskets, incorrect motor type, or abnormally low line voltage. Because the VFC flow rate is not adjustable, the VFC manufacturer must be consulted to resolve cases of substantially incorrect VFC flow rates.

7.6 Sampler Calibration Frequency

To ensure accurate measurement calibrate HV samplers upon installation and recalibrate as follows:

7.6.1 At least quarterly or annually (see 40 CFR 58, Appendix A for a description of the quality assurance requirements);

7.6.2 After any repairs that might affect sampler calibration (e.g., replacing the motor);

7.6.3 After relocation of the sampler to a different site;

7.6.4 If the results of a field flow-check exceed quality control limits (e.g., greater than $\pm 7\%$ from the sampler's indicated flow rate); or

7.6.5 Whenever a field flow-check or performance audit indicates that the sampler is out (or nearly out) of the acceptable flow-rate range.

[Note: Multipoint flow-rate calibrations should be distinguished from single-point, quality control flow checks (see Section 13). The latter are done more frequently than calibrations and are intended to check if the sampler flow rate, $Q_a(\text{sampler})$, or the calibration relationship has changed significantly since the last calibration.]

8. Filters

8.1 Pre-weighing of Filters

8.1.1 Filters ready for field use have been pre-weighed in the laboratory, under prescribed climate control conditions of temperature and relative humidity, using Inorganic Compendium Method IO-3.1, *Selection, Extraction and Preparation of Filter Material*.

8.1.2 Within Method IO-3.1, the user is provided guidance on proper selection of filter material in order to meet project specific data quality objectives (DQOs), how to visually inspect a new lot of filters for consistency and identification of defects, and initial weighing of the filters so a net concentration of particulate matter can be calculated after sampling.

8.1.3 The user should follow the procedures outlined within Method IO-3.1 as part of meeting the program's standard operating procedures (SOPs) and quality control (QC) requirements.

8.2 Filter Handling

8.2.1 Filter material may be brittle and subject to shearing and breakage. Laboratory and field personnel must be aware of these characteristics and handle sample filters with care.

8.2.2 For convenience, filters can be packed in groups of 50 or less in their original containers or in a box of comparable size. The filters should be separated by a sheet of 8 ½ x 11" tracing paper. Filter inventory can be controlled by stacking the filters in numerical order so that the operator will use the proper filter first. One side of the shipping box can be cut away to allow the operator to remove the filter easily without damaging the corners.

8.2.3 A filter identification number must be assigned to each filter. Because of difficulty in seeing the "up" side (i.e., the side with the slightly rougher texture) of the filter, consistency in labeling these filters will allow the operator easy access to the filter ID number for documentation and cross-referencing laboratory data forms. This consistency will also eliminate confusion in loading the filter cassettes for subsequent sampling. If the filter ID number is embossed by the operating agency, gentle pressure must be used to avoid filter damage, and extreme care must be taken to avoid duplication or missed numbers.

8.2.4 If samples are to be mailed, the field operator should be supplied with reinforced envelopes and manila folders for protection of the exposed filters during their return to the analytical laboratory. These manila folders may be printed to serve as sample data sheets.

8.3 Visual Filter Inspection

All filters must be visually inspected for defects, and defective filters must be rejected if any are found. Batches of filters containing numerous defects should be returned to the supplier.

The following are specific defects to look for:

- **Pinhole** - a small hole appearing as a distinct and obvious bright point of light when examined over a light table or screen, or as a dark spot when viewed over a black surface.
- **Loose material** - any extra loose material or dirt particles on the filter that must be brushed off before the filter is weighed.
- **Discoloration** - any obvious visible discoloration that might be evidence of a contaminant.
- **Filter nonuniformity** - any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity across the face of the filter.
- **Other** - a filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

9. Sampling Procedure

[Note: This section describes routine operation of a monitoring site using an HV sampler and covers an array of topics, ranging from initial site selection to final data documentation. The procedures herein are intended to serve as guidelines for developing a monitoring program that will accurately reflect trends in local or regional air quality. The effectiveness of the monitoring program depends on responsible day-to-day operation of the monitoring site. The operators who conduct sampling activities offer a unique perspective on the sampler's performance, and their awareness and attention to detail will salvage data that may otherwise be lost. Note, however, that "routine" does not mean "unimportant." The site operator provides cohesiveness in a sampling program.]

9.1 Summary

9.1.1 The PM₁₀ sampler can be used in a number of ways. Procedure variations may include the kind of filter medium, the surface area of the filter, prescreening to exclude particles up to a given size, and the manner of placing and exposing the filter during the test. The procedure most commonly used will be described here.

9.1.2 Calibrate the sampler as described in the Section 7. Do not make any change or adjustment on the sampler flow indicator after calibrating. Remove the calibrating orifice. The filters may be packed into a box with sheets of glassine between the filters, or they may be individually packed in self-sealing plastic bags for transportation to the field.

9.1.3 Mount the filter sheet in the filter holder taking care not to lose any of the fiber. Clamp it in place by means provided. Seal into place easier by facing the smooth side into the housing if there is a difference in texture. If the filter holder is separate from the sampler, mount the holder on the intake port, making sure that the coupling gasket is in place and that it is tight.

9.1.4 Place the sampler in the position and location called for in the test, which is with the filter face up, in a horizontal plane, and inside a housing. The dimensions and clearances specified are intended to provide uniformity in sampling practice.

9.1.5 Start the sampler motor and record the time and date. Read the flow-rate indicator and record this reading and the corresponding flow rate as read from the calibration curve. Note also the temperature and barometric pressure. An electric clock should be connected to the same line as the motor so as to detect any loss of test time due to power interruption. A continuous record of the sampling flow rate and sampling time can be obtained by the use of a continuous pressure (or flow rate) recorder.

9.1.6 Allow the sample to run for the specified length of time, which is commonly 24 h, \pm 1 h. During this period several readings of flow rate, temperatures, barometric pressure, and time should be taken if this is feasible. A final set of reading is taken at the end of the test period. If only initial and final readings are made, assume that change of readings is linear over the period of test. Intermediate readings will improve the accuracy of volume measurement.

9.1.7 At the end of the sampling period, record all final readings. Remove the filter from the mount very carefully so as not to lose any of the fiber material or collected particulate matter. Fold the filter in half upon itself with the collected material enclosed within. Place the folded filter in a clean tight envelope and mark it for identification. In some applications it may be desirable to place the used filter in a tight metal container to prevent any loss or damage to the filter.

9.1.8 In the laboratory remove the filter from its container. Tap the container and knock any loose fiber or particulate matter onto the inside surface of the folded filter. Examine the inside surface and, with a pair of tweezers, remove any accidental objects such as insects.

9.2 Siting Requirements

9.2.1 As with any type of air monitoring study in which sample data are used to draw conclusions about a general population, the validity of the conclusions depends on the representativeness of the sample data. Therefore, the primary goal of a monitoring project is to select a site or sites where the collected particulate mass is representative of the monitored area.

9.2.2 Basic siting criteria for the placement of high-volume sampler (either TSP or PM₁₀) are documented in Table 3. This list is not a complete listing of siting requirements; instead, an outline to be used by the operating agency to determine a sampler's optimum location. Complete siting criteria are presented in 40 CFR 58, Appendix E.

9.2.3 Additional factors not specified in the Code of Federal Regulations (CFR) must be considered in determining where the sampler will be deployed. These factors include accessibility under all weather conditions, availability of adequate electricity, and security of the monitoring personnel and equipment. The sampler must be situated where the operator can reach it safely despite adverse weather conditions. If the sampler is located on a rooftop, care should be taken that the operator's personal safety is not jeopardized by a slippery roof surface during inclement weather. Consideration also should be given to the fact that routine operation (i.e., calibrations, filter installation and recovery, flow checks, and audits) involves transporting supplies and equipment to and from the monitoring site.

9.2.4 To ensure that adequate power is available, consult the manufacturer's instruction manual for the sampler's minimum voltage and power requirements. Lack of a stable power source can result in the loss of many samples because of power interruptions.

9.2.5 The security of the sampler itself depends mostly on its location. Rooftop sites with locked access and ground-level sites with fences are common. In all cases, the security of the operating personnel as well as the sampler should be considered.

9.3 Sampler Installation Procedures

9.3.1 On receipt of a high-volume sampler (TSP or PM₁₀) from the manufacturer, visually inspect it and account for all components. Compare the equipment delivered with the enclosed packing slip. Notify the manufacturer immediately of any missing or damaged equipment.

9.3.2 Perform a laboratory check to determine if the sampler is operational. Turn on the sampler and observe the vacuum motor performance and shift the recorder response (if so equipped).

9.3.3 Carefully transport the sampler to the field site. If possible, install the sampler in the center of the site platform. This practice will ensure easy access to the sampler's inlet during maintenance procedures and will reduce inlet damage if the sampler should topple over.

9.3.4 Following manufacturer's instructions, carefully assemble the base and inlet of the sampler. The sampler must be bolted down to a secure mounting surface.

9.3.5 Check all tubing and power cords for crimps, cracks, or breaks.

9.3.6 Plug the power cord into a line voltage outlet. If possible, this outlet should be protected by a ground fault interrupter (GFI) for the operator's safety. The use of waterproof interlocking electrical connectors is also recommended to ensure operator safety and to avoid shorts or power interruptions. Do not allow any electrical connections to be submerged during periods of inclement weather.

9.3.7 Turn on the sampler and make sure that it is still working properly. Investigate and correct any malfunctions before proceeding. Operate the sampler for approximately 30 min to ensure that the motor brushes are properly seated and that the motor is operating at full performance.

9.3.8 Perform a multipoint flow-rate calibration, as described in Section 7.

9.4 Sampling Operations

9.4.1 General.

9.4.1.1 Operational procedures will vary according to the sampler model and options (e.g., the types of flow-rate controller and timer) selected for use in the monitoring program. Consult the instrument manual before putting the sampler into operation. Significant differences exist in the field operation of the two types of flow-controlling systems and, hence, in the determination of operational flow rates. The following assumptions are made in this section:

- The flow rate through a sampler that is equipped with a mass-flow controller is indicated by the exit orifice plenum pressure. This pressure is measured with a manometer (or a flow recorder).
- The flow rate through a sampler that is equipped with a volumetric-flow controller is indicated by the stagnation pressure. This pressure is measured with a manometer.
- The sampler has been calibrated according to procedure presented in Section 7.

9.4.1.2 The sampler has been calibrated according to procedures presented in Section 7.

9.4.1.3 The average actual flow rate for MFC samplers is calculated by determining the following:

- The average of the initial and final manometer readings of the exit orifice plenum pressure (or the average flow recorder reading).
- The average ambient temperature (T_{av}).
- The average ambient barometric pressure (P_{av}) during the sampling period.

These values are then applied to the sampler's calibration relationship. The 4" pressure flow recorders often supplied with HV samplers generally are not sufficiently accurate and are *not recommended* for quantitative sampler pressure or flow rate measurements. These flow recorders should be used only for nonquantitative determination that the flow was approximately constant and uninterrupted over the sampling period. The flow recorder may be connected in parallel with the manometer or other pressure measuring device using a tee or "Y" tubing connector.

[Note: Because flow recorders are still widely used for quantitative flow rate measurements, the procedures in this section include specific instructions for the use of a flow recorder. These flow recorder instructions are enclosed in brackets.]

9.4.1.4 The average actual flow rate for VFC samplers is calculated by determining the following:

- The average of the initial and final relative stagnation pressures (P_{stg}).
- The average ambient temperature (T_{av}).
- The average barometric pressure (P_{av}) during the sampling period and then by applying these values to the calibration relationship.

*[Note: Consistency of temperature and barometric pressure units is required. All temperatures should be expressed in kelvin ($K = EC + 273$). Also, all barometric pressures should be expressed in either mm Hg or kPa (**but don't mix the two units**). Avoid calibrating a PM_{10} sampler using one set of units and then performing sample calculations using another set.]*

9.4.2 Presampling Filter Preparation Procedures.

9.4.2.1 Most high-volume samplers (TSP or PM_{10}) have been designed to accept filter cassettes. Loading these cassettes in the laboratory will minimize damage; however, if extreme care is exercised, they can be loaded at the site when ambient conditions permit. Wear protective gloves when handling filters to avoid contaminating the filters with body oils and moisture. Keep the filters in protective folders or boxes. Never bend or fold unexposed filters. The analytical laboratory (and/or filter manufacturer) will give each filter an ID number. Because it is extremely difficult to see the "up" side of a quartz filter (i.e., the side with the slightly rougher texture), the filters should be consistently labeled on one side. When a filter that has

been labeled on its "down" side is folded for transport to the laboratory, its sample number will be readily accessible for documentation on laboratory log sheets upon arrival at the laboratory.

9.4.2.2 Following the manufacturer's instructions, carefully load the pre-weighted filter in the filter cassette. The filter should be centered on the wire screen so that the gasket will form an airtight seal on the outer edge of the filter when the faceplate is in place. Poorly aligned filters show uneven white borders after exposure. Care should be taken to ensure that the filter cassette is not excessively tightened, as the filter may stick or the gasket may be permanently damaged. Check that the gasket is in good condition and has not deteriorated.

9.4.3 Sampling Procedures--MFC Sampler.

9.4.3.1 Filter Installation Procedure.

9.4.3.1.1 Following the manufacturer's instructions, loosen the nuts that secure the inlet to the base and gently tilt back the inlet to allow access to the filter support screen.

9.4.3.1.2 Examine the filter support screen. If the screen appears dirty, wipe it clean. If the filter cassette is equipped with a protective cover, remove it and place the loaded cassette in position on the sampler support screen. Tighten the thumb nuts to hold the filter cassette securely. Check that the gasket is in good condition and has not deteriorated.

Caution: Tighten the thumb nuts evenly on alternate corners to properly align and seat the gasket. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.

9.4.3.1.3 If an inlet is being used, lower the sample inlet. Inspect the sample inlet to make sure that it is resting on the filter cassette and not on the sampler's frame. Secure the sample inlet to the sampler base.

9.4.3.1.4 Open the front door of the sample and examine the flow recorder. Remove any moisture inside by wiping it with a clean cloth. Record the sampler S/N, filter ID number, site location, and sampling data on the back of a clean chart and install the chart in the flow recorder.

[Note: Charts used for PM_{10} samplers normally have square-root-function scales; however, linear-function scales may be used. If charts with linear-function scales are used, Equations in Section 7.4.3.3 and Section 7.4.3.5 will have to be modified from their current form by replacing I with $(I)^{1/2}$]

[Note: While installing the chart, do not bend the pen arm beyond its limits of travel. Raise the pen head by pushing on the very top of the pen air (or by using the pen lift). Be sure that the chart tab is centered on the slotted drive to ensure full 360° rotation in 24 h. Make sure that the chart edges are properly located beneath the retainers. Lower the pen arm and tap the recorder face lightly to make certain that the pen is free.]

[Note: During periods of inclement weather, the chart tends to stick to the recorder face. Two charts can be installed simultaneously to enable the sample (top, annotated) chart to rotate freely.]

9.4.3.1.5 Using a coin or slotted screwdriver, advance the chart and check to see that the pen rests on zero--the smallest circle diameter. If necessary, adjust the zero set screw while gently tapping on the side of the flow recorder. If a chart with a linear function scale is used, some positive zero offset may be desirable to allow for normal variation in the zero readings.

9.4.3.1.6 Turn on the sampler and allow it to equilibrate to operating temperature (3-5 min).

9.4.3.1.7 While the sampler is equilibrating, record the following parameters on the MFC Sampler Field Data Sheet (see Figure 14):

- Site Location.
- Sample date.
- Filter ID number.
- Sampler model and S/N.
- Operator's initials.

9.4.3.1.8 Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer while watching for the free flow of the fluid. Adjust the manometer's sliding scale so that its zero line is at the bottom of the menisci.

9.4.3.1.9 Measure the initial exit orifice plenum pressure (Pex) using an oil or water manometer, with a 0-200-mm (0-8") range and a minimum scale division of 1 mm (0.1"). Record the initial Pex on the MFC Sampler Field Data Sheet. If Pex is substantially different than for previous samples or otherwise appears abnormal, carry out a Quality Control (QC) flow check as described in Section 13.1.

9.4.3.1.10 Verify that the flow recorder (if used) is operational and that the pen is inking. Note the flow recorder reading. If it is substantially different than for previous samples or otherwise appears abnormal, carry out a QC flow-check as described in Section 13.1.

9.4.3.1.11 Turn the sampler off.

9.4.3.1.12 Check the time indicated by the time-set pointer on the flow recorder. If it is in error, rotate the chart clockwise by inserting a screwdriver or coin in the slotted drive in the center of the chart face until the correct time is indicated.

9.4.3.1.13 Reset the elapsed time meter to 0000 min and the sampler timer for the next run day. Close the sampler door, taking care not to crimp the vacuum tubing or any power cords. The sampler is now ready to sample ambient air.

9.4.3.2 Filter Recovery Procedure. As soon as possible after sampling, the operator should return to the monitoring site to retrieve the exposed filter. Particle loss or filter damage will result if the filter is left in the sampler for extended periods.

9.4.3.2.1 Turn on the sampler and allow it to equilibrate to operating temperature (3-5 min).

9.4.3.2.2 Measure the final Pex and record it on the MFC Sampler Field Data Sheet.

9.4.3.2.3 Turn off the sampler.

9.4.3.2.4 Open the door of the sampler, remove the flow recorder chart, and examine the recorder trace. If the trace indicates extensive flow fluctuations, investigate and correct before the next sampling day.

9.4.3.2.5 Record the following parameters on the MFC Sampler Field Data Sheet:

- Elapsed time of the sampling period, min.
- Average recorder response, arbitrary units.
- Average ambient temperature for the run day (Tav), K ($K = EC + 273$).
- Average ambient barometric pressure for the run day (Pav), mm Hg or kPa.

[Note: Tav and Pav readings may be recorded or estimated on site or may be obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the evaluation are not available, seasonal average temperature (Ts) and barometric pressure (Ps) may be substituted for Tav and Pav, respectively. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. Therefore, seasonal values may represent actual values within 20EC and 40 mm Hg.]

The calculations presented in this section assume that the sampler has been calibrated in terms of actual temperature and barometric pressure and that the substitution of seasonal values is used only to determine the sampler's operational flow rate during a sample period. Although additional calculations to convert the sampler's calibration curve to seasonal can be made, the error represented by this method is negligible.

9.4.3.2.6 Calculate and record the average actual flow rate (as determined by the sampler's calibration relationship) on the MFC Sampler Field Data Sheet and on the back of the chart. Attach the chart to the data sheet.

$$Q_a = \{I\} \overline{P_{ex}} (T_{av} + 30)/P_a]^{1/2} - b\} \{1/m\}$$

or for the flow recorder,

$$\overline{Q_a} = \{[\overline{I}] (T_{av} + 30)/P_a]^{1/2} - b\} \{1/m\}$$

where:

$\overline{Q_a}$ = average sampler flow rate, actual m³/min.

$\overline{P_{ex}}$ = average exit orifice plenum pressure, mm Hg.

I = average flow recorder response, arbitrary units.

T_{av} = average ambient temperature for the run day, K.

P_{av} = average ambient pressure for the run day, mm Hg.

b = intercept of the MFC sampler calibration relationship.

m = slope of the MFC sampler calibration relationship.

[Note: If charts with linear-function scales are used, substitute (I)^{1/2} for I.]

9.4.3.2.7 Observe conditions around the monitoring site; note any activities that may affect filter particle loading (e.g., paving, mowing, fire) and record this information on the MFC Sampler Field Data Sheet.

9.4.3.2.8 Raise the sampler inlet and remove the filter cassette. Replace the cassette protective cover (if so equipped). To avoid particle loss, be careful to keep the cassette as level as possible.

9.4.3.2.9 The sampler may now be readied for the next run day.

9.4.3.2.10 Keeping the filter cassette level, carefully transport it, the data sheet, and the flow recorder chart to the laboratory sample custodian.

9.4.4 Sampling Procedures--VFC Sampler.

9.4.4.1 Filter Installation Procedure.

9.4.4.1.1 Following the manufacturer's instructions, loosen the nuts that secure the inlet to the base and gently tilt back the inlet to allow access to the filter support screen.

9.4.4.1.2 Examine the filter support screen. If the screen appears dirty, wipe it clean. If the filter cassette is equipped with a protective cover, remove it and place the loaded cassette in position on the sampler support screen. Tighten the thumb nuts sufficiently to hold the filter cassette securely. Check that the gasket is in good condition and has not deteriorated.

***Caution:** Tighten the thumb nuts evenly on alternate corners to properly align and seat the gasket. The nuts should be only hand-tightened because too much compression can damage the sealing gasket.*

9.4.4.1.3 If an inlet is used, lower the sample inlet and secure it to the sampler base. For impaction inlets, inspect the sample inlet to make sure that it is resting on the filter cassette and not on the sampler's frame. Secure the sampler inlet to the sampler base.

9.4.4.1.4 Record the following parameters on the VFC Sampler Field Data Sheet (see Figure 15):

- Site location.
- Sample date.
- Filter ID number.
- Sampler model and S/N.
- Operator's initials.

9.4.4.1.5 Turn on the sampler and allow it to reach a stable operating temperature (3-5 min).

9.4.4.1.6 Bring an oil or water manometer to the side of the sampler. This manometer should have a range of 0-400 mm (0-16") and a minimum scale division of 1 mm (0.1").

[Note: Be sure to convert the manometer reading to mm Hg using the following equation before recording the reading on the VFC Sampler Field Data Sheet.]

$$\text{mm Hg} = (25.4) (\text{in. H}_2\text{O}/13.6)$$

Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer, while watching for the free flow of the fluid.

Adjust the manometer's sliding scale so that its zero line is at the bottom of the menisci.

9.4.4.1.7 Remove the vacuum cap from the stagnation pressure port located on the side of the sampler base. Using the connecting tubing, attach one side of the manometer to the port. Leave the other side of the manometer open to atmospheric pressure. Make sure the tubing snugly fits the port and the manometer.

9.4.4.1.8 Measure the initial relative stagnation pressure () Pstg) and record this reading on the VFC Sampler Field Data Sheet.

9.4.4.1.9 Turn off the sampler, disconnect the manometer, and replace the vacuum cap on the stagnation pressure port.

9.4.4.1.10 Reset the elapsed-time meter to 0000 min and the sampler timer for the next run day. The sampler is now ready to sample ambient air.

9.4.4.2 Filter Recovery Procedure. As soon as possible after sampling, the operator should return to the monitoring site to retrieve the exposed filter. Particle loss or filter damage will result if the filter is left in the sampler for extended periods.

9.4.4.2.1 Turn on the sampler and allow it to warm up to operating temperature (3-5 min).

9.4.4.2.2 While the sampler is equilibrating, record the following parameters on the VFC Sampler Field Data Sheet:

- Elapsed time of the sampling period, min.
- Average ambient temperature for the run day (Tav), EC and K.
- Average ambient barometric pressure for the run day (Pav), mm Hg.

[Note: Tav and Pav readings may be recorded or estimated on site or may be obtained from a nearby U.S. National Weather Service Forecast Office, National Weather Service (NWS) station, or an airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If Tav and Pav readings are not available, seasonal average temperature (Ts) and barometric pressure (Ps) can be substituted. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. Therefore, seasonal values may represent actual values within 20EC and 40 mm Hg.]

9.4.4.2.3 Inspect the manometer for crimps or cracks in its connecting tubing. Open the valves and blow gently through the tubing of the manometer, while watching for the free flow of the fluid. Adjust the manometer sliding scale so that its zero line is at the bottom of the meniscuses.

9.4.4.2.4 Remove the vacuum cap from the stagnation pressure port located on the side of the sampler base. Using the connecting tubing, attached one side of the manometer to the port. Make sure that the tubing snugly fits the port and the manometer. Leave the other side open to atmospheric pressure.

9.4.4.2.5 Record the final Pstg on the VFC Sampler Field Data Sheet. Turn off the sampler and replace the vacuum cap.

[Note: Be sure to convert the manometer reading to mm Hg using the following equation before recording the reading on the Sampler Field Data Sheet.]

$$\text{mm Hg} = 25.4 (\text{in. H}_2\text{O}/13.6)$$

9.4.4.2.6 Calculate the average relative stagnation pressure (\overline{Pstg}) and record it on the data sheet.

9.4.4.2.7 Calculate the average absolute stagnation pressure ($\overline{P1}$) for the sample run day and record it on the data sheet.

$$\overline{P1} = P_{av} - \overline{Pstg}$$

where:

$\overline{P1}$ = average absolute stagnation pressure, mm Hg.

P_{av} = average ambient barometric pressure for the run day (not the retrieval day), mm Hg.

\overline{Pstg} = average stagnation pressure drop, mm Hg.

9.4.4.2.8 Calculate and record the average stagnation pressure ratio:

$$\text{Average stagnation pressure ratio} = P1/P_{av}$$

where:

P1 = average absolute stagnation pressure, mm Hg.

Pav = average ambient barometric pressure on the sample run day, mm Hg.

9.4.4.2.9 Using the manufacturer's lookup table (or an alternate calibration relationship as described in Section 7.5.4), locate the column and row corresponding to $\overline{P1}/P_{av}$ and the Tav value for the sample run day. Read and record the indicated \overline{Qa} value.

9.4.4.2.10 Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the VFC Sampler Field Data Sheet.

9.4.4.2.11 Raise the sampler inlet and remove the filter cassette. Replace the cassette protective cover (if so equipped). To avoid particle loss, be careful to keep the cassette as level as possible.

9.4.4.2.12 The sampler may now be readied for the next sampling period.

9.4.4.2.13 Keeping the filter cassette level, carefully transport it and the Sampler Field Data Sheet to the laboratory sample custodian.

9.4.5 Post-Sampling Filter Handling Procedures. If a sample will not be analyzed immediately, the sample custodian should store the filter within a protective covering. Because filter cassettes often prove too expensive and unwieldy for storage purposes, the use of a manila folder and a protective envelope of comparable size to that of the filter is recommended. Laboratory personnel should adhere to the following procedure:

9.4.5.1 Following the manufacturer's instructions, remove the top frame of the filter cassette.

9.4.5.2 Conduct a secondary check of a sample's validity as presented in "Laboratory Validation Criteria" (see Section 9.5).

9.4.5.3 Carefully slip a manila folder underneath the edge of the exposed filter. The filter may stick in the cassette because of overcompression of the filter cassette gasket. Be extremely careful to avoid damage to the brittle quartz filter.

9.4.5.4 Center the filter on the folder. If the filter must be touched, do not touch or jar the deposit. Fold the manila folder lengthwise at the middle with the exposed side of the filter in. If the collected sample is not centered on the filter (i.e., the unexposed border is not uniform around the filter), fold it so that only deposit touches deposit. **Do not crease the folder**--the sample filter may tear. If the filter shears or breaks, ensure that all pieces of the filter are included within the folder.

9.4.5.5 Insert the folder into the protective envelope.

9.4.5.6 Deliver the filter in its protective folder and envelope, accompanied by the completed data sheet, to the analytical laboratory.

9.5 Sample Validation and Documentation

9.5.1 **Field Validation Criteria.** After each sampling period, calculate the percentage difference between Q_a and the design flow rate (1.13 m³/min) using the following formula:

$$\% \text{ Difference} = 100 \frac{\overline{Q_a} - 1.13}{1.13}$$

Record this value on a control chart for the field validation of the sampler's actual volumetric flow rate as is shown in Figure 16.

- Decreases in flow rate during sampling (due to mechanical problems) of more than 10% from the initial set point result in sample invalidation. Recalibrate the sampler. A sample flow rate may also fluctuate due to heavy filter loading. If a high concentration is suspected, the operator should indicate this on the field data sheet. The laboratory supervisor will make the final decision regarding the sample's validity.
- Changes in flow-rate calibration of more than 10%, as determined by a field QC flow-rate check (see Section 13), will invalidate all samples collected back to the last calibration or valid flow check. Recalibrate the sample.

9.5.2 Laboratory Validation Criteria.

9.5.2.1 Upon receiving the filter from the field, check the filter for signs of air leakage by observing the border around the filter. If the border is clear, then the gasket on the sampler is still usable. However, if particulate matter is on the border, then air leakage has occurred and the gasket on the sampler should be changed. Leakage may result from a worn or improperly installed faceplate gasket. A gasket generally deteriorates slowly. The sample custodian should be able to decide well in advance (by the increased fuzziness of the sample outline) when to change the gasket before total gasket failure results. If signs of leakage are observed, void the sample, determine the cause, and instruct the operator to take corrective actions before starting another sampling period.

9.5.2.2 Check the exposed filter for physical damage that may have occurred during or after sampling. Physical damage after sampling would not invalidate the sample if all pieces of the filter were put in the

folder; however, complete losses of loose particulate after sampling (e.g., loss when folding the filter) would void the sample. Mark such samples as "void" on the HV data sheet.

9.5.2.3 Check the appearance of the particles. Any changes from normal color may indicate new emission sources or construction activity in the area. Note any change on the data sheet.

9.5.2.4 The filters should be weighed according to the procedures described in Inorganic Compendium Method IO-3.1, Section 5, *Gravimetric Analysis*.

9.5.3 Data Documentation. Recordkeeping is a critical part of the QA program. Careful documentation of sampling data will salvage samples that may otherwise be lost. The sheer repetition of recording data may result in errors; however, this cross-referencing between data sheets, log books, and (for those samplers so equipped) the continuous-flow recorder charts will allow the operator to pinpoint discrepancies that may result in a sample's invalidation.

[Note: The use of log books at monitoring sites is highly encouraged.]

9.5.3.1 Presampling Documentation and Inspection. The following information should be recorded on the Sampler Field Data Sheet (SFDS), sampler recorder chart (RC), flow-rate control chart (CC), and in the site log book (LB):

- Site Location.
- Sample Date.
- Filter ID number.
- Sample model and S/N.
- Operator's initials.

9.5.3.2 Post-Sampling Documentation and Inspection. Upon receipt of exposed filters from the field, the sample custodian should adhere to the following procedures.

9.5.3.2.1 Examine the field data sheet. Determine whether all data needed to verify sample validity and to calculate mass concentration are provided (e.g., average flow rate, ambient temperature, barometric pressure, and elapsed time). Void the sample if data are missing or unobtainable from a field operator or if a sampler malfunction is evident.

9.5.3.2.2 If the exposed filter was packaged for shipment, remove the filter from its protective envelope and examine the shipping envelope. If sample material has been dislodged from a filter, recover as much as possible by brushing it from the envelope onto the deposit on the filter with a soft camel's-hair brush.

9.5.3.2.3 Match the filter ID number with the correct laboratory data/coding form on which the original balance ID number, filter ID number, filter tare weight, and other information are inscribed. The sample custodian should group filters according to their recorded balance ID numbers. Initial separation of filters by balance ID number will decrease the probability of a balance error that could result from the use of different balances for tare and gross weights.

9.5.3.2.4 Remove the filter from the protective manila folder. Should the filter be retained in its filter cassette, loosen the nuts on the top and remove the filter. Overtightening the nuts may cause the filter to adhere to the cassette gasket. Gently remove it by the extreme corners to avoid damage. Inspect the filters for any damage that may have occurred during sampling. Conduct a secondary check of a sample's validity (as presented in Section 9.4). If insects are embedded in the sample deposit, remove them with Teflon®-tipped tweezers and disturb as little of the sample deposit as possible. If more than 10 insects are observed, refer the sample to the supervisor for a decision on acceptance or rejection of the filter for analysis.

9.5.3.2.5 Place defect-free filters in protective envelopes and forward them to the laboratory for weighing and analysis. File the data sheets for subsequent mass concentration calculations.

9.5.3.2.6 Place defective filters, with the type of defect(s) listed, in separate clean envelopes. Label the envelopes and submit them to the laboratory supervisor for final approval of filter validity.

10. Interferences

10.1 Large extraneous objects, such as insects, may be swept into the filter.

10.2 Liquid aerosols, such as oil mists and fog droplets, are retained by the filter. If the amount of liquid so collected is sizeable, the filter can become wet and its function may be impaired.

10.3 Any gaseous or vaporous constituent of the atmosphere under test that is reactive with or absorbed on the filter will be retained.

10.4 As the filter becomes loaded with collected matter, the sampling rate is reduced. If a significant drop in flow rate occurs, the average of the initial and final flow rate will not give an accurate estimate of total flow during the sampling period. The magnitude of such errors will depend on the amount of reduction of airflow rate and on the variation of the mass concentration of dust with time during the 24-h sampling period. As an approximate guideline, any sample should be suspect if the final flow rate is less than one-half the initial rate.

10.5 Power failure or voltage change during the test period will lead to an error, depending on the extent and time duration of such failure.

10.6 The passive loading of the filter left in place for any time prior to or following a sampling period can introduce an error. The timely installation and removal of the filter is advisable, or a sampler with shutters may be used.

10.7 If two or more samplers are used at a given location, they should be placed at least 2 meters apart so that one sampler will not affect the results of an adjacent sampler.

10.8 Recent wind tunnel studies have shown significant possible sampling errors as a function of sampler orientation in atmospheres containing high relative concentrations of large particles.

10.9 Metal dusts from motors, especially copper, may significantly contaminate samples under some conditions.

10.10 Under some conditions, atmospheric SO₂ and NO_x may interfere. Artifact formation errors are caused by the retention of sulfur dioxide in the form of sulfate particulate on alkaline filters. Experiments involving a variety of filters indicate that sulfate loading errors of 0.3-3.0 µg/m³ can be expected with the use of common glass fiber filters under normal sampling conditions and that larger sulfate errors are possible under extreme sampling conditions. A neutral or low-alkalinity filter medium will eliminate excessive artifact formation.

10.11 Guidelines to help prevent post-sampling particle loss are presented in Section 8.

11. Calculations, Validations, and Reporting of TSP and PM₁₀ Data

11.1 Basic Information Used for Calculations

11.1.1 The design flow rate is specified as an actual volumetric flow rate (Q_a), measured at existing conditions of temperature (T_a) and pressure (P_a). The sampler's operational flow rate should be very close to the design flow rate. All samplers have some means for measuring the operational flow rate, and that flow rate measurement system must be calibrated periodically with a certified flow rate transfer standard. Usually, measurements (or estimates) of ambient temperature and barometric pressure are required to get an accurate indication of the operational flow rate. To determine the average sampler flow rate over a sample period, use the average temperature (T_{av}) and average barometric pressure (P_{av}) over the sample period. However, if average temperature and pressure values (or reasonable estimates) cannot be obtained for each sample period, seasonal average temperature (T_s) and barometric pressure (P_s) for the site may be substituted.

[Note: T_{av} and P_{av} readings may be recorded on site or estimated from data obtained from a nearby U.S. National Weather Service Forecast Office, NWS station, or local airport weather station. Barometric pressure readings obtained from airports or other sources must be at station pressure (i.e., not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the airport. If individual T_{av} and P_{av} readings cannot be obtained for each sample period and seasonal averages for the site are routinely substituted, care must be taken to ensure that the actual temperature and barometric pressure at the site are reasonably represented by such averages. Therefore, seasonal average temperature and pressure values (T_s and P_s) for the site by should be used only when these values are within 20 K and 40 mm Hg (5 kPa) of the actual average temperature and barometric pressure (T_{av} and P_{av}) for the sample period.]

11.1.2 The calculations presented in this section assume that the sampler has been calibrated in actual volumetric flow rate units (Q_a) and that individual average temperature and barometric pressure values are used for each sample period. If seasonal average temperature and pressure values for the site are to be used, T_s may be substituted for T_{av} , and P_s may be substituted for P_{av} .

11.1.3 The true or actual flow rate through the sampler inlet must be known and controlled. A common source or error in a monitoring program is confusion of various air volume flow-rate measurement units. Although the sampler's operational flow rate must be monitored in terms of actual volume flow rate units (Q_a), sampler flow rates can be corrected to standard volume flow rate units (Q_{std}) at EPA standard conditions of temperature (25°C) and pressure (760 mmHg).

- Q_a : Actual volumetric air flow rates, measured and expressed at existing conditions of temperature and pressure and denoted by Q_a (Q_{actual}). Typical units are L/min and m³/min. Inlet design flow rates for PM₁₀ samplers are always given in actual volumetric flow rate units.
- Q_{std} : Airflow rates that have been corrected to equivalent standard volume flow rates at EPA standard conditions of temperature and pressure (25°C or 298 K and 760 mm Hg or 101 kPa) and denoted by Q_{std} ($Q_{standard}$). Typical units are std. L/min, and std. m³/min. Standard volume flow-rate units are often used by engineers and scientists because they are equivalent to mass flow units.

11.1.4 The Q_a and Q_{std} measurement units must not be confused or interchanged. The flow-rate units can be converted as follows, provided the existing temperature and pressure (or in some cases the average temperature and pressure over a sampling period) are known:

$$\begin{aligned}\overline{Q_{std}} &= \overline{Q_a} (P_a/P_{std})(T_{std}/T_a) \\ Q_{std} &= (P_{av}/P_{std})(T_{std}/T_{av}) \\ Q_a &= Q_{std}(P_{std}/P_a)(T_a/T_{std})\end{aligned}$$

where:

- Q_{std} = standard volume flow rate, std m³/min.
- Q_a = actual volume flow rate, actual m³/min.
- P_a = ambient barometric pressure, mm Hg.
- P_{std} = EPA standard barometric pressure, 760 mm Hg.
- T_{std} = EPA standard temperature, 298 K.
- T_a = standard temperature, K (K = EC + 273).
- $\overline{Q_{std}}$ = average standard volume flow rate for the sample period, std. m³/min.
- $\overline{Q_a}$ = average actual volume flow rate for the sample period, m³/min.
- P_{av} = average ambient barometric pressure during the sample period, mm Hg.
- T_{av} = average ambient temperature during the sample period, K.

Inorganic Compendium Method IO-2.4 provides guidance on calculating sample volume corrected to EPA standard temperature and pressure.

11.2 Flow-Rate Calculations. Because flow control methods (and hence, calibration procedures) vary among different sampler models, the calculations necessary to determine the average actual flow rate during a sample run will also differ. The following general procedures are recommended for calculating the average ambient flow rate of the sampler. In this section, it is assumed that the samplers have been calibrated according to procedures outlined in Section 7.

[Note: Consistency in units is required. Adoption of uniform designations of K for temperature and mm Hg (or kPa) for pressure is recommended in all calculations.]

11.2.1 MFC Sampler.

11.2.1.1 The average actual flow rate for sample period is calculated by determining:

- The average of the initial and final manometer readings ($\overline{P_{ex}}$) [or the average flow recorder trace];
- The average ambient temperature (T_{av}); and
- The average ambient barometric pressure (P_{av}) during the sampling period and applying these values to the calibration relationship.

11.2.1.2 Each sampler's flow measurement system should be calibrated periodically, and the calibration should be described by a mathematical expression (e.g., a least-squares linear regression equation) that indicates the slope and intercept of the calibration relationship. Following the procedure in Section 7, this expression is in the form of:

$$\overline{Q_a} = \{[\overline{P_{ex}}(T_{av} + 30)/P_{av}]^{1/2} - b\} \{1/m\}$$

where:

- $\overline{Q_a}$ = the sampler's average actual flow rate for the sample period, m³/min.
- $\overline{P_{ex}}$ = average of initial and final sampler manometer readings, $(P_{ex_i} + P_{ex_f})/2$, in. H₂O.
- T_{av} = average barometric pressure for the sample period, K (K = EC + 273).
- P_{av} = average barometric pressure for the sample period, mm Hg.
- b = intercept of the sampler calibration relationship.
- m = slope of the sampler calibration relationship.

For the flow recorder,

$$\overline{Q_a} = \{[\overline{I} (T_{av} + 30)/P_{av}]^{1/2} - b\} \{1/m\}$$

where:

- \overline{I} = average flow recorder reading for the sample period.

11.2.1.3 The average actual flow rate is then corrected to EPA-standard conditions, calculated as:

$$\overline{Q_{std}} = \overline{Q_a} (P_{av}/P_{std})(T_{std}/T_{av})$$

where:

- $\overline{Q_{std}}$ = average sampler flow rate corrected to EPA-standard volume flow rate units, std. m³/min.
- $\overline{Q_a}$ = average actual sampler flow rate for the sample period, m³/min.
- P_{std} = standard barometric pressure, 760 mm Hg.
- T_{std} = standard temperature, 288 K.

11.2.2 VFC Sampler.

11.2.2.1 The average actual flow rate for the sample period is calculated by determining the ratio of the average absolute stagnation pressure of the average ambient barometric pressure ($\overline{P_1}/P_{av}$) and the ambient average temperature (T_{av}) for the sampler period.

11.2.2.2 Calculate the value of P_1 in mm Hg:

$$\overline{P_1} = P_{av} - \overline{P_{stg}}$$

where:

- P_1 = average absolute stagnation pressure for the sample period, mm Hg.
- P_{av} = average barometric pressure for the sample period, mm Hg.
- $\overline{P_{stg}}$ = average of initial and final relative stagnation pressure readings, mm Hg.

[Note: Be sure to convert a water manometer reading to mm Hg using the following equation before recording the reading on the data sheet:]

$$\text{mmHg} = 25.4 (\text{H}_2\text{O}/13.6)$$

11.2.2.3 Calculate and record the value of the average stagnation pressure ratio.

$$\text{Average stagnation pressure ratio} = (\overline{P_1}/P_{av})$$

11.2.2.4 Use the manufacturer's lookup table (or alternate calibration relationship; see Section 7) to determine Q_a from the average stagnation pressure ratio ($\overline{P_1}/P_{av}$) and T_{av} for the sample period. The value of $\overline{Q_a}$ is the average volumetric flow rate for the sampler period.

11.2.2.5 The average actual flow rate is then corrected to EPA-standard conditions using the following equation:

$$\overline{Q_{std}} = \overline{Q_a} (P_{av}/P_{std})(T_{std}/T_{av})$$

where:

$\overline{Q_{std}}$ = average sampler flow rate corrected to EPA-standard volume flow rate units, std. m³/min.

$\overline{Q_a}$ = average actual sampler flow rate for the sample period, m³/min.

P_{std} = standard barometric pressure, 760 mm Hg.

T_{std} = standard temperature, 298 K.

11.3 The total standard volume of air sampled is calculated by the following equation:

$$V_{std} = (\overline{Q_{std}})(t)$$

where:

V_{std} = total volume of air sampled in standard volume units, std m³.

$\overline{Q_{std}}$ = average sampler flow rate corrected to EPA-standard conditions, std m³/min.

t = total elapsed sampling time, min.

11.4 Percent Difference

11.4.1 After each sampling period, calculate the percentage difference between Q_a and the design flow rate (1.13 m³/min) using the following formula:

$$\% \text{ Difference} = 100 \frac{Q_a - 1.13}{1.13}$$

Record this value on a control chart for the field validation of the sampler's actual volumetric flow rate as is shown in Figure 14.

11.4.2 The following criteria should be used as the basis for determining a sample's validity:

- Decreases in flow rate during sampling (due to mechanical problems) of more than 10% from the initial set point cause sample invalidation. A sample flow rate may also fluctuate due to heavy filter loading. If a high concentration is suspected, the operator should indicate it on the field data sheet. The laboratory supervisor will make the final decision regarding the sample's validity.
- Changes in flow-rate calibration of more than 10%, as determined by a field QC flow-rate check, will invalidate all samples collected back to the last calibration or valid flow check.

12. Records

12.1 MFC Sampler

Record the following parameters on the MFC Sampler Field Data Sheet (see Figure 14):

- Final Pex.
- Elapsed time of the sampling period, min.
- Average record response, arbitrary units.
- Tav for the run day K ($K = EC + 273$).
- Pav for the run day, mm Hg.

12.2 VFC Sampler

Record the following parameters on the VFC Sampler Field Data Sheet (see Figure 15):

- Site location.
- Sample date.
- Filter ID number.
- Sampler model and S/N
- Operator's initials.
- Initial Relative Stagnation Pressure (Pstg).
- Elapsed time of the sampling period, min.
- Tav for the run day Tav, EC and K.
- Pav for the run day Pav, mm Hg.
- Pstg, mm Hg.
- Relative Stagnation Pressure.
- Absolute Stagnation Pressure.
- Qa value (from chart generated in Section 7.5.4.).

12.3 Tav and Pav readings may be recorded or estimated on site or may be obtained from a nearby U.S. National Weather Service Forecast Office or airport weather station. Barometric pressure readings obtained from remote sources must be at station pressure (not corrected to sea level); they may have to be corrected for differences between elevation of the monitoring site and that of the airport. If Tav and Pav readings are not available, seasonal average temperature (Ts) and barometric pressure (Ps) may be substituted for Tav and Pav, respectively. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. Therefore, seasonal values should represent actual values within 20EC and 40 mm Hg.

12.4 Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the VFC Sampler Field Data Sheet.

Document any factors that may cause a sample's invalidation on the sample data sheet. Forward the data sheet and the filter to the laboratory supervisor, who will make the final decision regarding the sample's validity.

12.5 Record the percentage difference between Qa and the design flow rate on Figure 16.

12.6 Recordkeeping is a critical part of the QA program. Careful documentation of sampling data will salvage samples that may otherwise be lost. The sheer repetition of recording data may result in errors;

however, this cross-referencing between data sheets, log books, and (for those samplers so equipped) the continuous-flow-recorder charts will allow the operator to pinpoint discrepancies that may result in a sample's invalidation.

[Note: The use of log books at monitoring sites is highly encouraged. The following information should be recorded on the Sampler Field Data Sheet (SFDS), sampler recorder chart (RC), in the site log book (LB), and on the flow-rate control chart (CC).]

12.6.1 The following information should be recorded by the operator who starts the sample. (The designation in parentheses indicates where the data must be inscribed):

- Site designation and locations (SFDS)(RC)(LB). This information should be recorded in the log book only once, at the initiation of a monitoring program.
- Sampler model and S/N (SFDS)(RC)(LB). This information needs to be recorded in the log book only at the commencement of monitoring, unless there is more than one sampler or a new sampler has been deployed.
- Filter ID number (SFDS)(RC)(LB).
- Sample date (SFDS)(RC)(LB).
- Initial Pex for MFC or initial) Pstg for VFC (SFDS)(LB).
- Unusual conditions that may affect the results (e.g., subjective evaluation of pollution that day, construction activity, weather conditions) (SFDS)(LB).
- Operator's initials (SFDS).
- Signature (LB).

12.6.2 The following information should be recorded by the operator who removes the samples.

- Elapsed time of the sample run (SFDS)(RC)(LB).
- Final) Pex [or mean I] for MFC or final) Pstg, $\overline{P_1}$, and $\overline{P_1}/P_{av}$ for VFC (DS)(LB)[RC].
- The calculated standard average flow rate (Qstd) in std m³/min (SFDS)(LB).
- The percentage difference between the actual and design flow rates (CC).
- Average ambient temperature and barometric pressure on the sample run day (SFDS)(LB).
- Seasonal average temperature and pressure, if needed (SFDS/LB). This information needs to be recorded in the logbook once, at the change of each season.
- Existing conditions that may affect the results (SFDS)(LB).
- Explanations for voided or questionable samples (SFDS)(LB).
- Operator's initials (SFDS).
- Signature (LB).

13. Field QC Procedure

For HV samplers, a field-calibration check of the operational flow rate is recommended at least once per month. The purpose of this check is to track the sampler's calibration stability. A control chart (e.g., Figure 14) that contains the percentage difference between a sampler's indicated and measured flow rates should be maintained. This chart is a quick reference of instrument flow-rate drift problems and is useful for tracking the performance of the sampler. Either the sampler log book or a data sheet must be used to document flow-check information. This information includes, but is not limited to, instrument and transfer standard model and serial numbers, ambient temperature and pressure conditions, and collected flow-check data.

In this section, the following is assumed:

- The flow rate through sampler that is equipped with a mass-flow controller is indicated by the exit orifice plenum pressure. This pressure is measured with a manometer [or a flow recorder].
- The flow rate through a sampler that is equipped with a volumetric flow controller is indicated by the stagnation pressure. This pressure is measured with a manometer.
- The acceptable flow-rate fluctuation range is 10% of the design flow rate.
- The transfer standard will be an orifice device equipped with a water or oil manometer.
- The orifice transfer standard's calibration relationship is in terms of the actual volumetric flow rate (Qa).

13.1 QC Flow-Check Procedure--MFC Sampler. The indicated flow rate [Qa (sampler)] for MFC samplers is calculated by determining:

- The manometer reading of the exit orifice plenum pressure [or the flow recorder reading],
- The ambient temperature (Ta), and
- The barometric pressure (Pa) during the flow check.

These values are then applied to the sampler's calibration relationship. The 4" pressure (flow) recorders of the type often supplied with high-volume PM₁₀ samplers are generally not sufficiently accurate and are not recommended for quantitative sampler pressure or flow measurements. The flow recorder may be connected in parallel with the manometer or other pressure measuring device, using a tee or "Y" tubing connector. An alternate QC flow-check procedure may be presented in the manufacturer's instruction manual. The manual should be reviewed and the various methods evaluated. Inhouse equipment and procedural simplicity should be considered in determining which method to use.

[Note: Do not attempt to conduct a flow check of samplers under windy conditions. Short-term wind velocity fluctuations will produce variable pressure readings by the orifice transfer standard's manometer. The flow check will be less precise because of the pressure variations.]

13.1.1 Collect the following equipment and transport it to the monitoring station:

[Note: An independent person should perform the QC flow check, with an outside observer present.]

- A water, oil, or digital manometer with a 0-200 mm (0-8") range and a minimum scale division of 1 mm (0.1") for measuring the sampler's exit orifice plenum pressure. This manometer should be the same as is used routinely for sampler flow rate measurements.
- An orifice transfer standard and its calibration relationship (different from initial orifice standard).
- An associated water or oil manometer with a 0- to 400-mm (0- to 16") range and a minimum scale division of 1 mm (0.1") for measuring the orifice transfer standard.
- A thermometer capable of accurately measuring temperature 0-50EC (273-323 K) to the nearest ± 1EC and referenced to an NIST or ASTM thermometer within ± 2EC at least annually.
- A portable aneroid barometer (e.g., a climber's or engineer's altimeter) capable of accurately measuring ambient pressure 500-800 mm Hg (66-106 kPa) to the nearest millimeter Hg and referenced within ± 5 mm Hg of a barometer of known accuracy at least annually.
- The sampler's calibration information.
- Spare recorder charts and a clean flow-check filter.
- MFC Sampler Flow-Check Data Sheet or site log book.

13.1.2 Record the site location, sampler S/N, and date on the back of a clean chart and install it in the flow recorder. While installing the chart, do not bend the pen arm beyond its limits of travel. Raise the pen head by pushing on the very top of the pen arm (or by using the pen lift) and simultaneously insert the chart.

13.1.3 Lower the pen arm and tap the recorder face lightly to make certain that the pen can move freely.

13.1.4 Using a coin or slotted screwdriver, advance the chart and check to see that the pen head rests on zero (i.e., that smallest diameter circle). If necessary, adjust the zeroset screw while gently tapping on the side of the recorder. A quarter turn of the set screw usually results in large offsets; adjust the set screw carefully.

13.1.5 Set up the flow-check system as previously illustrated in Figure 10. MFC samplers are normally flow-checked with a filter in line (i.e., between the orifice transfer standard and the motor). Install a clean filter in the sampler. Place the filter directly upon the sampler's filter screen. Do not use a filter cassette. A flow-check filter should never be used for subsequent sampling because particles larger than 10 Fm can be collected on the filter while the inlet is raised. The sample mass will be biased as a result of using a filter for both a flow check and subsequent sampling.

13.1.6 Install the orifice transfer standard and its faceplate on the sampler. Do not restrict the flow rate through the orifice (i.e., by using fixed resistance plates or closing the variable-resistance valve).

Caution: Tighten the faceplate nuts on alternate corners first to eliminate leaks and to ensure even tightening. The nuts should be hand-tightened; too much compression can damage the sealing gasket. Make sure the orifice transfer standard gasket is in place and the orifice transfer standard is not cross-threaded on the faceplate.

13.1.7 Connect the orifice manometer to the pressure port of the orifice transfer standard and the sampler manometer to the sampler's exit orifice plenum. Inspect the manometers' connecting tubings for crimps and cracks. Open the manometer valves and blow gently through the tubings. Watch for the free flow of fluid. Adjust the manometers' scales so that their zero lines are at the bottom of the meniscuses. Make sure that the connecting tubing snugly fits the manometer and the pressure port.

13.1.8 Turn on the sampler and allow it to warm up to operating temperature (3-5 min).

[Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2" of clearance at the bottom for air flow and for the manometer tubing.]

13.1.9 Read and record the following parameters on the MFC Sampler Flow-Check Data Sheet:

- Sampler location and date.
- Sampler model and S/N.
- Ambient temperature (T_a), EC and K.
- Ambient barometric pressure (Pa), mm Hg.
- Unusual weather conditions.
- Orifice transfer standard S/N and calibration relationship.
- Operator's signature.

13.1.10 Observe the H_2O across the orifice by reading the manometer deflection. Record the manometer deflection on the MFC Sampler Flow-Check Data Sheet (see Figure 11).

13.1.11 Measure the exit orifice plenum pressure (P_{ex}) by reading the manometer deflection. Record the manometer deflection on the MFC Sampler Flow-Check Data Sheet.

13.1.12 Using a coin or small screwdriver, advance the recorder chart to read the sampler's corresponding response (I) and record on the data sheet. A gentle tap on the recorder face is often necessary to ensure that the pen is not sticking to the chart.

13.1.13 Turn off the sampler and remove the orifice transfer standard, but not the filter. Turn on the sampler and repeat Section 13.1.11 [or Section 13.1.12] to check the flow rate under normal operating conditions. Turn off the sampler and remove the filter.

13.1.14 Calculate and record $Q_a(\text{orifice})$ at actual conditions using the following equation:

$$Q_a(\text{orifice}) = \{[(\text{H}_2\text{O})(T_a/P_a)]^{1/2} - b\} \{l/m\}$$

where:

- $Q_a(\text{orifice})$ = actual volumetric flow rate as indicated by the orifice transfer standard, m^3/min
- H_2O = pressure drop across the orifice, in. H_2O .
- T_a = ambient temperature, K.
- P_a = ambient barometric pressure, mm Hg.
- b = intercept of the orifice calibration relationship.
- m = slope of the orifice calibration relationship.

13.1.15 Calculate and record the corresponding sampler flow rate at actual conditions using the following equation:

$$Q_a(\text{sampler}) = \{P_{\text{ex}} (T_a + 30)/P_a\}^{1/2} - b\} \{l/m\}$$

or use the following if a flow recorder is being used to measure the exit orifice plenum pressure:

$$Q_a(\text{sampler}) = \{I(T_a + 30)/P_a\}^{1/2} - b\} \{l/m\}$$

where:

- $Q_a(\text{sampler})$ = sampler flow rate, actual m^3/min .
- P_{ex} = exit orifice plenum pressure, in. H_2O .
- T_a = ambient temperature during the flow check, K ($K = \text{EC} + 273$).
- P_a = ambient barometric pressure during the flow check, mm Hg.
- b = intercept of the MFC sampler calibration relationship.
- m = slope of the MFC sampler calibration relationship.

[Note: If charts with linear-function scales are used, substitute $(I)^{1/2}$ for I .]

13.1.16 Using this information and the formulas provided on the MFC Sampler Flow-Check Data Sheet, calculate the QC check percentage differences.

$$\text{QC\&check \% difference} = \frac{[Q_a(\text{sampler}) \& Q_a(\text{orifice})]}{Q_a(\text{orifice})} [100]$$

where:

$Q_a(\text{sampler})$ is measured with the orifice transfer standard being installed.

Record this value on the MFC Sampler Flow-Check Data Sheet and plot on the QC control chart. If the sampler flow rate is within 93-107% ($\pm 7\%$ difference) of the calculated $Q_a(\text{orifice})$ flow rate (in actual volumetric units), the sampler calibration is acceptable. If these limits are exceeded, investigate and correct any malfunction. Recalibrate the sampler before sampling is resumed. Differences exceeding $\pm 10\%$ may result in the invalidation of all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the orifice transfer standard's calibration and all calculations.

13.1.17 Calculate the corrected sampler flow rate, $Q_a(\text{corr. sampler})$, using the following equation:

$$Q_a(\text{corr. sampler}) = [Q_a(\text{sampler})] \frac{[(100 \pm \% \text{ difference})]}{100}$$

where:

$Q_a(\text{sampler})$ is measured without the orifice transfer standard being installed and where the QC-check percentage difference was obtained from the equation above.

[Note: Take care to use the correct sign (i.e., positive or negative) for the percent difference.]

13.1.18 Calculate and record on the MFC Sampler Flow-Check Data Sheet the percentage difference between the inlet's design flow rate and the corrected sampler flow rate as:

$$\text{Design flow rate \% difference} = \frac{[Q_a(\text{corr. sampler}) \pm 1.13]}{1.13} [100]$$

[Note: The author assumes in this section that the inlet is designed to operate at a flow rate of 1.13 actual m^3/min . If the design flow rate percentage difference is less than or equal to $\pm 7\%$, the sampler calibration is acceptable. If the difference is greater than $\pm 7\%$, investigate potential error sources and correct any malfunction. Recalibrate the sampler before sampling is resumed. Differences exceeding $\pm 10\%$ may invalidate all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calculations.]

[Note: Deviations from the design flow rate may be caused in part by deviations in the site temperature and pressure from the seasonal average conditions. Recalculate the optimum set-point flow rate (SFR) according to Section 7.4.4 to determine if the flow controller should be adjusted.]

13.1.19 Set up the sampler for the next sampling period according to the operating procedure in Section 9.4.

13.2 QC Flow-Check Procedure--VFC Sampler

The indicated flow rate ($Q_a(\text{sampler})$) for VFC samplers is calculated by determining:

- The relative stagnation pressure (Pstg),
- The ambient temperature (T_a), and
- The barometric pressure (P_a) during the flow check.

These values are then applied to the sampler's calibration relationship. An alternative QC flow-check procedure may be presented in the manufacturer's instruction manual. The manual should be reviewed and the various methods evaluated. Inhouse equipment and procedural simplicity should be considered in determining which method to use.

[Note: Do not attempt to conduct a flow check of samplers under windy conditions. Short-term wind velocity fluctuations will provide variable pressure readings by the orifice transfer standard's manometer.]

The flow check will be less precise because of the pressure variations.

13.2.1 Collect the following equipment and transport it to the monitoring station:

- An orifice transfer standard and its calibration relationship in actual volumetric flow units (Qa).
- An associated oil, water, or digital manometer, with a 0-400 mm (0-16") range and minimum scale divisions of 1 mm (0.1").
- An oil, water, or digital manometer, with a 0-400 mm (0-16") range and minimum scale divisions of 1 mm (0.1") or other pressure measurement device for measurement of the sampler stagnation pressure. Ideally, this manometer (or other pressure measurement device) should be associated with the sampler.

[Note: Manometers used for QC flow-checks may be subject to damage or malfunction and thus should be checked frequently.]

- A thermometer capable of accurately measuring temperature from 0E-50EC (273-323 K) to the nearest ± 1 EC and referenced to an NIST or ASTM thermometer within 2EC at least annually. To calculate the orifice flow rates, convert EC to K.
- A portable aneroid barometer (e.g., a climber or engineer's altimeter) capable of accurately measuring ambient barometric pressure over the range of 500-800 mm Hg to the nearest millimeter Hg and referenced within 5 mm Hg of a barometer of known accuracy at least annually.
- The sampler's calibration relationship (i.e., lookup table or alternative calibration relationship).
- A clean flow-check filter loaded into a filter cassette.
- A VFC Sampler Flow-Check Data Sheet (see Figure 13) or a site log book.

13.2.2 Set up the flow-check system as previously illustrated in Figure 12. VFC samplers are normally flow-checked with a loaded filter cassette in line (i.e., between the orifice transfer standard and the motor). The orifice transfer standard should be installed without fixed resistance plates or with the adjustable resistance value fully open. A flow-check filter should never be used for subsequent sampling because particles larger than 10 Fm can be collected on the filter while the inlet is raised. The sample mass will be biased as a result of using a filter for both a flow check and subsequent sampling.

Caution: Tighten the faceplate nuts on alternate corners first to eliminate leaks and to ensure even tightening. The fittings should be hand-tightened; too much compressing can damage the sealing gasket. Make sure the orifice gasket is in place and the orifice transfer standard is not cross-threaded on the faceplate.

13.2.3 Turn on the sampler and allow the sampler to warm up to operating temperature (3-5 min).

[Note: The sampler inlet may be partially lowered over the orifice transfer standard to act as a draft shield (if a shield is not otherwise provided). Use a block to provide at least 2" of clearance at the bottom for air flow and for the manometer tubing.]

13.2.4 Read and record the following parameters on the VFC Sampler Flow-Check Data Sheet (see Figure 13):

- Sampler location and date.
- Sampler S/N and model.
- Ambient temperature (Ta), EC and K.
- Ambient barometric pressure (Pa), mm Hg.
- Unusual weather conditions.
- Orifice transfer standard S/N and calibration relationship.
- Operator's signature.

13.2.5 Inspect the manometers for crimps or cracks in the connecting tubing. Open the valves and blow gently through the tubing, watching for the free flow of the fluid. Adjust the manometers' sliding scales so that the zero lines are at the bottom of the meniscuses.

13.2.6 Connect the orifice manometer to the orifice transfer standard and the sampler manometer to the sampler stagnation pressure port located on the side of the sampler base. Ensure that one side of each manometer is open to atmospheric pressure. Be sure that the connecting tubing snugly fits the pressure ports and the manometers.

13.2.7 Read the pressure drop as indicated by the orifice manometer (ΔH_2O) and record the value on the VFC Sampler Flow-Check Data Sheet. Read the stagnation pressure drop and record it as ΔP_{stg} (mm Hg) on the data sheet.

[Note: Be sure to convert the manometer reading to mm Hg using the following equation before recording the reading on the data sheet.]

$$\text{mm Hg} = 25.4(\text{in. H}_2\text{O}/13.6)$$

13.2.8 Turn off the sampler and remove the orifice transfer standard.

13.2.9 With only a loaded filter cassette in line, turn on the sampler and allow it to warm up to operating temperature.

13.2.10 Read and record the stagnation pressure drop (ΔP_{stg}) for the normal operating flow rate. Turn off the sampler. Replace the vacuum cap on the stagnation pressure port.

13.2.11 Calculate and record $Q_a(\text{orifice})$ flow rate for the flow-check point, as in the equation, reproduced below:

$$Q_a(\text{orifice}) = \{[(\Delta H_2O)(T_a/P_a)]^{1/2} - b\} [l/m]$$

where:

- $Q_a(\text{orifice})$ = actual volumetric flow rate as indicated by the transfer standard orifice, m^3/min .
- ΔH_2O = pressure drop across the orifice, in. H_2O .
- T_a = ambient temperature, K ($K = ^\circ C + 273$).
- P_a = ambient barometric pressure, mm Hg.
- b = intercept of the orifice calibration relationship.
- m = slope of the orifice calibration relationship.

13.2.12 Calculate and record the value of P_l (mm Hg) for the measurements, with and without the orifice installed, according to the following equation:

$$P_l = [P_a - \Delta P_{stg}]$$

where:

- P_l = stagnation pressure, mm Hg.
- P_a = ambient barometric pressure, mm Hg.
- ΔP_{stg} = stagnation pressure drop, mm Hg.

13.2.13 Calculate and record the stagnation pressure ratio for the measurements, with and without the orifice installed, according to the following equation:

$$\text{Stagnation pressure ratio} = P_l/P_a$$

where:

- P_l = stagnation pressure, mm Hg.
- P_a = ambient barometric pressure, mm Hg.

13.2.14 Refer to the instrument manufacturer's lookup table (or alternative calibration relationship as described in Section 7.5.4) and determine the $Q_a(\text{sampler})$ flow rates (m^3/min) for the measurements with

and without the orifice installed as indicated for the ratio of P_l/P_a and ambient temperature in EC. Record these values on the VFC sampler flow check data sheet.

13.2.15 Using $Q_a(\text{orifice})$ and $Q_a(\text{sampler})$ for the measurements with the orifice installed, calculate the QC-check percentage difference as:

$$\text{QC\&check \% difference} = \frac{[Q_a(\text{sampler}) \& Q_a(\text{orifice})]}{Q_a(\text{orifice})} [100]$$

Record this value on the VFC Sampler Flow-Check Data Sheet and plot it on the control chart for QC flow checks. If the QC-check percentage difference is less than or equal to $\pm 7\%$, the sampler calibration is acceptable. Those differences exceeding $\pm 7\%$ will require recalibration. Differences exceeding $\pm 10\%$ may invalidate all data collected subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calibrations.

13.2.16 Using this percentage difference and $Q_a(\text{sampler})$ from the measurements without the orifice installed (i.e., for the normal operating flow rate), calculate the corrected sampler flow rate as:

$$Q_a(\text{corr. sampler}) = [Q_a(\text{sampler})] \frac{[(100 \& \% \text{ difference})]}{100}$$

Record $Q_a(\text{corr. sampler})$ on the VFC Sampler Flow-Check Data Sheet.

13.2.17 Determine the design flow rate percentage difference between the PM_{10} sampler inlet design flow rate (e.g., $1.13 \text{ m}^3/\text{min}$) and $Q_a(\text{corr. sampler})$ as:

$$\text{QC\&check \% difference} = \frac{[Q_a(\text{sampler}) \& Q_a(\text{orifice})]}{Q_a(\text{orifice})} [100]$$

Record this design flow rate percentage difference on the VFC Sampler Flow-Check Data Sheet and plot it on the control chart for the field validation of flow rates. When plotting this value, use a different symbol than is normally used for plotting values that are obtained during sampling periods. If the design flow rate percentage difference is less than or equal to $\pm 7\%$, the sampler calibration is acceptable. Those differences exceeding $\pm 7\%$ will require recalibration. Differences exceeding $\pm 10\%$ may invalidate all data obtained subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the orifice transfer standard's certification, and all calculations.

14. Maintenance

Maintenance is defined as a program of positive actions aimed toward preventing failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and provide more complete data acquisition. The general maintenance procedures for HV samplers are outlined in this section. For more complete information on a particular sampler or on laboratory equipment maintenance, refer to the manufacturer's instruction manual for the individual instrument. Maintenance activities for the HV sampler are summarized in Table 4. Records should be maintained for the maintenance schedule of each HV sampler. Files should reflect the history of maintenance, including all replacement parts, suppliers, costs, expenditures, and in inventory of on-hand spare equipment for each sampler. Check sheets should be used to record preventive and/or corrective maintenance activities and the subsequent sampler calibration curve.

14.1 Maintenance Procedures

The HV sampler is comprised of two basic components: the inlet and the flow control system. Because of the differences between sampler models, refer to the manufacturer's instruction manual for specific maintenance guidelines and necessary supplies.

14.2 Recommended Maintenance Schedules

14.2.1 MFC Base. The MFC base is equipped with the following items:

14.2.1.1 Connecting tubing and power lines, which must be checked for crimps, cracks, or obstructions on sample recovery days. Fittings should be inspected periodically for cross-threading and tightness.

14.2.1.2 A filter screen, which should be inspected on sample recovery days for any impacted deposits.

14.2.1.3 Filter cassette gaskets, which need to be inspected each time a cassette is loaded. A worn cassette gasket is characterized on exposed filters by a gradual blending of the boundary between the collected particulate and the filter border.

14.2.1.4 Motor and housing gaskets, which should be checked at 3-month intervals and replaced as necessary.

14.2.1.5 Blower motor brushes, which should be replaced before they become worn to the point that damage may occur. Although motor brushes usually require replacement after 600-1,000 hours of operation, the optimum replacement interval must be determined by experience. A pumice stone can be used against the motor's contacts to ensure high conductivity. Change the brushes according to manufacturer's instructions and perform the operator's field-calibration check as presented in Section 13. If the sampler's indicated flow rate exceeds the manufacturer-specified design-flow-rate range, adjust the sampler before the next run day.

To achieve the best performance, new brushes should be properly seated on the motor's commutator before full voltage is applied to them. After the brushes have been changed, operate the sampler at 50-75% of normal line voltage for approximately 30 min. The motor should return to full performance after an additional 30-45 min at normal line voltage.

[Note: The motors that are used for HV samplers are higher-current versions of the motors that have been used for HV total suspended particulate samplers. The brushes for the two types of motors are different. Make sure that the correct replacement brushes are used for the maintenance of HV samplers. If a motor needs to be replaced, be sure to use the higher-current versions that are needed for HV sampling. When lower-current motors are installed in HV samplers, the flow rate has been found to vary with changes in the line voltage.]

14.2.1.6 A flow controller should be replaced if the flow recorder indicates no flow, low flow, excessive flow, or erratic flow. Minor adjustments can be made to alter sampling flow rates; however, the controller generally cannot be repaired in the field.

[Note: A flow recorder requires very little maintenance, but does deteriorate with age. Difficulty in zeroing the recorder and/or significant differences (i.e., greater than 0.3 m³/min) in average flow rates obtained from consecutive sampling periods usually indicate a faulty recorder. The recorder pens should be replaced every 30 recording days. In dry climates, a more frequent replacement schedule may be required.]

14.2.2 VFC Base. The VFC base is equipped with the following items:

14.2.2.1 Power lines, which must be checked for crimps or cracks on sample recovery days. Fittings should be inspected periodically for cross-threading and tightness.

14.2.2.2 A filter screen at the throat of the choked-flow venturi, which should be inspected on sample recovery days for any impacted deposits.

14.2.2.3 Filter cassette gaskets, which should be checked each time a filter is installed. A worn cassette gasket is characterized on exposed filters by a gradual blending of the boundary between the collected particulates and the filter border.

14.2.2.4 Motor and housing gaskets, which should be checked at 3-month intervals and replaced as necessary.

14.2.2.5 Blower motor brushes, which should be replaced before they become worn to the point that damage may occur. Although motor brushes usually require replacement after 600-1,000 hours of operation, the optimum replacement interval must be determined by experience. A pumice stone can be used against the motor's contacts to ensure high conductivity. Change the brushes according to manufacturer's instructions, and perform the operator's field-calibration check as presented in Section 13. If the sampler's indicated flow rate exceeds the manufacturer-specified design flow-rate range, recalibrate the sampler before the next run day.

To achieve the best performance, new brushes should be seated properly on the motor's commutator before full voltage is applied to them. After the brushes have been changed, operate the sampler at 50-75% of normal line voltage for approximately 30 min. The motor should return to full performance after an additional 30-45 min at normal line voltage.

Caution: Motors that are used for HV PM₁₀ samplers are higher-current versions of the motors that have been used for HV total suspended particulate samplers. The brushes for the two types of motor are different. Make sure that the correct replacement brushes are used for the maintenance of HV PM₁₀ samplers.

14.2.2.6 If a motor needs to be replaced, be sure to use the higher-current versions that are needed for HV PM₁₀ sampling. When lower-current motors are installed in HV PM₁₀ samplers, the flow rate has been found to vary with changes in the line voltage.

14.3 Refurbishment of HV Samplers

If operated in the field for extended periods, HV PM₁₀ samplers may require major repairs or complete refurbishment. If so, refer to the manufacturer's instrument manual before work is undertaken. A sampler that has undergone major repairs or refurbishment must be leak-checked and calibrated prior to sample collection.

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TABLE 1. EXAMPLE OF BASIC CHARACTERISTICS OF SOME COMMON FILTER MATERIAL

<p>QUARTZ FIBER FILTER (Glass Spun with Organic Binder)</p>
<p>! Whatman QMA Filter</p> <ul style="list-style-type: none"> ! Maximum temperature of up to 540EC ! High Collection Efficiency ! Non-hydroscopic ! Good for Corrosive Atmospheres ! Fragile ! Lowest background metals content
<p>CELLULOSE FIBER FILTER (Cellulose Pulp)</p>
<p>! Whatman # 41/MSA "s"</p> <ul style="list-style-type: none"> ! Low Ash ! Maximum Temperature of 150EC ! High Affinity for Water ! Enhanced Artifact Formation for SO₄ and NO₃ ! Good for X-Ray/Neutron Activation Analysis ! Low Metal Content
<p>MEMBRANE FILTER (Dry Gel of Cellulose Esters)</p>
<p>! Whatman #41</p> <ul style="list-style-type: none"> ! Fragile, Therefore Requires Support Pad ! High Pressure Drop ! Low Residue when Ashed

TABLE 2. EXAMPLE OF SUMMARY OF USEFUL FILTER PROPERTIES

Filter and Filter Composition	Density mg/cm ²	pH	Filter Efficiency %
Teflon® (Membrane) (CF ₂) _n (2µm Pore Size)	0.5	Neutral	99.95
Cellulose (Whatman 41) (C ₆ H ₁₀ O ₅) _n	8.7	Neutral (Reacts with HNO ₃)	58% at 0.3 µm
Glass Fiber (Whatman GF/C)	5.16	Basic pH - 9	99.0
"Quartz" Gelman Microquartz	6.51	pH - 7	98.5
Polycarbonate (Nuclepore) C ₁₅ H ₁₄ + CO ₃ (0.3µm Pore Size)	0.8	Neutral	93.9
Cellulose Acetate/Nitrate Millipore (C ₉ H ₁₃ O ₇) _n (1.21 µm Pore Size)	5.0	Neutral (Reacts with HNO ₃)	99.6

TABLE 3. EXAMPLE OF MINIMUM SAMPLER SITING CRITERIA

Scale	Height above ground, meters	Distance from supporting structure, meters		Other spacing criteria
		Vertical	Horizontal ^a	
Micro	2 to 7	> 2	> 2	1. Should be > 20 meters from trees. 2. Distance from sampler to obstacle, such buildings, must be twice the height and the obstacle protrudes above the sampler. 3. Must have unrestricted airflow 270 degrees around the sampler inlet. 4. No furnace or incineration flues should be nearby. ^b 5. Spacing from roads varies with traffic (see 40 CFR 58, Appendix E). 6. Sampler inlet is at least 2 m but not greater than 4 m from any collocated PM ₁₀ sampler. (See 40 CFR 58, Appendix E.)
Middle, neighborhood, urban, and regional scale	2 to 15	> 2	> 2	

^aWhen inlet is located on rooftop, this separation distance is in reference to walls, parapets, or penthouses located on the roof.

^bDistance depends on the height of furnace or incineration flues, type of fuel or waste burned, and quality of fuel (sulfur, ash, or lead content). This is to avoid undue influences from minor pollutant sources.

TABLE 4. EXAMPLE OF ROUTINE MAINTENANCE ACTIVITIES
 FOR SAMPLERS

Equipment	Frequency and/or method	Acceptance limits	Action if requirements are not met
Sampler inlet	Dismantle and clean at manufacturer-specified intervals	No obvious particulate deposits or damage	Clean, replace damaged equipment before sampling
Sampler base			
Power lines	Check for crimps or cracks	No obvious damage	Replace as necessary
Filter screen and throat	Visually check on sample-recovery days	No obvious deposits; clean with wire brush	Clean
Gaskets	At 3-mo intervals, inspect all gaskets in the sampler	No leaks; no compression damage evident	Replace as necessary
Brushes	Replace after 600-1,000 h of operation	Stable flow rate	Replace as necessary
Motor	Replace if needed	Correct model must be used	Obtain correct model
Flow controller	Check when flowrate changes are evident	Stable flow rate throughout sample run	Replace or repair if possible
Recording device	Inspection with experiencing difficulty in zeroing, or when large changes in flow rates occur	Recorder stays zeroed; chart advances; pen inks	Replace or repair if possible
Tubing, fittings	Visually inspect on sample-recovery days	No crimps, cracks, or obstructions; no crossthreading	Replace as necessary.

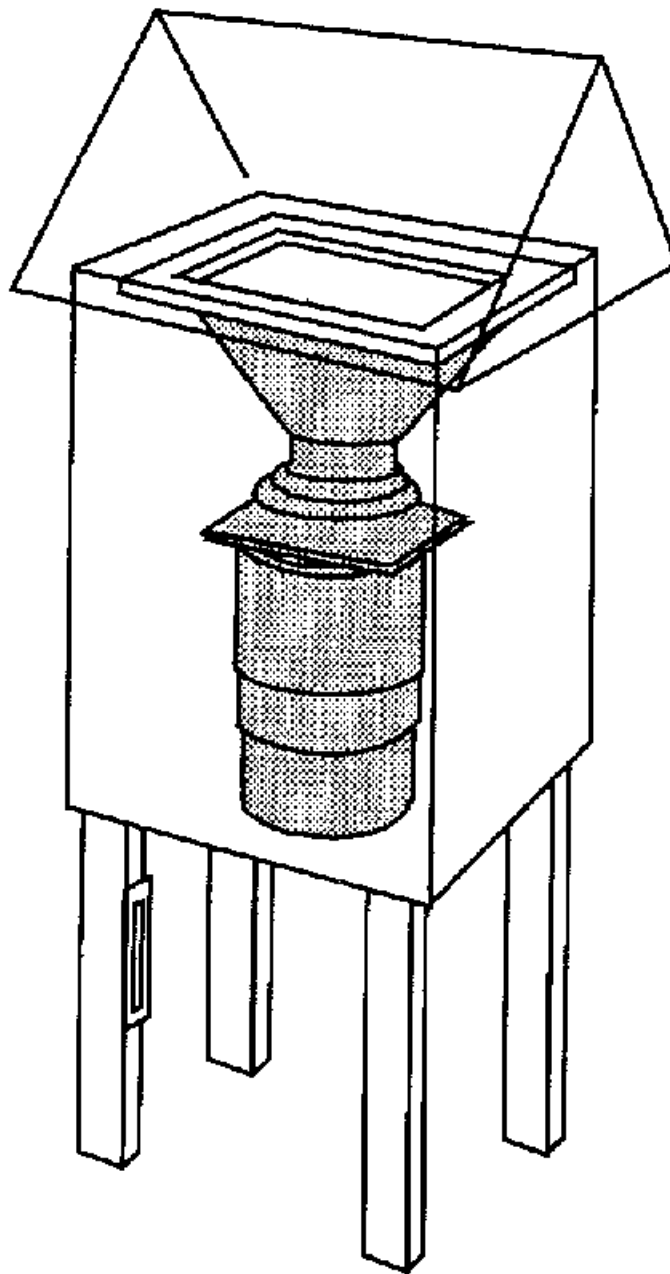


Figure 1. High-volume sampler with shelter.

Hi Volume Sampler in Shelter

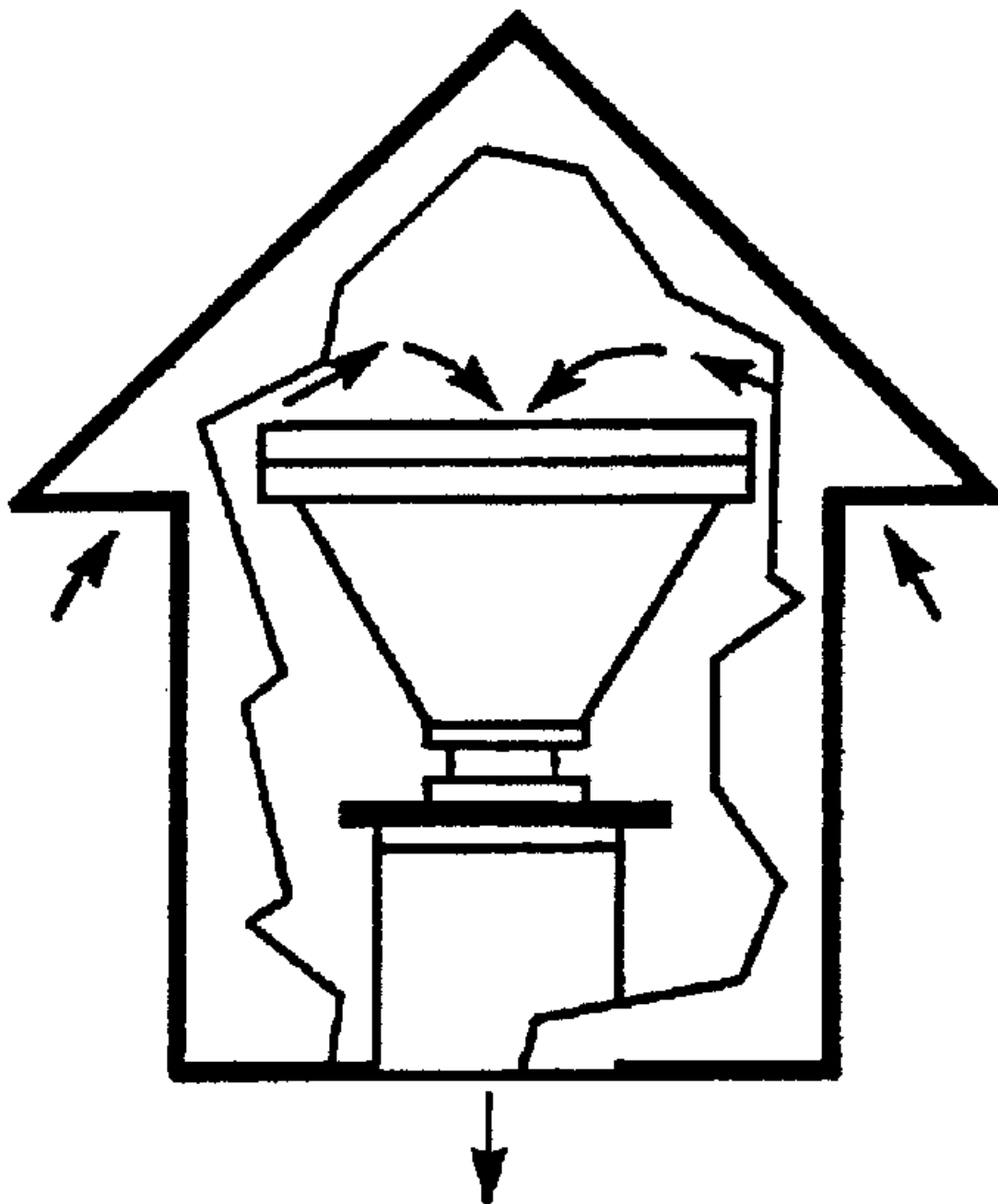


Figure 2. Inlet to EPA approved high volume sampler.

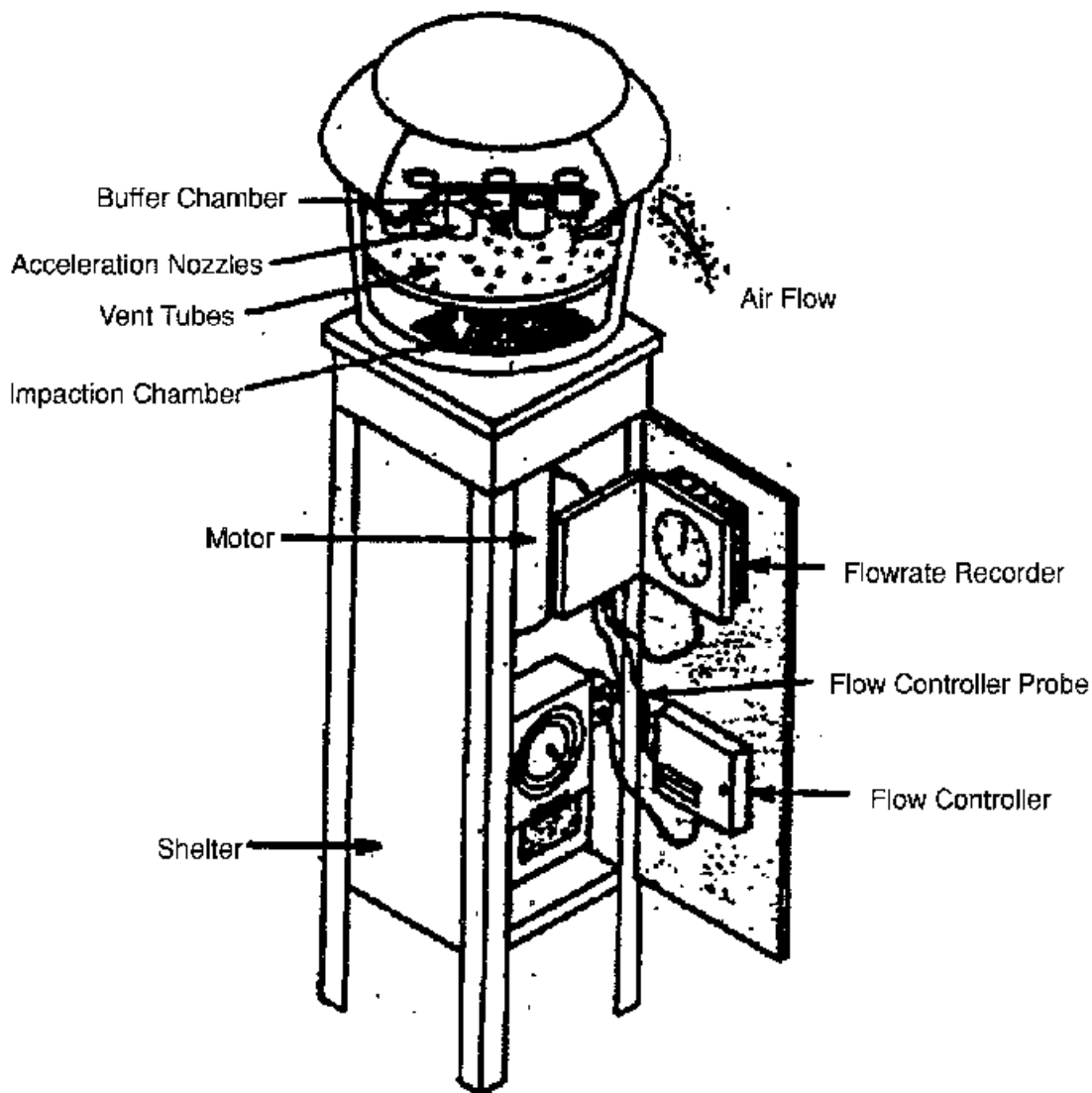


Figure 3. High-volume sampler with mass flow controller and impactor design size select inlet.

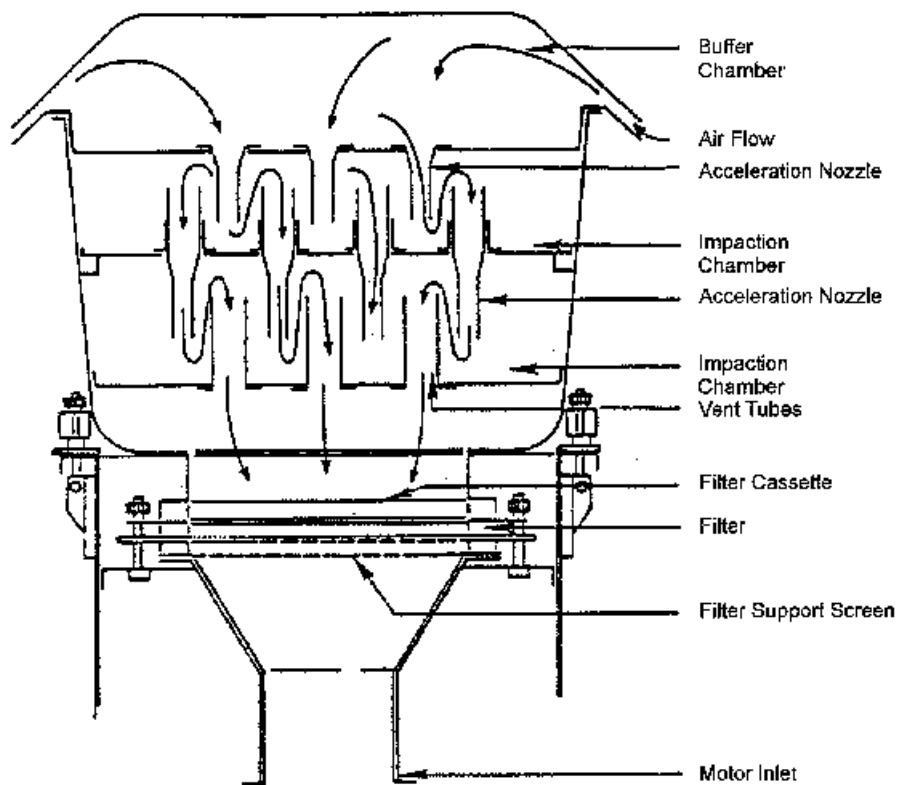


Figure 4. Schematic diagram of an impaction inlet for size select sampling for particulate matter.

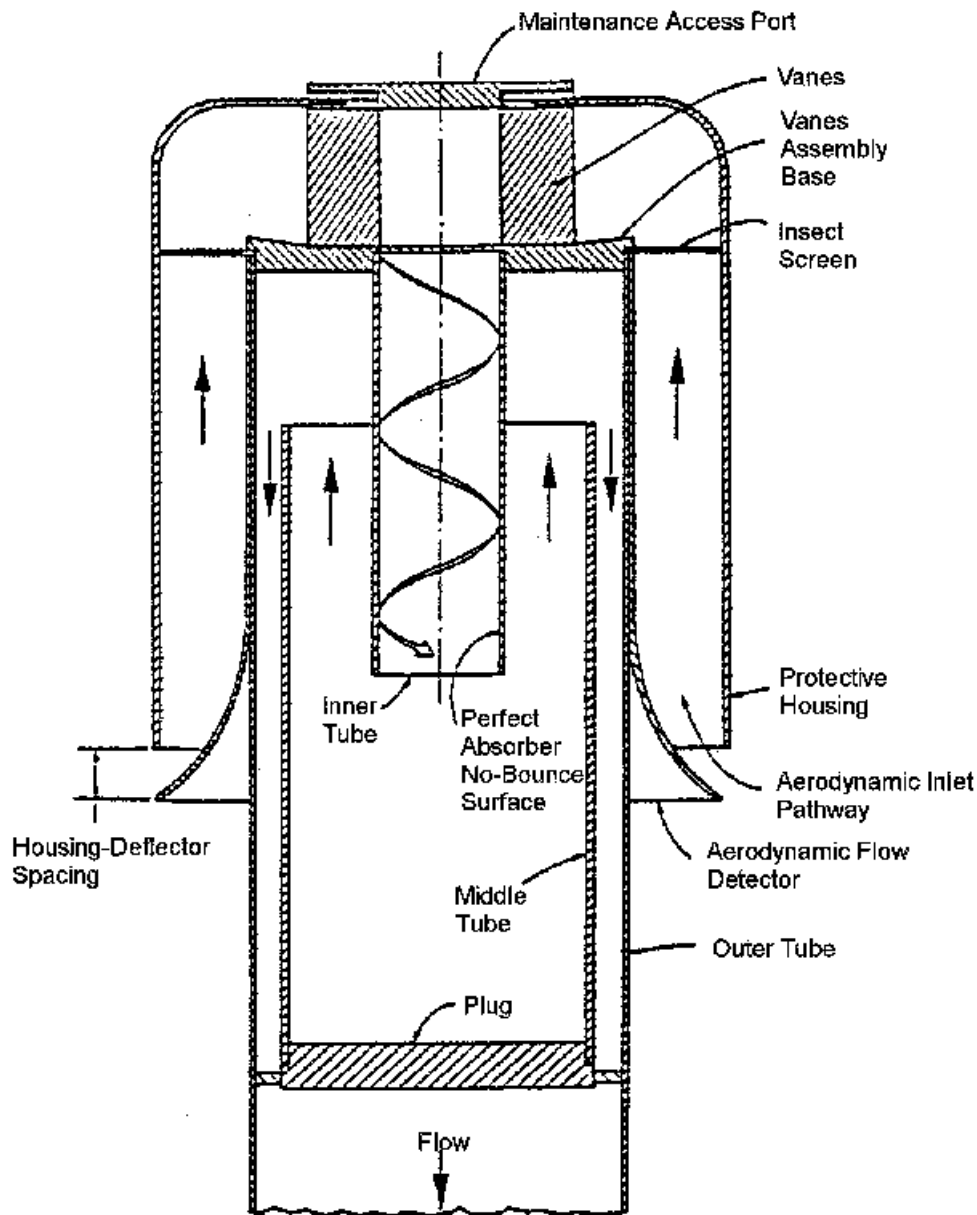


Figure 5. Schematic diagram of a cyclonic inlet for size select sampling for particulate matter.

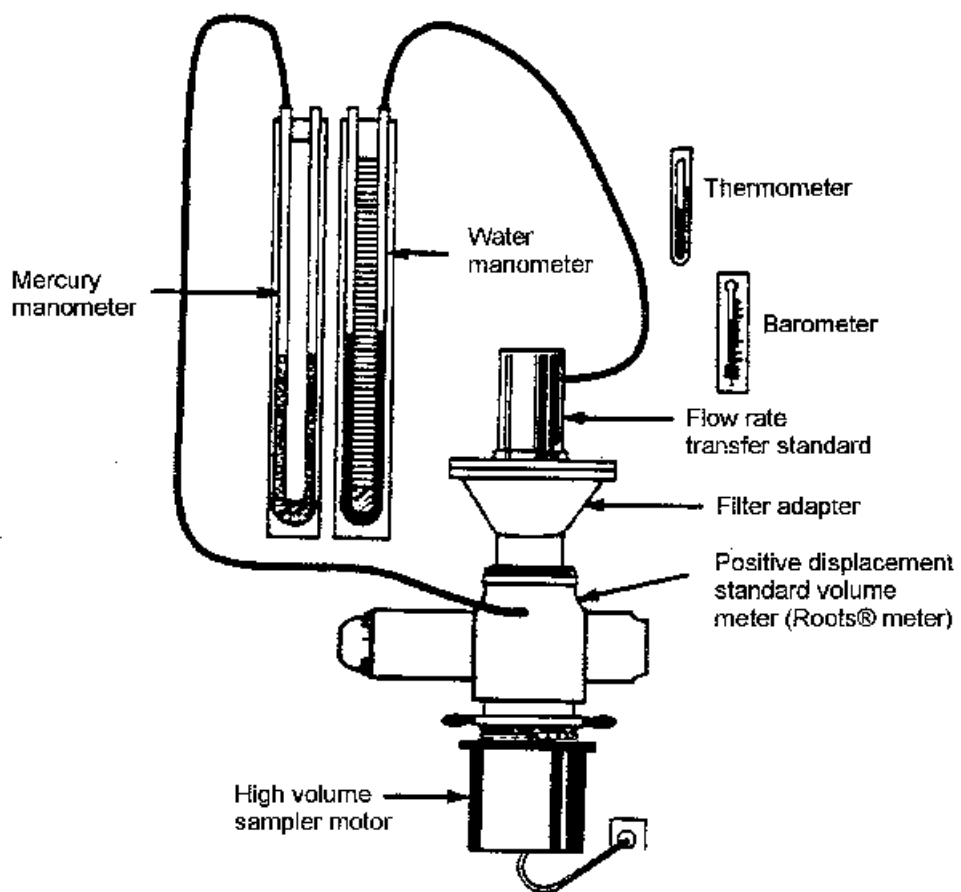


Figure 6. Flow rate transfer standard calibration setup.

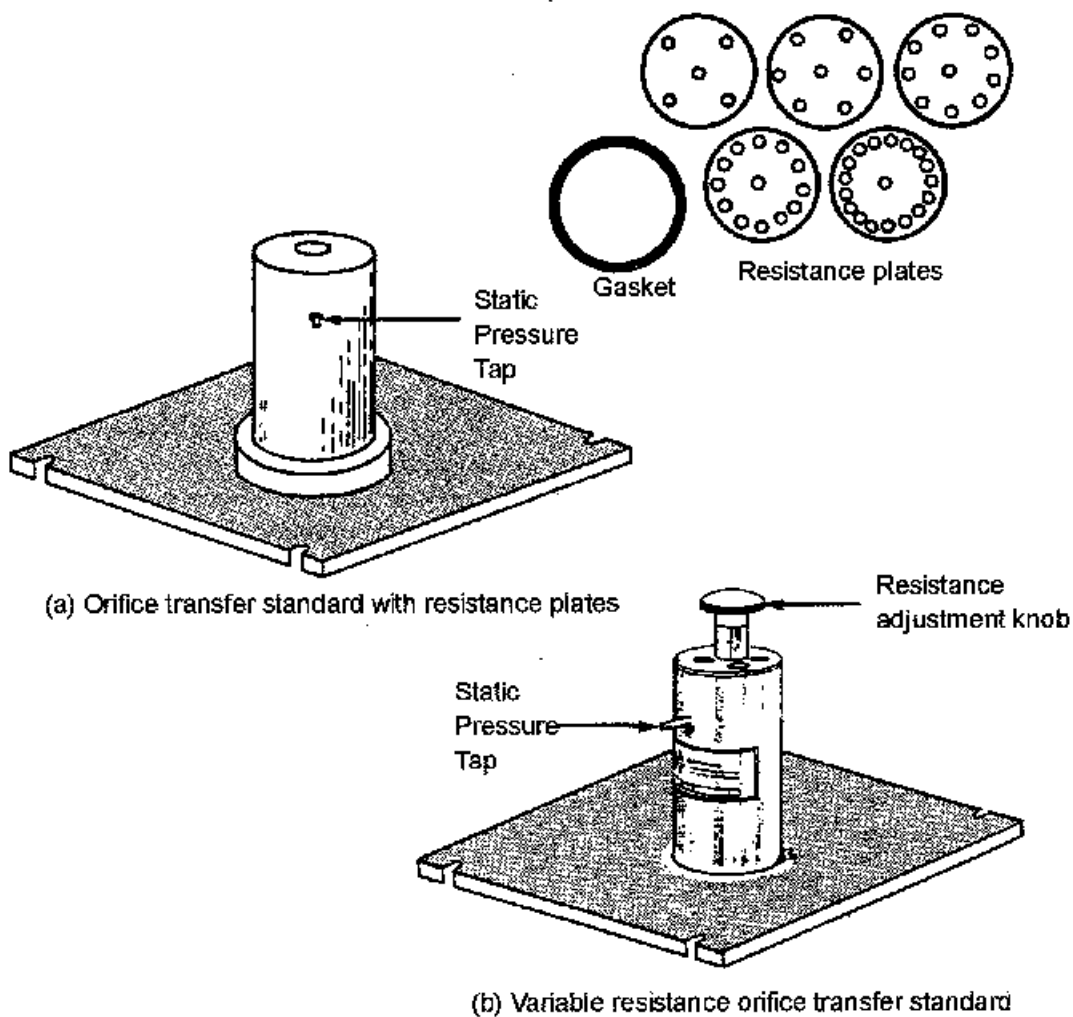


Figure 7. Typical orifice-type flow rate transfer standards.

ORIFICE TRANSFER STANDARD CERTIFICATION WORKSHEET						
Date:		Roots meter S/N:		Ta:		K
Operator:		Orifice S/N:		Pa:		mm Hg
Plate or Volts AC	Initial Volume	Final Volume	Δ Vol.	ΔTime (min)	ΔHg (mm)	ΔH ₂ O (in.)
DATA TABULATION						
Vstd	(x-axis) Qstd	(y-axis) [ΔH ₂ O (Pa/Ta)] ^{1/2}	Va	(x-axis) Qa	(y-axis) [ΔH ₂ O (Ta/Pa)] ^{1/2}	
	m =			m =		
	b =			b =		
	r =			r =		
CALCULATIONS						
Vstd = Δ Vol [(Pa - ΔHg)/760] (298/Ta) Qstd = Vstd/ΔTime y = mx + b			Va = ΔVol [(Pa - ΔHg)/Pa] Qa = Va/ΔTime y = mx + b			
For subsequent flow rate calculations:						
Qstd = {[ΔH ₂ O (Pa/Ta)] ^{1/2} - b} {1/m}			Qa = {[ΔH ₂ O (Ta/Pa)] ^{1/2} - b} {1/m}			
NOTE: For PM10 monitoring, a calibration curve corrected to standard conditions is optional.						

Figure 8. Example orifice transfer standard certification worksheet.

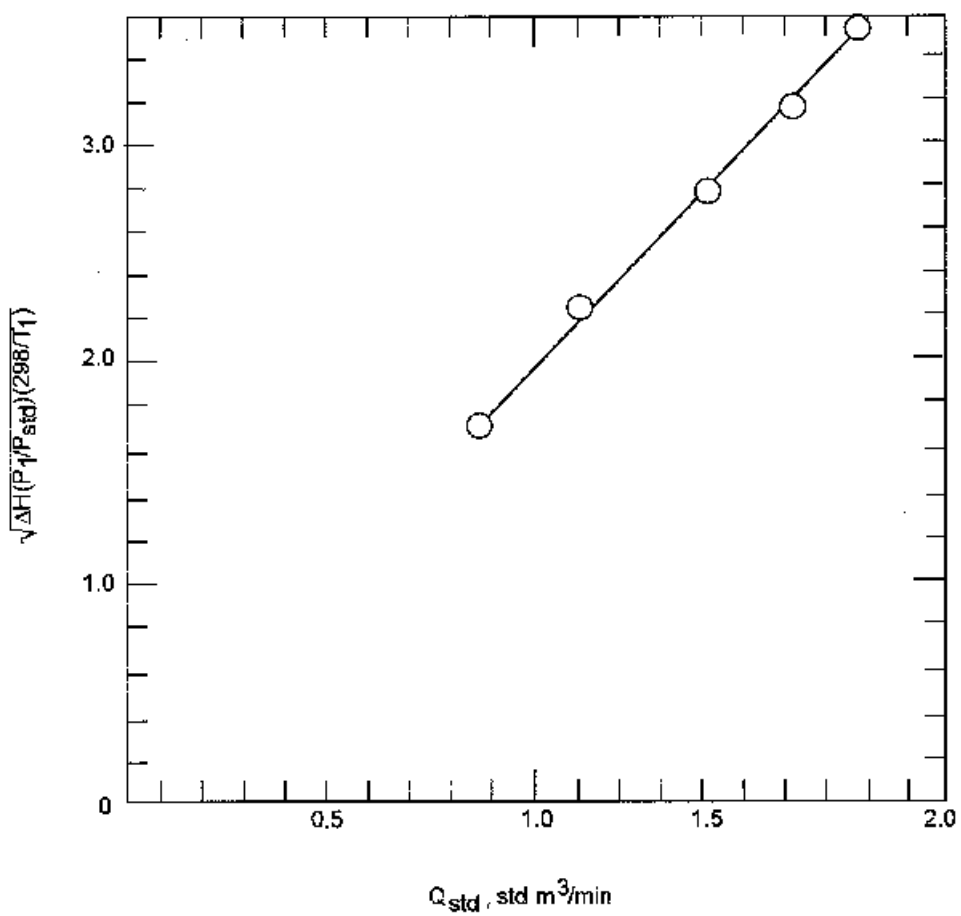


Figure 9. Typical calibration curve for a flow rate transfer standard.

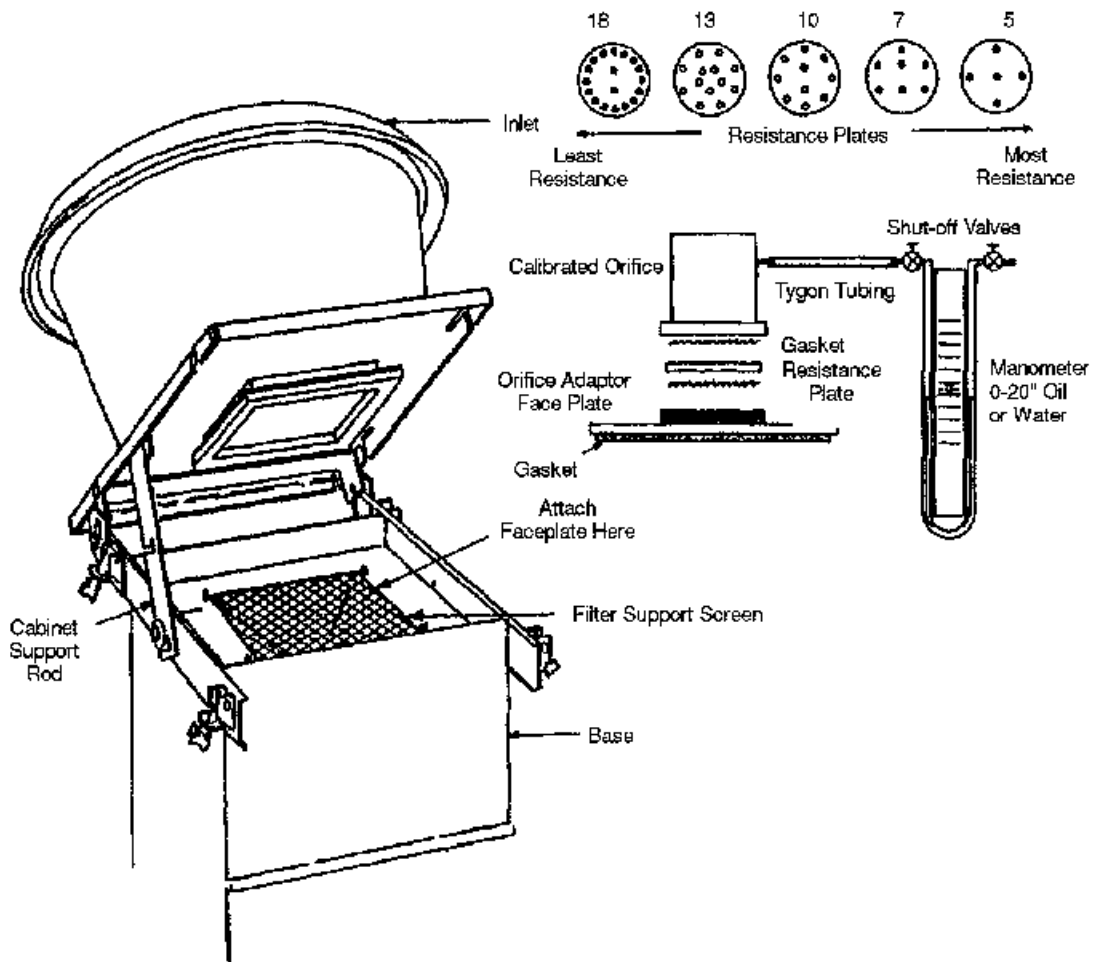


Figure 10. Typical calibration set-up for a mass flow controller (MFC).

MFC SAMPLER CALIBRATION DATA SHEET

Station Location _____ Date _____ Time _____

Sampler Model _____ S/N _____ Operator _____

Pa _____ mm Hg. Ta _____ °C _____ K. Unusual conditions: _____

Ps _____ mm Hg. Ts _____ °C _____ K. (*seasonal average Ta and Pa)

Orifice S/N _____ Orifice Calibration Date _____

Orifice calibration relationship: m = _____ b = _____ r = _____

Plate Number	Total ΔH ₂ O (in.)	X-Axis = Qa (orifice) flow rate ^a (m ³ /min)	Sampler ΔPex (in. H ₂ O) [or I for flow recorders]	Y-Axis = Sampler ΔPext ^b [or It for flow recorders] ^c

^aQa = $\{[(\Delta H_2O)(Ta/Pa)]^{1/2} - b\} \{1/m\}$
^bΔPext = $\{\Delta Pex(Ta + 30)/Pa\}^{1/2}$
^cIt = $\{I\} [(Ta + 30)/Pa]^{1/2}$ if a flow recorder is used

Sampler Calibration Relationship (Qa on x-axis; ΔPext or It on y-axis):
 ΔPext = m[Qa (Orifice)] + b or It = m[Qa(Orifice)] + b
 m = _____ b = _____ r = _____

For subsequent calculation of sampler flow rate:
 $\overline{Qa} = \{[(\text{mean } \Delta Pex (Tav + 30)/Pav)]^{1/2} - b\} \{1/m\}$
 or $\overline{Qa} = \{[\text{mean } I(Tav + 30)/Pav]^{1/2} - b\} \{1/m\}$

Set point flow rate (SFR) _____ Sampler set point (SSP) _____
 SFR = 1.13 (Ps/Pa) (Ts/Ts) SSP = $[Pa(Ta + 30)] \{m(SFR) + b\}^2$
 or SSP = $[Pa(Ta + 30)]^{1/2} \{m(SFR) + b\}$ for flow recorders

Figure 11. Example MFC sampler calibration data sheet.

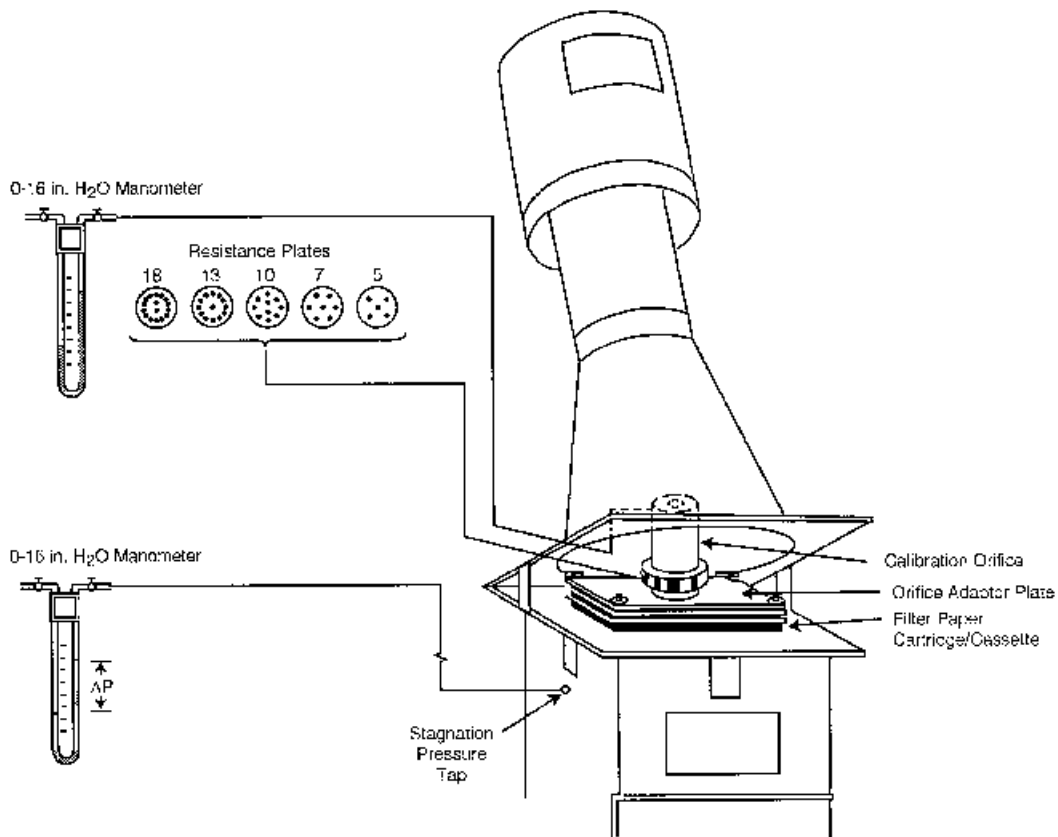


Figure 12. Calibration of a typical volumetric flow controller (VFC).

VFC SAMPLER CALIBRATION DATA SHEET																								
Station Location _____		Date _____		Time _____																				
Sampler Model _____		S/N _____		Operator _____																				
Pa _____ mm Hg.		Ta _____ °C		K, Unusual Conditions _____																				
Orifice S/N _____		Orifice Calibration Date _____																						
Orifice Calibration Relationship: $m =$ _____ $b =$ _____ $r =$ _____																								
Plate No.	ΔH_2O (in.)	ΔP_{stg} (mm Hg) ^a	$P1 = Pa - \Delta P_{stg}$ (mm Hg)	$P1/Pa$ (mm Hg)	Q_a (orifice) flow rate ^b (m ³ /min)	Q_s (orifice) $[Ta]^{1/2}$																		
Operational Flow Rate																								
^a mm Hg = 25.4 (in. H ₂ O/13.6) ^b Q_a (orifice) = $1/m \{[(\Delta H_2O) (Ta/Pa)]^{1/2} - b\}$ ^c % Difference = $\left[\frac{Q_a \text{ (sampler)} - Q_a \text{ (orifice)}}{Q_a \text{ (orifice)}} \right] \{100\}$					<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th>Q_s (Orifice)</th> <th>Q_a (sampler) (Lookup Table)</th> <th>% Difference^c</th> </tr> </thead> <tbody> <tr><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td></tr> </tbody> </table>		Q_s (Orifice)	Q_a (sampler) (Lookup Table)	% Difference ^c															
Q_s (Orifice)	Q_a (sampler) (Lookup Table)	% Difference ^c																						
Sampler Calibration Relationship <input type="checkbox"/> Lookup Table Validated (i.e., % difference < 4) <input type="checkbox"/> New calibration relationship: $X = \frac{Q_a \text{ (orifice)}}{[Ta]^{1/2}}, Y = (P1/Pa)$ $m =$ _____ $b =$ _____ $r =$ _____ For subsequent calculation of sampler flow rate: $Q_a = \{[P1/Pa - b][Ta]^{1/2}\} \{1/m\}$ Operational Flow Rate _____ m ³ /min																								

Figure 13. Example VFC sampler calibration data sheet.

MFC SAMPLER FIELD DATA SHEET	
Station Location _____	Date _____ SAROAD# _____
Sampler Model _____	S/N _____
Filter ID No. _____	Pav _____ mm Hg. Tav _____ °C _____ K
Sampler Manometer Readings	Flow Recorder Readings
Initial ΔPex _____ in. H ₂ O	Mean I _____
Final ΔPex _____ in. H ₂ O	
Mean ΔPex _____ in. H ₂ O	
Sampler Calibration Relationship: m = _____ b = _____ r = _____	
\bar{Q}_a _____ m ³ /min	Elapsed Time _____ min
$\bar{Q}_a = \{[\text{mean } \Delta P_{ex} (T_{av} + 30)/P_{av}]^{1/2} - b\} \{1/m\}$	
$\bar{Q}_a = \{\text{mean } I [(T_{av} + 30)/P_{av}]^{1/2} - b\} \{1/m\}$ for flow recorders	
Operator _____	
Comments: _____	

Laboratory Calculations:	
\bar{Q}_{std} _____ std m ³ /min	Gross weight (Wg) _____ g
$\bar{Q}_{std} = \bar{Q}_a (P_{av}/P_{std}) (T_{std}/T_{av})$	Tare weight (Wt) _____ g
V_{std} _____ std m ³	Net Weight (Wn) _____ g
$V_{std} = (\bar{Q}_{std}) (\text{elapsed time})$	PM10 Concentration _____ μg/std m ³
	PM10 Concentration = (Wn) (10 ⁶)/Vstd

Figure 14. Example MFC sampler field data sheet.

VFC SAMPLER FIELD DATA SHEET	
Station Location _____	Date _____ SAROAD# _____
Sampler Model _____ S/N _____	
Filter ID No. _____ P _{av} _____ mm Hg, T _{av} _____ °C _____ K	
Relative Stagnation Pressure Readings	Absolute Stagnation Pressure
Initial ΔP _{stg} _____ mm Hg	P ₁ = _____ mm Hg
Final ΔP _{stg} _____ mm Hg	P ₁ = P _{av} - Average ΔP _{stg}
Average ΔP _{stg} = _____ mm Hg	
Average Stagnation Pressure Ratio (P ₁ /P _{av}) _____	
Average Flowrate (Q _a)* _____ m ³ /min	Elapsed Time _____ min
*Obtained from manufacturer's lookup table (or from alternate calibration relationship)	
Operator _____	
Comments: _____	

Laboratory Calculations:	
Q _{std} _____ Std m ³ /min	Gross Weight (W _g) _____ g
Q _{std} = Q _a (P _{av} /P _{std}) (T _{std} /T _{av})	Tare Weight (W _t) _____ g
V _{std} _____ std m ³	Net Weight (W _n) _____ g
V _{std} = (Q _{std}) (Elapsed Time)	PM10 Concentration _____ μg/std m ³
	PM10 Concentration = (W _n) (10 ⁶)/V _{std}

Figure 15. Example VFC sampler field data sheet.

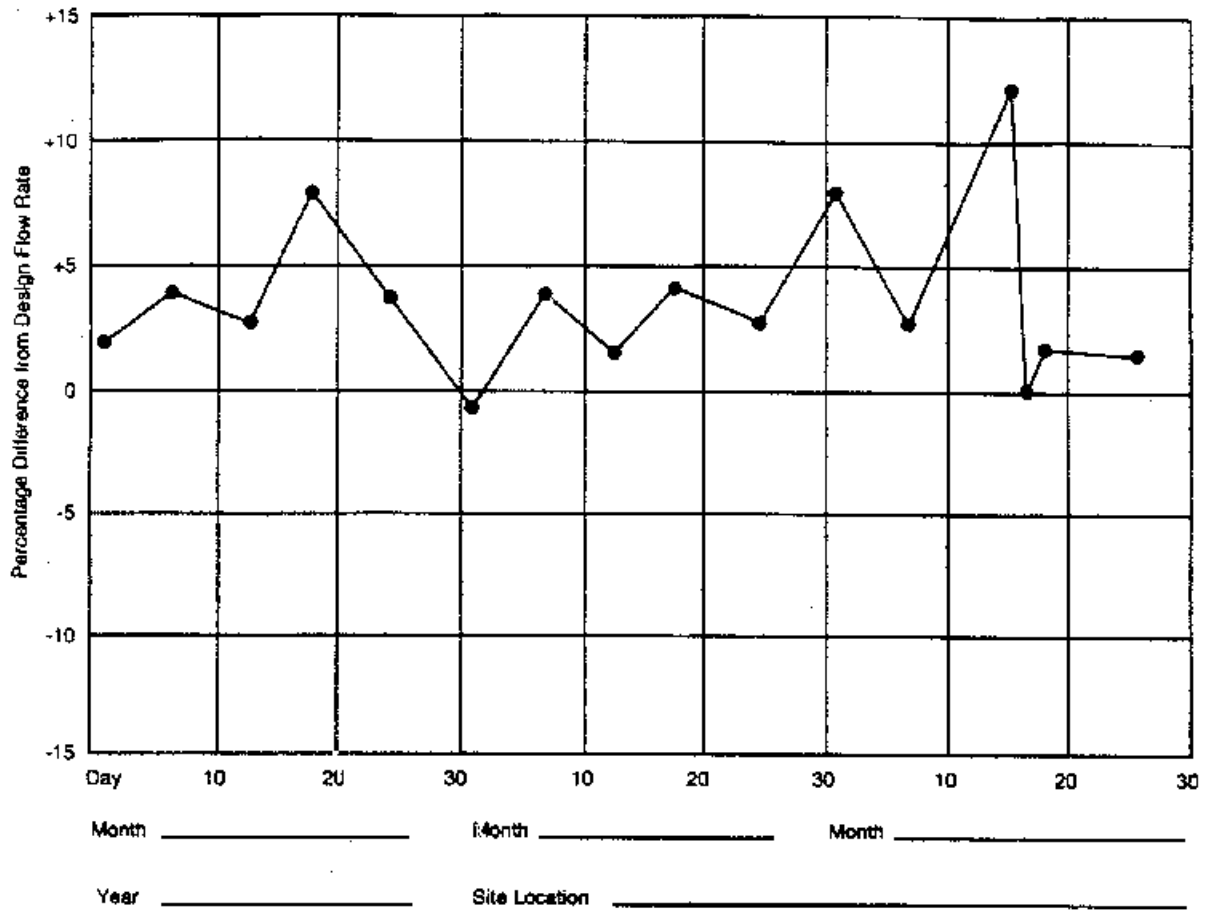


Figure 16. Field data control chart.

ATTACHMENT L-B

USEPA COMPENDIUM METHOD IO-3.1

**Compendium of Methods
for the Determination of
Inorganic Compounds
in Ambient Air**

Compendium Method IO-3.1

**SELECTION, PREPARATION
AND EXTRACTION OF
FILTER MATERIAL**

Center for Environmental Research Information
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, OH 45268

June 1999

Method IO-3.1

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- Justice A. Manning, U.S. EPA, ORD, Cincinnati, OH
- William A. McClenny, U.S. EPA, NERL, RTP, NC
- Frank F. McElroy, U.S. EPA, NERL, RTP, NC
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

This Method is the result of the efforts of many individuals. Gratitude goes to each person involved in the preparation and review of this methodology.

Author(s)

- Avie Mainey, Midwest Research Institute, Kansas City, MO
- William T. "Jerry" Winberry, Jr., EnviroTech Solutions, Cary, NC

Peer Reviewers

- David Brant, National Research Center for Coal and Energy, Morgantown, WV
- John Glass, SC Department of Health and Environmental Control, Columbia, SC
- Jim Cheney, U.S. Army Corps of Engineers, Omaha, NE
- Dewayne Ehman, Texas Natural Resource Conservation Committee, Austin, TX
- Gary Wester, Midwest Research Institute, Kansas City, MO
- Lauren Drees, U.S. EPA, NRMRL, Cincinnati, OH

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Method IO-3.1
Selection, Preparation and Extraction of
Filter Material

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Chapter IO-3
CHEMICAL SPECIES ANALYSIS
OF
FILTER-COLLECTED SUSPENDED PARTICULATE MATTER (SPM)

Method IO-3.1
SELECTION, PREPARATION AND EXTRACTION OF
FILTER MATERIAL

1. Scope

1.1 This methodology consists of (1) filter media selection, (2) numbering and pre-field tare weighing of filters, (3) post-field final weighing of filters, (4) microwave or hot acid extraction, and (5) analysis for metal analysis by ICP, FAA, ICP/MS or GFAA.

1.2 Pre-field filters are conditioned in a room of constant humidity and temperature and are gravimetrically tared. After air samples have been collected, the filters are returned to the laboratory and conditioned as before and weighed. The final filter weight minus the tare weight is calculated. The procedure for the weighing of filters is based on 40 CFR 50, Appendix B, entitled "*Reference Method for the Determination of Suspended Matter in the Atmosphere (High-Volume Method)*."

1.3 After the post-field filter final weights have been obtained, the filter is subsampled by cutting a filter strip consisting of one-ninth of the overall filter and digested using a microwave or hot acid extraction technique; these extracts are then analyzed by one of many analytical techniques. The results are multiplied by a factor of 9 to obtain the actual total μg of each metal found on the entire 8" x 10" filter. Based upon the analysis of a blank filter, background metal concentration may be subtracted from the total metal concentration to get a net value. Therefore, the analytical results represent the total μg found on the 8" x 10" filter but do not represent the volume of air sampled.

1.4 Sectioning the filter for extraction is based on 40CFR50, Appendix B entitled "*Determination of Lead in Suspended Particle Matter Collected From Ambient Air*." The procedure for the microwave extraction is based on a method developed by EPA entitled *Microwave Extraction of Glass-Fiber Filters*, as identified in Section 2.2. This procedure has been modified for extracting quartz fiber filters.

2. Applicable Documents

2.1 ASTM Documents

- D4096 *Application of the High Volume Sample Method for Collection and Mass Determination of Airborne Particle Matter.*
- D1356 *Definition of Terms Related to Atmospheric Sampling and Analysis.*
- D1357 *Practice for Planning the Sampling of the Ambient Atmosphere.*
- D2986 *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test.*

2.2 Other Documents

- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: A Field Guide for Environmental Quality Assurance*, EPA-600/R-94/038a.
- U. S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition)*, EPA-600/R-94/038b.
- *Reference Method for the Determination of Particulate Matter in the Atmosphere*, Code of Federal Regulations (40 CFR 50, Appendix J).
- *Reference Method for the Determination of Suspended Particulates in the Atmosphere (High Volume Method)*, Code of Federal Regulations (40 CFR 50, Appendix B).
- *Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air*, Federal Register 43 (194): 46258-46261.
- U. S. Environmental Protection Agency, *Microwave Extraction of Glass Fiber Filters*, Method Research and Development Division, RTP, NC 1989.

3. Apparatus

3.1 Equipment For Gravimetric Analysis

3.1.1 Controlled Temperature. Temperature between 15 and 30EC with less than ± 2 EC variation during equilibration period.

3.1.2 Controlled Humidity. Less than 50% relative humidity, constant within $\pm 5\%$

3.1.3 Analytical Balance. Sensitive to 0.1 mg, with weighing chamber designed to accept an unfolded 20.3 x 25.4 cm (8" x 10") filter.

3.1.4 Area Light Source. Similar to X-ray film viewer to backlight filters for visual inspection.

3.1.5 Numbering Device. Capable of printing identification numbers on the filters before they are placed in the filter conditioning environment if not numbered by the supplier.

3.1.6 Hygrothermograph. Capable of recording temperature and relative humidity in the weighing room.

3.2 Microwave Digestion Apparatus and Materials

3.2.1 Microwave Digestive System and Capping Station. With programmable power settings up to 600 watts, best source.

[Note: Commercial kitchen or home-use microwave should NOT be used for digesting samples. The oven cavity must be corrosion resistant and well ventilated. All electronics must be protected against corrosion for safe operation.]

3.2.2 PFA Teflon® Digestion Vessels. Capable of withstanding pressures of up to 120 psi. Pressure vessels capable of controlled pressure relief at pressures exceeding 120 psi (60-120 mL capacity), best source.

3.2.3 Teflon® PFA Overflow Vessel. Double ported (60-120 mL capacity), best source.

3.2.4 Rotating Table. Uniform exposure of samples within the oven.

3.2.5 Volumetric Glassware. 50-100 mL capacity (Class A borosilicate).

3.2.6 Bottles, Linear Polyethylene or Polypropylene with Leakproof Caps, for Storing Samples. Teflon® bottles for storing multielement standards (500 mL, 125 mL, and 30 mL), best source.

- 3.2.7 Centrifuge Tubes.** Oak Ridge polysulfone tubes with screw caps of polypropylene, 30 mL.
- 3.2.8 Nylon or Teflon® 0.45 µm Syringe Filters.** Acrodisc® No. 4438 or equivalent and syringes for rapid nonmetals contributing filtering.
- 3.2.9 Sterile Polypropylene Tubes with Screw Caps of Polypropylene, 15 mL Capacity.** Best source.
- 3.2.10 Pipette.** Automatic dispensing with an accuracy of setting 0.1 mL or better and repeatability of 20 FL, Grumman Automatic Dispensing Pipette, Model ADP-30DT or equivalent.
- 3.2.11 Particle Mask.** 3M, No. 8500, to be worn while cutting and handling glass-fiber filters.
- 3.2.12 Template.** Aid in sectioning the glass fiber filter. Federal Register 43 (194): 46258-46261.
- 3.2.13 Pizza Cutter, Thin Wheel.** Clean razor blade (< 1 mm).
- 3.2.14 Vortex Mixer.** VWR2 variable speed or equivalent.
- 3.2.15 Hydrochloric Acid.** Baker Instra-Analyzed, concentrated (sp.gr.1.19) or equivalent, for preparing samples.
- 3.2.16 Nitric Acid.** Baker Instra-Analyzed, concentrated (sp.gr.1.41) or equivalent, for preparing samples.
- 3.2.17 ASTM Type I Water.** ASTM D193.
- 3.2.18 Extraction Solution (5.55% HNO₃/16.75% HCl).** Prepare by adding in ~ 500 mL of Type I DI water, 55.5 mL of concentrated HNO₃, and 167.5 mL of concentrated HCl, dilute to one liter with Type I DI water.

3.3 Hot Acid Extraction Apparatus and Materials

3.3.1 Thermolyne Model 2200 Hot-Plate or Equivalent.

[Note: Temperature of the extracts may be monitored by the use of a beaker containing a thermometer and similar reagents as the samples.]

- 3.3.2 Volumetric Glassware.** 50-100 mL capacity (Class A borosilicate).
- 3.3.3 Bottles, Linear Polyethylene or Polypropylene with Leakproof Caps, for Storing Samples.** Teflon® bottles for storing multielement standards (500 mL, 125 mL, and 30 mL).
- 3.3.4 Centrifuge Tubes.** Polypropylene or Oak Ridge polysulfone tubes with screw caps of polypropylene, 30 mL (Nalgene 3119-0050/3115-0030 or equivalent).
- 3.3.5 Nylon or Teflon® 0.45 µm Syringe Filters.** Acrodisc® No. 4438 or equivalent and syringes for rapid nonmetals contributing filtering.
- 3.3.6 Sterile Polypropylene Tubes with Screw Caps of Polypropylene, 15 mL capacity.** Falcon Model No. 2099 or equivalent.
- 3.3.7 Pipette.** Automatic dispensing with an accuracy of setting 0.1 mL or better and repeatability of 20 FL. (Grumman Automatic Dispensing Pipette, Model ADP-30DT or equivalent).
- 3.3.8 Particle Mask.** 3M, No. 8500. To be worn while cutting and handling glass-fiber filters.
- 3.3.9 Vortex Mixer.** VWR2 variable speed or equivalent.
- 3.3.10 Hydrochloric Acid.** Baker Instra-Analyzed, concentrated (36.5%-38%/12.3 M) or equivalent, for preparing samples.
- 3.3.11 Nitric Acid.** Baker Instra-Analyzed, concentrated (70% 16M) or equivalent, for preparing samples.
- 3.3.12 ASTM Type I Water.** ASTM D193.

4. Filter Medium Selection

4.1 Introduction

4.1.1 In general, the filter medium depends on the purpose of the test. For any given standard test method, the appropriate medium will be specified. However, it is important to be aware of certain filter characteristics that can affect selection and use.

4.1.2 Selecting a filtration substrate for time-integrated SPM monitoring must be made with some knowledge of the expected characteristics and a pre-determined analytical protocol. For any given standard test method, the appropriate medium will normally be specified.

4.1.3 In high-volume sampling, four types of filter material to capture SPM are commonly used. They include cellulose fiber, quartz/glass fiber, mixed fiber, and membrane filter types. Selecting a filter depends upon variables such as background metal content, artifact formation, and affinity for moisture. The basic characteristics of the types of filter material used in high volume are outlined in Table 1 sampling. Useful filter properties are described in Table 2. Several characteristics are important in the selection of filter media. They are:

- **Particle Sampling Efficiency.** Filters should remove more than 99% of SPM drawn through them, regardless of particle size or flow rates.
- **Mechanical Stability.** Filters should be strong enough to minimize leaks during sampling and wear during handling.
- **Chemical Stability.** Filters should not chemically react with the trapped SPM.
- **Temperature Stability.** Filters should retain their porosity and structure during sampling.
- **Blank Correction.** Filters should not contain high concentrations of target compound analytes.

Quartz fiber filter medium is most widely used for determining mass loading. Weight stability with respect to moisture is an attractive feature. Quartz fiber filters provide high efficiency and collect airborne particles of practically every size and description. Typical characteristics of quartz fiber filters are (1) a fiber content of high purity quartz, (2) a binder of below 5% (zero for binderless types), (3) a thickness of approximately 0.5 mm, (4) a surface with no pinholes, and (5) an allowance of no more than 0.05% of smoke particles to pass through the filter at a pressure of 100mm of water with a flow rate of 8.53 m/min (28 ft/min), as determined by ASTM-D2986, *Method for Evaluation of Air Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test*.

Particulate matter collected on quartz fiber filters can be analyzed for many constituents. If chemical analysis is anticipated, binderless filters should be used. Glass is a commercial product generally containing test-contaminating materials; therefore, appropriate background corrections should be made. Background concentration of various metals associated with different grades of quartz fiber filters are documented in Table 3.

4.1.4 Silica fiber filters are used when it may be required or desirable to use a mineral fiber filter, which may later be extracted by strong reagents. These fibers are usually made by leaching glass fibers with strong mineral acids followed by washing with deionized water. The fibers are rather weak but can be formed into filter sheets using little or no binder. These filters have been recently developed and are commercially available.

4.1.5 For some purposes, airborne particles may be collected on cellulose fiber filters. Cellulose low-ash filters are especially useful when the filter is to be destroyed by ignition or chemical digestion. However, these filters have higher flow resistance (lower sampling rate) and have been reported to have much poorer

collection efficiency than the glass fiber media. Furthermore, cellulose is very sensitive to moisture conditions, and even with very careful conditioning before and after sampling, accurately weighing the collected particles is difficult. The filter should be enclosed in a lightweight metal can with a tight lid and weighed.

4.1.6 As documented in the 40 CFR Part 58, Appendices A and B, identify the filter specifications when used as part of the *Federal Reference Method for Particulate Matter in Ambient Air*. These specifications include (1) a quartz-fiber, nonhygroscopic filter, (2) a size of approximately 8" x 10", (3) an exposure area of approximately 63 in.², (4) a 99% collection efficiency as measured by ASTM-2986 (DOP test) for particles 0.3 µm diameter, (5) a pressure drop range of 42-54 mm Hg at a flow rate of 1.5 m³/min through the nominal exposed area, (6) a pH of 6 to 10, and (7) a maximum weight integrity of 2.4 mg.

4.2 Visual Filter Inspection

4.2.1 After purchased, all filters must be visually inspected for defects, and defective filters must be rejected if any are found. Batches of filters containing numerous defects should be returned to the supplier.

4.2.2 The following are specific defects to look for:

4.2.2.1 Pinhole. A small hole appearing as a distinct and obvious bright point of light when examined over a light table or screen, or as a dark spot when viewed over a black surface.

4.2.2.2 Loose material. Any extra loose material or dirt particles on the filter that must be brushed off before the filter is weighed.

4.2.2.3 Discoloration. Any obvious visible discoloration that might be evidence of a contaminant.

4.2.2.4 Filter nonuniformity. Any obvious visible nonuniformity in the appearance of the filter when viewed over a light table or black surface that might indicate gradations in porosity across the face of the filter.

4.2.2.5 Other. A filter with any imperfection not described above, such as irregular surfaces or other results of poor workmanship.

4.2.3 Visually inspect each filter in front of an area light and observe for any specific defects listed above.

4.2.4 Use a renumbering stamp to code the filter on its noncollection side with a 7-digit code before tare weighing. The noncollection side of the filter is designated by the manufacturer printed number and by a mesh texture. The number code might be as follows:

Example: Filter Number Code = **9622001**

First 2 digits = yr, such as **96** for 1996

Third digit = project, such as **2**

Fourth digit = filter type, such as **2** for 8" x 10" quartz fiber, Whatman QMA type

Last 3 digits = filter number, such as **001**

5. Gravimetric Determination

5.1 Introduction

5.1.1 The filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain. The total volume of air sampled corrected to EPA standard conditions (25°C, 760 mm Hg) is determined from the measured flow rate and the sampling time. The concentration of TSP matter in the ambient air is computed as the mass of collected particles divided by the volume of air sampled (corrected to standard conditions) and expressed in µg/std m³ (see Inorganic Compendium Method IO-2.4). For samples collected at temperatures and pressures significantly different than standard conditions, the corrected

concentrations may differ substantially from actual concentrations Fg/m^3 , particularly at high elevations. The actual particulate matter concentration can be calculated from the corrected concentration using the actual temperature and pressure during the sampling period.

5.1.2 Verify that the weighing room conditions are within the limits. Filter equilibrium and weighing should be performed under controlled atmospheric conditions--a temperature of 25 ± 10 EC and a relative humidity $< 50\%$ (normally $50 \pm 5\%$ humidity).

5.1.3 Use the results from the motorized psychrometer to verify the temperature and relative humidity indicated by the hygrothermograph. Record the psychrometer values on the strip chart, along with the date, time, and your initials.

[Note: For traceability purposes, document your initials and full name in the front of the weighing room notebook.]

5.1.4 Record the room equilibration data on the Weighing Room Atmospheric Condition Form (see Table 4).

5.2 High Volume Filter Weighing Procedure

5.2.1 Filter Handling Procedure.

5.2.1.1 Filters should only be handled with finger cots or vinyl (nonpowdered) gloves. This procedure applies to filter handling in the field as well as in the weigh room.

5.2.1.2 Avoid using metal tweezers since the filters later will be used for metals analysis. When handling filter with gloved fingers or with any type of tweezers, avoid touching the sampled area.

5.2.2 Initial Weighing of High Volume Filter.

5.2.2.1 Upon receipt of new high volume filters (8" x 10" quartz fiber), take them to the climate controlled room, remove the paper and plastic envelope (wearing clean plastic gloves), place each on edge in a clean metal file rack, and cover with clean white paper towels.

5.2.2.2 Allow the filters to equilibrate in the metal file rack in the weighing room atmosphere for at least 24 h. Humidity and temperature must be within Federal Reference method specification, (i.e., $< 50\%$ and 15-35EC, respectively).

5.2.2.3 Zero the high volume balance before weighing.

5.2.2.4 Manually calibrate the balance. However, checks against two working NIST traceable weights (Class S) standards should be conducted before the daily weighing. If the difference between the traceable weights is more than 0.5 mg, do not use the balance until it has been repaired.

5.2.2.5 Record the results on the Weighing Balance Check Form (see Table 5).

5.2.2.6 Weigh each filter and record filter numbers and tare weights on the Filter Weighing Form (see Table 6).

5.2.2.7 Return the weighed filters to the plastic and paper envelopes.

5.2.2.8 Weigh filters in lots of approximately 100, if possible. After every tenth weighing, recheck the zero of the balance. The balance response should be ± 1 mg from 0. All differences should be corrected. Any difference exceeding 1 mg requires reweighing the previous ten filters. Any filter weight outside the normal range of 3.5-5.0 g requires immediate investigation. In addition, after every tenth filter weighing, the analyst should review at least one of the working standards. Once again, if this measurement disagrees from the verified value by more than 0.5 mg, reweigh the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both

working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 0.5 mg, then all weighings from the previous acceptable check must be repeated.

5.2.2.9 A second analyst should reweigh 10% of the filters. If the difference between the weights is less than 1.0 mg, the results are acceptable.

5.2.2.10 If the difference is greater than this limit, wait another 24 h and reweigh them.

5.2.2.11 If the results are still outside acceptable limits, wait another 24 h and reweigh them again. Then report the last reweigh values as the pre-field tare weights.

5.2.3 Final Weighing of High Volume Filter.

5.2.3.1 Exposed filters should be logged into the laboratory computer and received in individual manila folders, with computer printed identification labels affixed. No exposed filter should be touched until this label is affixed.

5.2.3.2 Condition all filters in the manner specified by the Federal Reference Method, as documented in Sections 5.1.2 and 5.2.2.

5.2.3.3 Weigh all filters according to the Tare Weighing Procedure in Section 5.2.2. Record final weights on the Filter Weighing Form (see Table 6).

5.2.3.4 For filters not to be analyzed, put an asterisk in the space preceding the four-letter code. Leave this space blank for samples to be analyzed. Sign and date the forms.

5.2.3.5 Archive asterisked high volume filters.

5.2.3.6 Have a second analyst reweigh 10% of the filters and verify that the weights have not changed.

- If the difference between the weights is less than 2.0 mg, the results are acceptable. Use the results from the first weighing.
- If the difference is greater than this limit, reweigh 100% of that lot and use the last reweigh weight.

5.2.3.7 Calculate and report the particulate matter concentrations as:

where:

$$\text{SPM} = \frac{(W_f - W_i) \times 10^6}{V_{\text{std}}}$$

SPM = mass concentration of suspended particulate matter (TSP or PM₁₀), µg/std m³.

W_i = initial weight of clean filter, g.

W_f = final weight of exposed filter, g.

V_{std} = air volume sampled, converted to standard conditions (25°C and 760 mm Hg), std m³.

10⁶ = conversion of g to µg.

5.3 Dichotomous and Partisol® Filter Weighing Procedure

5.3.1 Initial Weighing of Dichotomous or Partisol® Filters.

5.3.1.1 Fabric filters, 37-mm or 47 mm (as appropriate for the samples) in diameter, with a circumferential plastic reinforcing ring are usually supplied in small boxes. Open the boxes in the climate-controlled room under conditions suitable for high volume weighing. Cover with a clean paper towel and allow to equilibrate for 24 h.

5.3.1.2 Weigh filters on a Mettler microbalance; each balance is identified by a balance number.

5.3.1.3 Assign each balance a block of 7-digit sample numbers to be used sequentially. Assign a sample number to each filter when it is tared.

[Note: Inaccuracies in this aspect of the procedure will cause irremedial sample loss.]

5.3.1.4 Turn on the microbalance and allow it to warmup for at least 15 min. If the balance is used daily, leave it on at all times.

5.3.1.5 Manually calibrate the microbalance with two working NIST traceable weight (Class S) standards (for example, a 100-mg standard and a 200-mg standard). If the difference between the traceable weights is more than 3 µg, reweigh the working standards. Record the Results on the Weighing Balance Check Form (see Table 7).

5.3.1.6 If the values still disagree, troubleshoot and take appropriate corrective action, which may include (1) recertifying the working standards against the laboratory primary standards, and/or (2) having a service technician repair the microbalance.

5.3.1.7 Using clean nonserrated tweezers that will not damage the filter, remove the filter from the Lexan jig or filter cassette and place it on the weighing pan. Turn the release lever to "1" and dial in tare weights until a reading between 0.000 and 7.000 is obtained. Allow the reading to stabilize (which may require 2 to 4 min). Record the reading and the dialed-in tare weight on the Filter Weighing Form (see Table 8). Return the release lever to "0" and remove the filter from the weighing pan.

[Note: Do not use metal tweezers.]

5.3.1.8 Place a white label on a clean 50-mm diameter plastic petri dish (tight fitting lid type).

5.3.1.9 Assign a sample number to each filter (from those assigned to that balance), taking extreme care to avoid duplication or missed numbers.

5.3.1.10 Record the assigned sample number on the petri dish label, leaving sufficient room for one more letter to be written following the number. Do not record the balance number on this label.

5.3.1.11 Record the balance number, the assigned sample number, the dialed-in tare weight, and the digital-displayed tare weight on the sample form. Number each sheet of the form sequentially in the upper right-hand corner. Write "Tare Weight, Dichot or Partisol® Filters" on the top of each sheet. When bound, these forms may serve as the laboratory notebook.

5.3.1.12 Place the weighed filter in its numbered petri dish for future use.

5.3.1.13 After every tenth filter weighing, the analyst should check the "zero" and reweigh at least one of the working standard. Record the measurement on the Weighing Balance Check Form. Once again, if this measurement disagrees from the verified value by more than 3 µg, reweigh the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 3 µg then all weighings from the previous acceptable check must be repeated.

5.3.1.14 At the end of the weighing session, at least 10% of the filters should be reweighed by a second analyst. Record the replicate measurement on the Filter Weighing Form (see Table 6). If the replicate measurement disagrees from the original measurement by more than 15 µg, reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

5.3.1.15 Return the filter to the filter-handling container, replace the lid, and return it to conditioning chamber to protect it from contamination prior to sampling.

5.3.2 Final Weighing of Dichotomous or Partisol® Filter.

5.3.2.1 Filters should be returned from the field with a computer printed label affixed to the petri dish. The label should contain a five-character identification code that is different from the original sample number, a balance ID, the balance tare, and other information. All filters should be accompanied by extra labels. Some will have the words "To Be Analyzed" on the labels. The filter in each petri dish should rest in a Lexan jig or filter cassette.

5.3.2.2 Weigh each filter on the balance on which its tare weight was obtained. In the climate-controlled room, group the filters according to recorded balance numbers. Open the petri dishes, making certain that lids are placed under the bottoms and that no mixup occurs. Cover with a clean white paper towel and allow to equilibrate.

5.3.2.3 Repeat Section 5.3.1.4 to 5.3.1.6 of the filter tare weighing procedure.

5.3.2.4 Using clean, nonserrated tweezers that will not damage the filter, remove the filter from the Lexan jig or filter cassette and place it on the weighing pan. Dial in the tare weight recorded on the information label and turn the release lever to "1." Allow the reading to stabilize (which may require 2 to 4 min). Record the reading and the dialed-in tare weight. Return the release lever to "0" and remove the filter from the weighing pan.

5.3.2.5 After every tenth filter weighing, the analyst should check the "zero" and reweigh at least one of the working standards. Record the measurement on the Weighing Balance Check Form. Once again, if this measurement disagrees from the verified value by more than 3 ug, review the standard. If the two measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing some or all of the previously weighed filters, (2) recertifying the working standards against the laboratory primary standards, and/or (3) having a service technician repair the balance. At the end of the weighing session, reweigh both working standards. Record the measurements on the Weighing Balance Check Form. If both do not agree within 3 µg, then all weighings from the previous acceptable check must be repeated.

5.3.2.6 At the end of the post weighing session, at least 10% of the filters should be reweighed by a second analyst. Record the replicate measurement on the Filter Weighing Form (see Table 8). If the replicate measurement disagrees from the post measurement by more than 15 µg, reweigh the filter. If the measurements still disagree, troubleshoot and take appropriate corrective action, which may include (1) reweighing all or some of the previously weighed filters, (2) reweighing the working standards, and/or (3) having a service technician repair the microbalance. The analyst should not attempt to repair the microbalance.

5.3.2.7 If the dichotomous filter is not to be analyzed, use the tweezers to place it in a small glass envelope to which one of the extra labels has been affixed. Place an asterisk before the five-character code on the form. Deliver these filters to the filter bank for archiving.

5.3.2.8 If the filter is to be analyzed, use tweezers to carefully put it back into the petri dish. Place the petri dish **carefully** in a box.

5.3.2.9 Place a label on a sheet of 8 ½" x 11" paper for NAA, XRF, or other analysis as appropriate. Indicate the page number and balance number on each list. Keep the samples in the box in an order corresponding with the lists.

5.3.2.10 Without jostling the box, deliver it, the two lists, and the original Field Test Data Sheets with two copies of each to the sample custodian who will initial the original forms and return them upon receipt.

5.3.2.11 Calculate and report the particulate matter concentration for both fine and coarse samples utilizing the following equation:

$$PM = \frac{(W_f - W_i) \times 10^6}{V_{std}}$$

where:

PM = mass concentration of particulate matter (TSP, fine or coarse fraction), $\mu\text{g}/\text{std m}^3$.

W_i = average initial weight of clean filter, g.

W_f = average final weight of exposed filter, g.

V_{std} = air volume sampled, converted to standard conditions, std m^3 (see Inorganic Compendium Method IO-2.4).

10^6 = conversion of g to μg .

5.4 Transport of Filters

5.4.1 After collecting samples, transport the filters to the laboratory, taking care to minimize contamination and loss of the sample. Glass fiber filters should be transported or shipped in a shipping envelope. Cover the exposed surface of the membrane filters with an unexposed filter and seal the filter in plastic filter holders.

5.4.2 Assign numbers to the filters and log them into the data record form, ensuring that any necessary sampling information is included (Untreated filter samples may be stored indefinitely.)

5.4.3 Provide one blank sample with every 10 actual samples. No air is drawn through the blank filter, but it is subjected to the same handling and shipping manipulations as the actual samples.

6. Extraction of Glass Fiber Filters in Preparation for Metal Analysis

6.1 Introduction

This section describes both a microwave-extraction procedure and a hot-acid extraction procedure to extract inorganics from the particulate quartz glass-fiber filter. Following extraction, target analytes are analyzed by ICP, ICP/MS, FAA, or GFAA.

6.1.1 Ambient air quartz fiber filters should be received folded in half lengthwise with the particulate material inward and enclosed in protective envelopes. Store these protective envelopes approximately 15E-30EC until analysis.

6.1.2 The maximum sample holding times is usually 180 days. Analyze the samples within 180 days, even if these times are less than the maximum data submission times allowed.

6.2 Microwave Extraction Procedure

6.2.1 Filter Cutting Procedure.

6.2.1.1 Cut a 1" x 8" strip from the 8" x 10" filter using a template (see Figure 1) and cutting tool (see Figure 2) as described in the Federal Reference Method for lead. Use a laboratory microwave extraction system to extract the metals with a hydrochloric/nitric acid solution. After cooling, mix the digestate and use

Acrodisc® syringe filters to remove any insoluble material. Microwave extraction is used to prepare samples for ICP, ICP/MS, FAA, or GFAA.

6.2.1.2 Prior to use, acid wash the plexiglass filter template, the polysulfone centrifuge tubes and caps, and all other laboratory equipment that will come into contact with the filter samples to prevent contamination.

6.2.1.3 Using vinyl gloves, place the acid-cleaned filter template and cover inside a balance hood for cutting quartz fiber filters.

6.2.1.4 Wipe plexiglass template base, cover, and cutting blade with a clean, dry Kimwipe® to prevent sample cross-contamination.

6.2.1.5 Unfold the 8" x 10" quartz filter to be sectioned and carefully place sampled side up (numbered side down) within the plexiglass template filter margins.

6.2.1.6 Carefully (without disturbing sampled area of filter) place the grooved cover, notch side down, within the margins of the base template. Use a clear cutting blade to cut a 1" x 8" strip.

6.2.1.7 Using gloved fingers, accordion-fold or tightly roll the filter strip and transfer on edge to an acid cleaned polysulfone® tube, labeled with wax pencil. DO NOT use barcodes or tape in microwave.

6.2.1.8 Clean filter template between samples with dry Kimwipes®. (Gloves should be changed after 50 filters to minimize cross-contamination.)

6.2.1.9 Duplicate sample frequency is normally 1 per 20 field samples (see Table 9). Prepare a sample filter duplicate by moving the template cover to a second portion of the field collected filter. Cut an additional filter strip by moving the template cover to a second section of the filter and repeat Sections 6.2.1.6 through 6.2.1.8 above using a separate polysulfone tube.

6.2.1.10 Select a field collected filter for matrix spiking. In addition to the filter strip cut for determining metals, section a second portion of the filter, and fortify (spike) with target metals.

6.2.1.11 Prepare matrix spike samples at a frequency of 1 per 20 field samples or a minimum of 1 per extraction day (see Table 9). Move the template to a second section of the filter and repeat Sections 6.2.1.6 through 6.2.1.8, using a separate polysulfone tube and spike as shown in Table 9.

6.2.2 Microwave Calibration Procedure. Calibration of the microwave unit is a critical step prior to its use. In order that absolute power settings may be interchanged from one microwave unit to another, the actual delivered power must be determined, which allows the analyst to relate power in Watts to the partial power setting of the unit (% Power).

Calibration of a laboratory microwave unit (see Figure 3) depends on the type of electronic system used by the manufacturer. If the unit has a precise and accurate linear relationship between the output power and the scale used in controlling the microwave unit, the calibration can be a three-point calibration in the range of 50% to 100% power. If the unit does not prove linear (± 10 W) using the three-point technique, a multiple-point calibration is necessary. A bracketed calibration range of the digesting power to be used is recommended for determining the calibration points. If the unit power calibration needs multiple-point calibration, the point where the linearity begins must be identified. For example, a calibration at 100, 99, 98, 97, 95, 90, 80, 70, 60, and 50% power settings can be applied and the data plotted. The nonlinear portion of the calibration curve can be excluded or restricted. Each percent is equivalent to approximately 5.5-6.5 W and becomes the smallest unit of power that can be controlled. If 20-40 W are contained from 99-100%, that portion of the microwave calibration is not controllable by 3-7 times that of the linear portion of the control scale and will prevent duplication of precise power conditions specified in that portion of the power scale.

6.2.3 Microwave Power Evaluation. The equation in the following section evaluates the power available for heating in a microwave cavity. The variables are determined by measuring the temperature rise in 1 kilogram of water exposed to electromagnetic radiation for a fixed period of time. The following procedure is used for evaluating each calibration point, represented as % power output for each microwave.

6.2.3.1 Measure and record a 1 kilogram (1,000 g ± 0.1 g) sample of room temperature (23E± 2EC) distilled water in a thick-walled microwave transparent (Teflon®) beaker for each calibration point.

6.2.3.2 Measure and record the initial temperature of the water, (T_i), to within 0.1EC. The starting temperature should be between 22 and 26EC.

6.2.3.3 Place the Teflon® beaker in microwave and irradiate at full power (100% point) for 2 min (120 s). Each calibration point (i.e., 100%, 50% or multi-points) requires a separate clean beaker containing water at room temperature.

6.2.3.4 Remove beaker from the microwave and measure and record the maximum final temperature (T_f) to 0.1EC, within 30 s of the end of irradiation. This process should be done while stirring continuously (an electronic stirrer using a large stir bar works best).

Calculate the microwave power according to the following formula:

$$\text{Power} = \frac{K \times C_p \times M \times \Delta T}{t}$$

$$\frac{K \times C_p \times M}{t} = 34.87$$

$$\text{Power} = 34.87 \times \Delta T$$

where:

Power = The apparent power absorbed by the sample, watts (W = joule-s⁻²).

K = The conversion factor for thermochemical calories-s⁻¹ to W = 4.184.

C_p = The heat capacity, thermal capacity, or specific heat (cal-g⁻¹-EC⁻¹ = 1.0 for water).

M = The mass of the sample, grams.

ΔT = T_f - T_i, EC.

t = Time, s.

6.2.3.5 Derive an equation for the linear portion of the calibration range and determine the equivalent value in watts of the arbitrary setting scale. Use the actual power in watts to determine the appropriate setting of the particular microwave unit being used. Each microwave unit will have its own (% power) setting that corresponds to the actual power (in wattage) delivered to the samples.

6.2.3.6 An initial multipoint power evaluation should be performed for each microwave unit. If linear, the calibration should be checked on a regular basis, using the 3-point calibration verification routine. A single point verification may be appropriate when utilizing a single power output for digestion. If any part of the power source to the microwave has been serviced or altered, the entire calibration must be reevaluated.

6.2.4 Cleaning Procedure for PFA Vessels. All digestion vessels must be acid cleaned and rinsed with reagent water prior to use to prevent contamination.

6.2.4.1 Wash each PFA vessel with deionized detergent and rinse with reagent water.

6.2.4.2 Add 10 mL concentrated HNO₃ to each of 12 vessels, cap, and place in microwave.

6.2.4.3 Heat vessels at 100% power in microwave for 10 min as recommended by CEM (microwave manufacturer). Rinse the vessels with copious amounts of deionized, distilled water prior to use for any

analyses. If only 6 vessels are to be cleaned, 70% power may be utilized, which corresponds to approximately 5% per vessel.

6.2.5 Digestion Procedure for Microwave Extraction For Ambient Filter Samples.

[Note: Nitric and hydrochloric acid fumes are toxic. Prepare in a well-ventilated fume hood. Mixing results in an exothermic reaction. Stir slowly.]

6.2.5.1 Using vinyl gloves or plastic forceps, retrieve the filter strip from Section 6.2.1 and place on its edge in a labeled centrifuge tube. Using the plastic forceps, crush the filter strip down into the lower portion of the centrifuge tube to ensure acid volume will cover entire filter.

[Note: A breathing mask and vinyl gloves are required for safety of personnel handling dry glass-fiber filters. The breathing mask prevents the inhalation of minute glass shards and particulate material. The gloves protect the skin from the same materials and also prevent contamination of the sample by skin secretions. A recommended alternate to the use of a breathing mask would be performing cutting and transfer operations involving sample filters in a laminar flow hood, if available.]

[Note: More than one strip from a filter should be extracted to ensure adequate sample volume for sample and QC sample analysis. Blank filter samples should be extracted and analyzed, and digestion blanks should be run to ensure low levels of metals in the reagents used.]

6.2.5.2 Using a preset calibrated automatic dispensing pipette or Class A glass pipette, add 10.0 mL of the extraction solution to each of the centrifuge tubes. The acid should cover the strip completely. The sequence of adding the filter strip and acid to the centrifuge tube may be reversed, if more convenient, without affecting the results. Place the centrifuge tube in a Teflon® PFA vessel containing 31 mL of deionized water. Continue this process for a total of 12 samples to maximize microwave capacity.

6.2.5.3 Place the PFA vessel caps with the pressure release valves on the vessels hand-tight and tighten using the capping station to a constant torque of 12 ft-lb. Weigh and record the capped vessel assembly to the nearest 0.01 g. Place the vessels in the microwave carousel. Connect each sample vessel to the overflow vessel using the Teflon® PFA connecting tubes (see Figure 3).

6.2.5.4 Place the carousel containing the 12 vessels onto the turntable of the microwave unit. Any vessels containing 10 mL of acid solution for analytical blank purposes are counted as sample vessels. Irradiate the sample vessels at 486 W (power output) for 23 min. (Based on the calibration of the microwave as previously described). If fewer than 12 samples are to be digested, adjust the microwave system by reducing the power so that equivalent digesting power is delivered to the smaller sample batch. Generally, each vessel represents approximately 5% power. Therefore, a reduction in W would be reduced by 30% if only 6 vessels are digested. This reduction is only approximate, and each microwave unit will produce a different level of power output.

6.2.5.5 At the end of the microwave program, allow the pressure to dissipate (venting may be utilized with caution), then remove the carousel containing the vessels and cool in tap water for 10 min. Weigh the capped vessels assembly to the nearest 0.01 g and compare to the initial weight to verify no loss of sample. The initial and final weights should compare within 0.1 g. If the initial and final weights do not agree within 0.01 g, the appropriate action must be taken which may include rejecting the digested sample. Using the capping station uncapped the microwave vessels, remove the labeled centrifuge tubes containing samples and discard the water in the PFA vessels.

6.2.5.6 Using a calibrated automatic dispensing pipette or a Class A glass pipette, add 10 mL of deionized distilled water to each centrifuge tube. Cap the centrifuge tube tightly and vortex (mix) the contents thoroughly for 2-3 minutes to complete extraction. Using a nylon or teflon syringe pull-up a volume

of sample from the centrifuge tube, place Acrodisc filter on syringe and dispense into a pre-labeled sterile 15 mL centrifuge tube. Continue until centrifuge tube contains 10 mL of filtered digestate.

6.2.5.7 The final extraction volume is 20 mL based upon the above procedure. The final extraction solution concentration is 3% HNO₃/8% HCl. The filtered sample is now ready for analysis. Store for subsequent analysis by one or more of the Inorganic Compendium methods.

6.3 Hot Acid Extraction Procedure

6.3.1 Introduction. A hot extraction procedure to solubilize metals from the glass-fiber filter for subsequent analysis by ICP, ICP/MS, FAA, or GFAA is described in this method. An acid extraction solution is used to extract the metals from the quartz filter on a hot-plate.

6.3.2 Summary of Method.

6.3.2.1 Use the hot-acid extraction procedure as an alternate when microwave technology is not available.

6.3.2.2 Cut a 1" x 8" strip from the 8" x 10" filter as described in Federal Reference Method for lead. The inorganics are extracted from the filter strip by a HCl/HNO₃ acid solution using a hot acid extraction procedure. After cooling, pour the digestate rinses to a volumetric flask and dilute to volume. Filter to remove any insoluble material.

6.3.3 Hot Acid Extraction Procedure.

6.3.3.1 Using vinyl gloves or plastic forceps, retrieve the strip from Section 6.2.1 and place it in a labeled 150 mL Griffin beaker. Place the filter strip down into the lower portion of the beaker to ensure acid volume will cover entire filter.

[Note: More than one strip from a filter should be extracted to ensure adequate sample volume for sample and QC sample analysis. Blank filter samples should be extracted and analyzed and digestion blanks should be run to ensure low levels of metals in the reagents used.]

6.3.3.2 Using a preset calibrated automatic dispensing pipette or Class A glass pipette, add 10 mL of extracting acid (see Section 6.2.5.2) for analysis.

[Note: The acid should cover the strip completely.]

6.3.3.3 Place beaker on the hot-plate, contained in a fume hood, and reflux gently while covered with a watch glass for 30 min. Do not allow sample to dry. Remove the beakers from the hot-plate and allow to cool.

[Caution: Nitric acid fumes are toxic.]

6.3.3.4 Rinse the beaker walls and wash with D.I. water. Add approximately 10 mL reagent water to the remaining filter material in the beaker and allow to stand for at least 30 min. This critical step must not be deleted; it allows the acid to diffuse from the filter into the rinse. Transfer the extraction fluid in the beaker to a 20 mL volumetric flask or other graduated vessel. Rinse the beaker and any remaining solid material with Type I water and add the rinses to the flask. Some solids from the filter may be transferred to the flask with the rinses; this is acceptable. Dilute to the mark with Type I water and shake.

6.3.3.5 Using a nylon or Teflon[®] syringe, pull-up a volume of sample from the centrifuge tube, place disc filter on syringe, and dispense into a pre-labeled sterile 15 mL centrifuge tube. Continue until centrifuge tube contains 10 mL of filtered digestate.

6.3.3.6 The final extraction volume is 20 mL based upon the above procedure. The final extraction solution concentration is 3 % HNO₃/8% HCl. The filtered sample is now ready for analysis. Store for subsequent analysis by one or more of the Inorganic Compendium methods.

TABLE 1. CHARACTERISTICS OF FILTER MEDIUM

Cellulose Fiber (Cellulose Pulp)
<ul style="list-style-type: none"> • Low ash • Maximum temperature of 150EC • High affinity for water • Enhances artifact formation for $\text{SO}_4^{=}$ and NO_3^- • Good for x-ray/neutron activation analysis • Low metal content
Quartz Fiber (Quartz spun with/without organic binder)
<ul style="list-style-type: none"> • Maximum temperature up to 540EC • High collection efficiency • Non-hydroscopic • Good for corrosive atmospheres • Very fragile however • Difficult to ash; good with extraction
Synthetic Fiber (Teflon® and Nylon®)
<ul style="list-style-type: none"> • Collection efficiency > 99% for 0.01 μm particles • Low artifact formation • Low impurities • Excellent for X-ray analysis • Excellent for determining total mass due to non-hydroscopic nature • Nylon fiber good for HNO_3 collection
Membrane Fiber (Dry gel of cellulose esters)
<ul style="list-style-type: none"> • Fragile; requires support pad during sampling • High pressure drop • Low residue when ashed

TABLE 2. SUMMARY OF USEFUL PHYSICAL PROPERTIES OF VARIOUS FILTER MEDIUMS

Filter and Filter Composition	Density, mg/cm ²	pH	Filter Efficiency %
Teflon® (Membrane) (CF ₂) _n (2 µm Pore Size)	0.5	Neutral	99.95
Cellulose (Whatman 41) (C ₆ H ₁₀ O ₅) _n	8.7	Neutral (Reacts with HNO ₃)	58% at 0.3 µm
Glass Fiber (Whatman GF/C)	5.16	Basic pH - 9	99.0
"Quartz" Gelman Microquartz	6.51	pH - 7	98.5
Polycarbonate (Nuclepore) C ₁₅ H ₁₄ + CO ₃ (0.3 µm Pore Size)	0.8	Neutral	93.9
Cellulose Acetate/Nitrate Millipore (C ₉ H ₁₃ O ₇) _n (1.21 µm Pore Size)	5.0	Neutral (Reacts with HNO ₃)	99.6

TABLE 3. EXAMPLE OF TYPICAL BACKGROUND METAL CONCENTRATION (µg/FILTER) OF VARIOUS GRADES AND TYPES OF FILTERS

Metal	1	4I	42	542	Filter 17ch	Grades GF/A	EPM 2K	934QH	QMA	QMB
Al	1	2	2	1	--	4,300	170	2,950	4	--
Ag	--	--	--	--	--	< 1	< 1	--	< 1	--
As	< 0.02	< 0.02	< 0.02	< 0.02	--	< 6	< 6	--	< 6	--
B	1	< 2	2	2	--	6,100	42	--	42	--
Ba	< 1	< 1	< 1	< 1	--	8,500	50	< 1	10	--
Be	--	--	--	--	--	0.2	< 1	0.1	< 1	< 1
Bi	--	--	--	--	--	0.3	< 4	0.8	< 4	7
Ca	185	13	13	9	29	2,500	540	7,000	85	--
Cd	--	--	--	--	--	1	< 1	0.8	0.2	< 1
Co	--	--	--	--	--	11	< 1	5.5	1.1	< 1
Cr	0.3	0.3	0.3	0.7	--	0.2	4.5	3.0	1.6	0.7
Cu	1.2	0.1	0.4	0.2	0.6	56.0	1	7	3.4	3.4
Fe	5.0	6.0	6.0	3.0	5.0	100	15	265	23	29
Mg	< 0.005	--	< 0.005	< 0.005	--	--	--	--	--	--
K	4.0	1.5	1.5	0.6	7	775	573	125	--	--
Mg	7	1.8	1.8	1.0	6	1,090	238	2,800	--	--
Mn	0.1	< 0.05	< 0.05	< 0.05	--	8.0	< 1	2	0.5	< 1
Mo	--	--	--	--	--	2.0	< 2	4	< 2	< 1
N	28	--	12	260	--	--	--	--	--	--
Na	160	40	37	8	30	1,830	1,020	1,100	280	--
Ni	--	--	--	--	--	6.0	--	2.5	3.4	2.2
Pb	0.4	0.2	0.2	0.1	1	10	2.5	2.0	2.3	8.3
S	17	< 5	< 5	< 5	--	26	--	--	--	--
Sb	< 0.02	< 0.02	< 0.02	< 0.02	--	< 4	< 4	--	< 4	--
Si	15	< 2	< 2	< 2	--	--	--	--	--	--
Ti	--	--	--	--	--	0.1	< 1	< 30	< 1	< 4
V	--	--	--	--	--	0.1	--	10	--	< 4
Sn	2.5	0.6	0.6	0.3	--	5,500	15	34	10	13

TABLE 4. EXAMPLE WEIGHING ROOM ATMOSPHERIC CONDITION FORM

Equilibrium Period				Temperature Limits = 15EC to 35EC			Relative Humidity Limits = < 50%			Name
Begin Date	Begin Time	End Date	End Time	Max-Min	Avg	Limits met?	Max-Min	Avg	Limits met?	

TABLE 5. EXAMPLE WEIGHING BALANCE CHECK FORM

Date	Time	Balance Type	Balance ID	Class S Weights Serial No. or ID	mg			Limit = 0.5 mg Limits met?	Name
					Class S weight	Balance weight	Difference		

TABLE 6. EXAMPLE FILTER WEIGHING FORM

Filter No.	Pre-field Initial Weighing		10% Second Weighing by Second Analyst (Limit = 1.0 mg)				Post-field Final Weighing		10% Second Weighing by Second Analyst (Limit = 2.0 mg)			
	Weight (mg)	Name-Date	Weight (mg)	Diff. (mg)	Limit met?	Name-Date	Weight (mg)	Name-Date	Weight (mg)	Diff. (mg)	Limit met?	Name-Date

TABLE 7. EXAMPLE WEIGHING BALANCE CHECK FORM

Date	Time	Balance Type	Balance ID	Class S Weights Serial No. or ID	mg			Limit = 3 ug Limits met?	Name
					Class S weight	Balance weight	Difference		

TABLE 8. EXAMPLE FILTER WEIGHING FORM

Filter No.	Pre-field Initial Weighing		10% Second Weighing by Second Analyst (Limit = 15 Fg)				Post-field Final Weighing		10% Second Weighing by Second Analyst (Limit = 15 Fg)			
	Weight (mg)	Name-Date	Weight (mg)	Diff. (mg)	Limit met?	Name-Date	Weight (mg)	Name-Date	Weight (mg)	Diff. (mg)	Limit met?	Name-Date

TABLE 9. EXAMPLE QUALITY CONTROL SAMPLES

Type	Frequency	Contains 1"x8" filter strip	Comments ^a
Method Blank	1 per 20 samples	No	Containing reagents only, to evaluate background contributions from reagents.
Filter Lot Blank	1 per filter lot change	Yes	Analyzed prior to use of new filter lot.
Filter Duplicate	1 per 20 samples	Yes	This is a second 1"x8" filter strip cut from a single field sample.
Matrix Spike ^b	1 per 20 samples	Yes	See individual methods.
LCS ^c	1 per extraction day	Yes	See individual methods.

^aThese multimetal stock standards can be acquired from Spex Industries, Inc., Inorganic Ventures Inc., or equivalent.

^bThe matrix spike (MS) is a 1"x8". strip cut from a field sample filter and spiked at a target level.

^cThe Lab Control Sample (LCS) is a blank filter spiked with the same concentration of metals as the matrix spike.

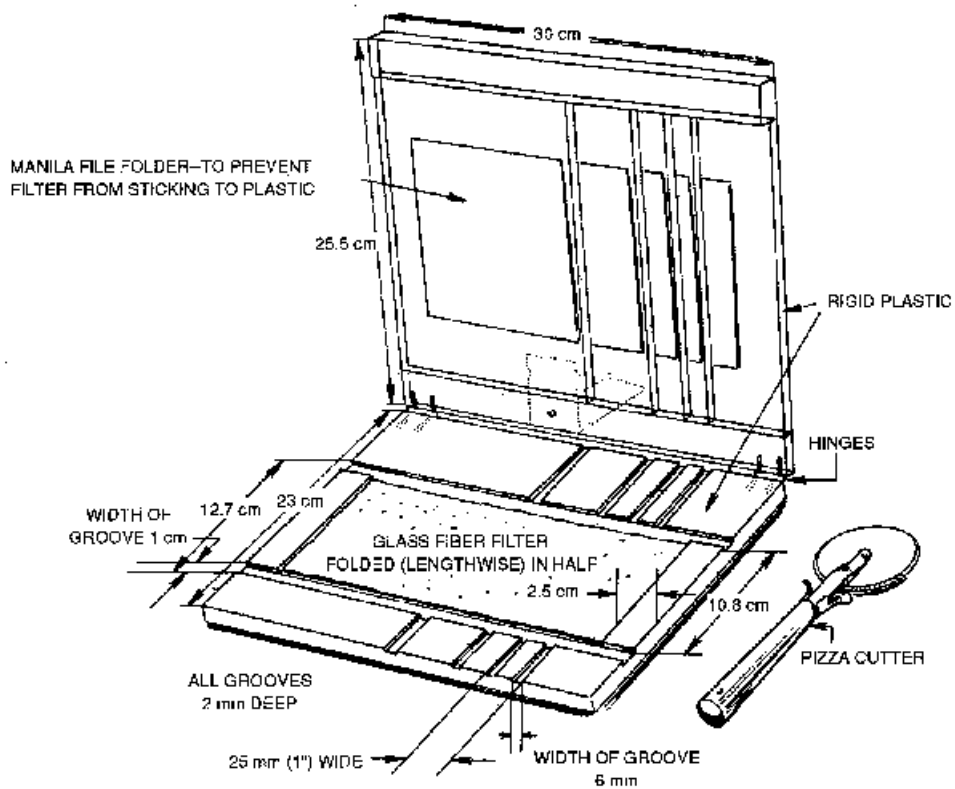


Figure 1. Example of templates for cutting filters.

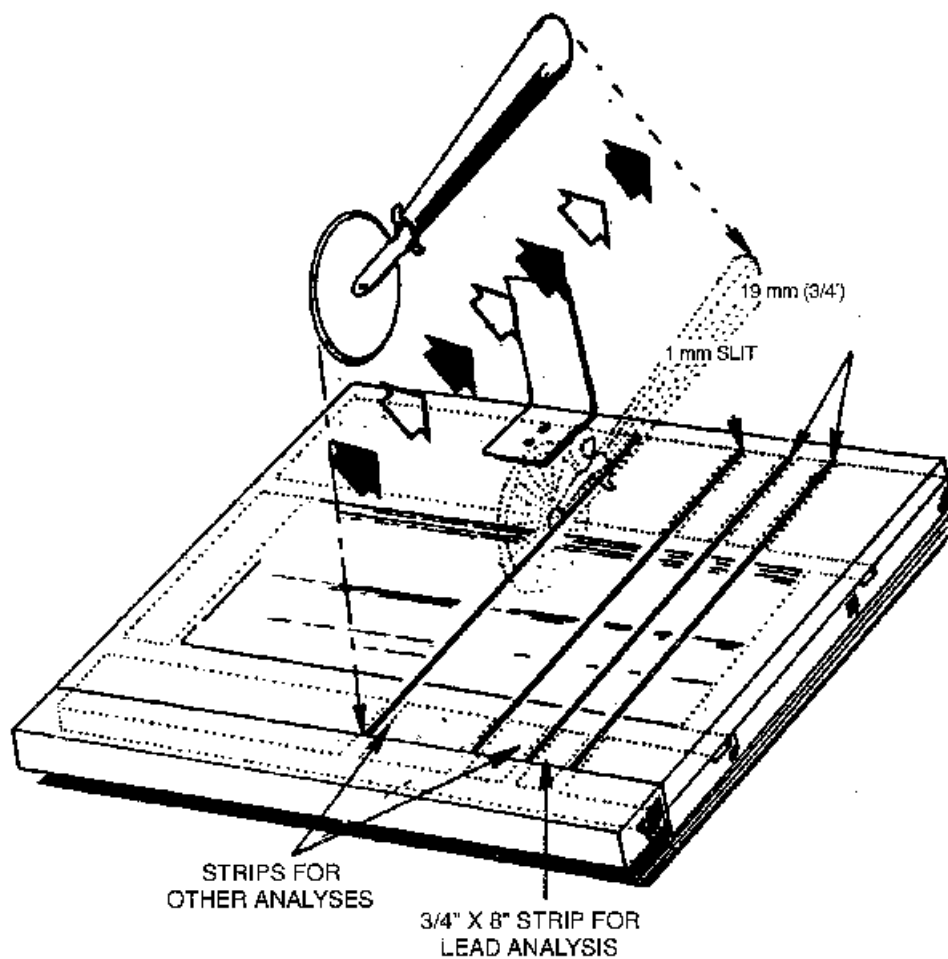


Figure 2. Diagram of filter cutting procedure.

MICROWAVE EXTRACTION

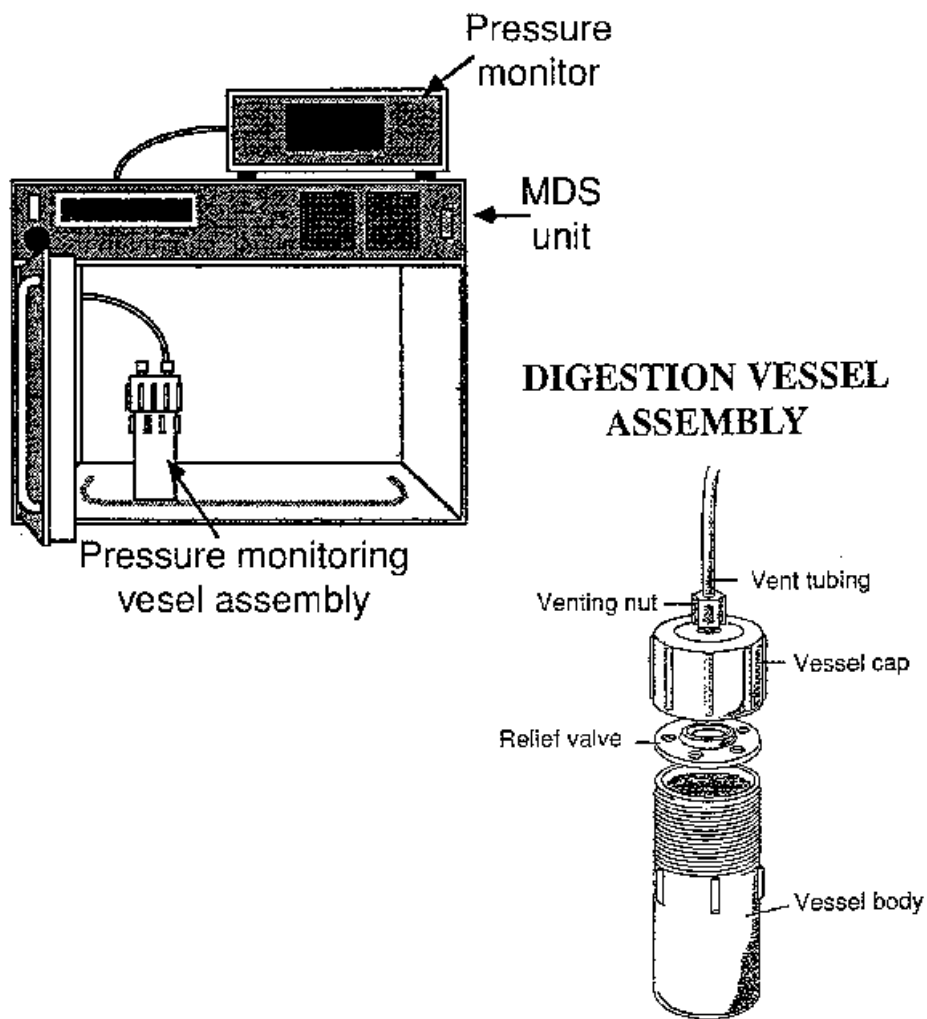


Figure 3. Example of microwave digestion system.

ATTACHMENT L-C

MACTEC STANDARD OPERATING PROCEDURE FOR
AIRMETRICS MINIVOL® PORTABLE AIR SAMPLER
MACTEC SOP-104

Harding ESE, Inc.
Standard Operating Procedure

90 Digital Dr.
Novato, CA 94949

Original Date Issued:	August 2002
Type:	Revision 0
Revision Date:	
Procedure #:	HESE SOP-104
Total Pages:	7

NAME	Standard Operating Procedure for AirMetrics MiniVol Portable Sampler
SCOPE	The scope of the SOP will detail the operating procedures and the procedures used to perform calibrations on AirMetrics MiniVol Portable Air Sampler
REFERENCE	EPA Compendium Method IO-2.1 AirMetrics MiniVol Portable Air Sampler User's Manual
EQUIPMENT	AirMetrics MiniVol Portable Air Sampler

INTRODUCTION MiniVol

Although accurate and precise, the monitors are bulky and immobile, causing difficulty in studying remote sites without a power source. The EPA and the Lane Regional Air Pollution Authority addressed the need for portable sampling devices with the production of the AirMetrics MiniVol Portable Air Samplers. These ambient air sampling machines are designed to collect either PM₁₀ or PM_{2.5}, depending on the attached impactor and filter head, and can also be used to measure non-reactive gases (CO & NO_x).

The MiniVols are not federal reference method (FRM) samplers, though their results closely approximate FRM air quality data. The MiniVol is a lightweight battery-operated sampler, which allows it to be used in remote areas. The low cost of the sampler allows for a network of MiniVols to be easily set up at a lower cost than a large machine network. Other features include a 7-day programmable timer, an elapsed time totalizer, rechargeable battery packs, and all-weather PVC construction.

The procedures that lie herein are to be used by the operator responsible for the collection of the PM₁₀ and PM_{2.5} filters. Refer to the manufacturer's operation manual for integrated gas sampling, hardware descriptions, and maintenance details.

2. FILTER HANDLING AND PRESAMPLING PREPARATION

2.1 Filter Handling

The current filter media approved by the U.S. EPA for sampling particulate matter is a 2µm pore-size Teflon material in a white 47mm diameter casing. Filters will be provided by the processing laboratory, and should be returned there for analysis with a copy of the filter's data

form. Touching the individual filters could void the sample and physically handling should be avoided. Storage of the filters should be in a dry, controlled environment. There are no control limits on humidity and temperature for unexposed filters, but exposed filters should be kept below ambient temperature. A cooler type container loaded with "blue ice" and transported in an air-conditioned vehicle is recommended.

2.2 Connecting the Sampler Body and the Battery Pack

When connecting the sampler body and battery pack, note that the body's pins are unevenly spaced and can only fit onto the battery pack in one way. The pin closest to a latch on the sampler body inserts into the odd-colored receptacle on the battery pack.

2.3 Sample Date and Time Programming

The Programmable Timer can be set to run for up to six cycles within 24 hours, in addition to separate time periods on different days within one week. The real-time clock must first be set to the correct day and time in order for sampling programming to be set. To set the day, hold down the "Clock" button until the correct day appears on the display. The time can be set by holding down the "Clock" button while depressing the "Hour" and "Min" buttons until the correct time is set.

To set the timer to sample, press the "Prog" button once to display the "Prog 1_{ON}" setting. Use the "Hour" and "Min" buttons to set the time of day for sampling to begin. Press the "Week" button until the correct sampling day appears on the display. Pressing the "Prog" button again will show the "Prog 1_{OFF}" setting. In this setting, program the monitor to stop sampling in the same manner as programming the monitor to turn on. Set the monitor to automatically run by pressing the "ON/AUTO/OFF" button until "AUTO" is selected.

2.3 Equipment Calibration

Calibration Apparatus A MiniFlo flow rate transfer standard device is used as the flow rate reference to calibrate the sampler's rotameter-. To be valid, the MiniFlo transfer standard must have been calibrated against a primary standard traceable to the NIST within the last year. A calibrated digital manometer, Magnehelic® gage, or water manometer is required to measure the pressure drop across the MiniFlo orifice element. The actual ambient temperature and barometric pressure must also be measured or obtained locally.

Precalibration System Check Procedures for the precalibration system check are as follows:

1. Place a filter into the MiniVol sampler filter holder and attach the filter holder assembly to the sampler. (Filters used for flow rate calibrations should not be used for subsequent sampling.)
2. Turn on the sampler and allow it to warm up to full operating temperature (at least 2 minutes).
3. While the sampler is running, depress and hold the reset button. (This allows the sampler to continue to run without tripping the low flow shut-off circuit.) Close off the inlet using the palm of your hand. Observe the rotameter, if there are no leaks the rotameter should drop to zero and remain there.

4. Verify that the transfer standard calibration equation is current and traceable to an acceptable primary standard.
5. Adjust manometer or Magnehelic® gage to read zero, and connect to the pressure tap on MiniFlo transfer standard. Note: Magnehelic® gage is sensitive to the position it is in when zeroed (vertical or horizontal). Maintain this position when taking readings.

Rotameter Calibration. Procedures for calibrating the sampler rotameter are as follows:

1. Install the MiniFlo transfer standard on the inlet tube of the MinVol sampler filter holder.
2. Turn on the sampler and allow it to warm up to normal operating temperature (at least 2 minutes). The transfer standard must also equilibrate before proceeding with the calibration.
6. Read and record the following parameters:
 - Ambient temperature (T_{act}), °K
 - Barometric pressure (P_{act}), mm Hg
4. Adjust the flow control until the rotameter displays 6.5 L/min.
5. Read and record the following parameters:
 - Transfer standard pressure drop (H), inches of water
 - Sampler rotameter indication (Q_{ind}), liters per minute
6. Repeat steps 4 and 5 for rotameter settings representing flow rates of 6.0, 5.5, 5.0, 4.5, and 4.0 L/min.

Calibration Calculations. Gather together all the calibration data, including the transfer standard calibration information and the MiniVol sampler calibration data sheet. The following calibration calculation procedures are recommended.

Note: These calculations should be done at the time of the calibration, rather than later. This approach will allow additional calibration points to be taken if questions arise about the data that has already been obtained.

Calculate Q_{act} for each calibration point as determined by the transfer standard calibration equation (**Eq.A** from MiniFlo calibration certificate).

Calculate and record the standard corrections ($Q_{@std}$) for each calibration point as:

Eq. 1

$$Q_{@std} = Q_{act} \times \sqrt{\frac{T_{std} P_{act}}{T_{act} P_{std}}}$$

where:

$Q_{@std}$ = standard correction flow rate, L/min

Q_{act} = flow rate at ambient conditions (transfer standard), L/min

T_{act} = ambient temperature, °K
 P_{act} = ambient barometric pressure, mm Hg
 T_{std} = standard temperature, 298 °K
 P_{std} = standard pressure, 760 mm Hg.

On a sheet of graph paper or computer spreadsheet plot the calculated standard correction flow rates, $Q_{@std}$ (y-axis) versus the corresponding rotameter indicated flow rate, Q_{ind} (x-axis). Using a programmable calculator or a spreadsheet and the following model, calculate the linear regression slope (m_{vol}), intercept (b_{vol}), and correlation coefficient (r) to obtain the MiniVol™ sampler flow rate calibration relationship.

For the regression model $y=mx+b$

Eq. 3

$$\text{let } y = Q_{@std} = Q_{act} \sqrt{\frac{T_{std} P_{act}}{T_{act} P_{std}}} \text{ and } x = Q_{ind}$$

So that the model is given by:

Eq. 4

$$Q_{@std} = m_{vol} Q_{ind} + b_{vol}$$

A six-point calibration should yield a regression equation with a correlation coefficient of $r > 0.990$, with no point deviating more than 2% from the value predicted by the regression equation.

Note: For actual sample periods, the sampler's average actual operational flow rate Q_{act} is calculated from the calibration slope and intercept using

Eq. 5

$$Q_{act} = (m_{vol} Q_{ind} + b_{vol}) \times \sqrt{\frac{T_{act} P_{std}}{T_{std} P_{act}}}$$

where

Q_{act} = sampler flow rate at ambient conditions, actual L/min
 Q_{ind} = rotameter response, L/min
 T_{act} = ambient temperature, °K
 P_{act} = ambient barometric pressure, mm Hg
 T_{std} = standard temperature, 298 °K
 P_{std} = standard pressure, 760 mm Hg
 m_{vol} = slope of the MiniVol™ flow rate calibration relationship
 b_{vol} = intercept of the MiniVol™ flow rate calibration relationship.

T_{act} and P_{act} readings may be measured on-site or obtained from a nearby U.S. National Weather Service or airport weather station. Barometric pressure readings obtained from remote stations must be at station pressure (not corrected to sea level), and they may have to be corrected for differences between the elevation of the monitoring site and that of the remote station. If ambient temperature and pressure readings are not available, a seasonal average temperature and barometric pressure can also be used. Care must be taken, however, that the actual conditions at the site can be reasonably represented by such averages. It is therefore recommended that seasonal values represent actual values within 20 EC and 40 mm Hg.

Rotameter Set Point Adjustment Procedure. Calculate and record the rotameter set point using temperatures and pressures expected on the day(s) sampling is conducted. These values can be seasonal set points or daily settings.

Eq. 6

$$I_{sp} = \frac{5.0 \times \sqrt{\frac{T_{std} P_{act}}{T_{act} P_{std}} - b_{vol}}}{m_{vol}}$$

where:

I_{sp} = sampler rotameter set point flow rate adjustment

5.0 = MiniVol design flow rate, L/min

P_{act} = ambient barometric pressure, mm Hg

T_{act} = ambient temperature, EK.

T_{std} = standard temperature, 298 EK

P_{std} = standard pressure, 760 mm Hg

m_{vol} = slope of the MiniVol flow rate calibration relationship

b_{vol} = intercept of the MiniVol flow rate calibration relationship.

Adjusting the sampler rotameter to seasonal average conditions will help minimize data loss caused by exceeding the MiniVol's design condition specifications.

Sampler Calibration Frequency

To ensure accurate measurement of the PM10/PM2.5 concentrations, calibrate the sampler upon installation and then recalibrate it as follows:

1. At least annually.
2. After any repairs that might affect sampler calibration.
3. If the field calibration flow check results exceed QC limits ($\pm 7\%$ from the sampler's indicated flow rate).

3. FIELD SAMPLING PROCEDURES

3.1 Sample Filter Cassette Recovery

1. Disconnect the AC cord from the battery pack and remove the MiniVol from its hanger. If possible, move indoors or under cover. Remove the handle by unscrewing a side cap, and removing the o-rings from the bar.
2. Lift the sampler out of the housing by holding the top 6" cap and lifting up gently. Support the sampler by either holding it above its housing or using the triangular mount stand on the bottom of the sampler. Use of the triangular stand should only be done when the battery pack is attached to the sampler. Do not pull the sampler completely out of the housing; the power cord will be torn.
3. Check for any light on the display board, such as "Low Flow" or "Low Battery" and record the findings in the Notes section of the data sheet.
4. Do a post flow check by turning on the pump and ensuring the Rotameter reads 5.0 LPM. Record this on the data sheet. If the pump does not start, press the reset button on the right side of the display board.
5. Record the hour meter value in the Hour Meter Stop field on the data sheet. Lower the sampler back into the housing.
6. Remove the filter head by pressing down on the quick-disconnect while pulling up on the filter assembly. Unscrew the base of the filter assembly and remove the filter with a gloved hand. Secure the filter cassette in the petri dish labeled with the correct filter number.
7. Return a copy of the data sheet with the exposed filter to the processing laboratory. If the PM₁₀ monitor is not to be used until the next sampling run, it should be disconnected from the battery pack and properly stored indoors.

3.2 Sample Filter Cassette Installation

1. The operator is to record the following information for the new filter on the AirMetrics MiniVol PM_{2.5} or PM₁₀ Field Data Form:
 - Filter Run Date
 - Site ID
 - Type of Impactor (PM_{2.5} or PM₁₀)
 - Model Number
 - Sample ID
 - Filter Number
 - Filter Date In
 - Start Date/Time
 - Stop Date/Time
 - Hour Meter Start
 - Start Flow
 - Impactor Run

2. Place a new filter in the opened (from directions above) filter assembly with a gloved hand and re-assemble the unit. Place filter assembly on the quick-disconnect and press firmly down to snap it in place. Pull up to insure connection. Do not pull up on the small rain hood cap.
3. Remove the sampler body from its housing and support it as defined above. On the Programmable Timer, check the current standard time. The reading should be correct during the fall and one hour ahead during the spring. To adjust the time, hold the clock button down while depressing the "Min" or "Hour" keys.
4. Press the "Prog" key to access the 6 available programmable times to turn the sampler on and off. Scroll through each program (1-6) by pressing "Prog" and reset any existing programmed times by using the "RST/RCL" button.
5. At "Prog 1_{ON}", set the time for the beginning of the sample run, which will be 12am of the sampling day on the calendar. At "Prog 1," set the day of the week using the "Week" key. The days will scroll through 10 different choices for day runs. Set the day according to the calendar date. At "Prog 1_{OFF}", set the time for the end of the sample run, which will be 12am the next day. Set the day of the week (same as above). Press the "Clock" key to exit the Program menus.
6. Select the Automatic mode by depressing the "ON/AUTO/OFF" button. The sampler will now start sampling at the programmed time automatically.
7. Reassemble the sampler's handle and place it on the hanger with the impactor away from the hanger stand. Plug in the AC power cord if applicable.

* After every five sampling runs, return the impactor assembly to the processing laboratory. A clean assembly will be supplied for the next run. Only impactors designed for the collection of PM_{2.5} will be supplied, so it is necessary to change the assembly if a new PM₁₀ impactor is needed. Disassemble the entire unit, and switch the PM_{2.5} impactor (with the small opening at its base) with the PM₁₀ impactor (with the larger opening at its base). The PM₁₀ impactor will be the only one present, and the entire unit will be half the size of the PM_{2.5}. Return the extra parts with the assembly after five runs.

3.3 Integrated Gas Sampling

For PM₁₀ Sampling, Follow the steps below:

1. Ensure that the PM₁₀ Impactor (silver) is installed in the preseparator assembly. The multiple impactor adapter is not needed, nor the PM_{2.5} Impactor (gold)
2. Clean and lubricate the target disk at least every 2-4 sampling periods, or more frequently depending on the degree of impaction stage soiling.
3. After the sampler has been assembled, After the sampler has been assembled, calibrated, verified to be in proper working order, and a filter loaded in the filter holder assembly, set flow rate adjustment in accordance with procedures as shown in the Operation Manual.
4. Follow all other procedures in the Operation Manual, noting that PM10 is being sampled.