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# Quality Assurance Guidance Document 2.10

# Monitoring PM<sub>10</sub> in Ambient Air Using a Dichotomous Sampler

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# Introduction

 $PM_{10}$  is particulate matter with an aerodynamic diameter less than or equal to a nominal 10 µm. (Note: In reference to  $PM_{10}$  samplers, all particle sizes are specified by their aerodynamic rather than physical diameter.) As described in 40 CFR Part 50, Appendix M, (the reference method for  $PM_{10}$  sampling), a  $PM_{10}$  sampler draws a measured quantity of ambient air at a constant flow rate through a specially designed particle size discrimination inlet. Particles in the  $PM_{10}$  size range are then collected on one or more filters during the specified 24-hour sampling period. Each sample filter is weighed before and after sampling to determine the net weight (mass) gain of the collected  $PM_{10}$  sample.

The total volume of air sampled is determined from the measured volumetric flow rate and the sampling time. The concentration of  $PM_{10}$  in the ambient air is computed as the total mass of collected particles in the  $PM_{10}$  size range divided by the volume of air sampled.  $PM_{10}$  data are expressed as micrograms per cubic meter ( $\mu$ g/m<sup>3</sup>). The particle size discrimination characteristics (sampling effectiveness) of the sampler inlet over the  $PM_{10}$  size range, and particularly the particle size at which the sampling effectiveness is 50 percent, are functional specifications tested in accordance with explicit procedures prescribed in 40 CFR Part 53. Sampling methods for  $PM_{10}$  that meet all requirements in both Parts 50 and 53 are designated as  $PM_{10}$  reference methods for use in State or local air monitoring stations (SLAMS) and prevention of significant deterioration (PSD) monitoring. These designated methods are usually identified by the name of the manufacturer and by the model of the sampler.

Two types of samplers that meet designation requirements are the high-volume (HV)  $PM_{10}$  sampler and the dichotomous sampler. Only the dichotomous sampler is discussed in this section of the Handbook; the HV  $PM_{10}$  sampler is discussed in Quality Assurance Guidance Document 2.11.

The most common commercially available dichotomous samplers are low flow rate (16.7-L/min) samplers that collect particles with an aerodynamic diameter up to a nominal size of 10  $\mu$ m. Dichotomous samplers further divide the sample into fine (0- to 2.5- $\mu$ m) and coarse (2.5- to 10- $\mu$ m) fractions, which are collected on separate filters.

Particles with aerodynamic diameters greater than 10  $\mu$ m are removed from the air sample by inertial separation in a specially designed fractionating inlet such as the one illustrated in Figure 1. Particle-laden air is drawn into the inlet and deflected downward into the acceleration jet of an impactor. Because of their greater inertia, particles larger than 10  $\mu$ m are removed by the impactor. Particles smaller than 10  $\mu$ m are drawn through the vent tube into a virtual impactor assembly, which further separates the particles into fine and coarse size fractions. Figure 2 illustrates the principle used to achieve this division. The air stream containing PM<sub>10</sub> particles is forced through an acceleration nozzle into the virtual impactor assembly where the air flow is split. Most of the fine particles make a sharp turn to follow the higher velocity flow stream and pass on to the fine particle filter. Because of their greater inertia, the coarse particle filter. Because a small proportion of the fine particles are collected on the coarse particle filter, a correction must be made when fine and coarse particle concentrations are calculated.

# **Method Highlights**

The procedures provided in this document are designed to serve as guidelines for the development of quality assurance (QA) programs associated with the operation of dichotomous samplers. Since recordkeeping is a critical part of QA activities, several data forms are included to aid in the documentation of necessary data.

The tables and figures included in some sections summarize the material covered in the text. The material covered in this section is summarized here:

**Section 1.0**, Procurement of Equipment and Supplies, describes recommended procurement procedures, equipment selection criteria, and minimum accuracy requirements. It also provides an example of a permanent procurement record.



Figure 1. Example of a PM<sub>10</sub> dichotomous sampler inlet head.

**Section 2.0**, Calibration Procedures, provides detailed calibration procedures for the dichotomous sampler. References are provided for calibration procedures for the flow rate transfer standards and other monitoring equipment. Table 2-3 provided in this section summarizes the acceptance limits for calibration.

**Section 3.0**, Field Operations, details procedures for filter installation, performance of operational quality control (QC) checks, sample handling, and data documentation. Complete documentation of background information during sampling is one of several QA activities that are important to future data validation; particularly important are any unusual conditions existing during collection of the sample. Such conditions should be noted.

**Section 4.0**, Filter Preparation and Analysis, presents important considerations for the handling, integrity, equilibration, and weighing of filters. A high-quality filter is recommended for use when additional chemical analyses are expected. The analytical balance must be calibrated annually, and the filters must be equilibrated in a controlled environment.



Figure 2. Principle of the secondary (2.5 µm) particle size separation in a dichotomous sampler by virtual impaction.

Section 5.0, Calculations, Validations, and Reporting of  $PM_{10}$  Data, presents calculations for determining  $PM_{10}$  mass concentrations and minimum data validation requirements. The final data review and validation, including standardized reporting procedures, are all important parts of a QA program. Independent checks of the data and calculations are required to ensure that the reported data are both accurate and precise.

**Section 6.0**, Maintenance, recommends periodic maintenance schedules to ensure that the equipment is capable of performing as specified. Minimum maintenance requirements and procedures are outlined. The objective of a routine maintenance program is to increase measurement system reliability.

**Section 7.0**, Auditing Procedures, presents independent audit activities and laboratory evaluations that provide performance checks of flow rate measurements and data processing. Filter weighing procedures and balance operation evaluations and a system audit checklist are also provided. Independent audits evaluate data validity.

**Section 8.0**, Assessment of Monitoring Data for Precision and Accuracy, describes the assessment procedures for determining the accuracy and precision of the data. The precision check is performed by using collocated samplers.

**Section 9.0**, Recommended Standards for Establishing Traceability, discusses the traceability of monitoring equipment to establish standards of higher accuracy, a prerequisite for obtaining accurate data.

# **1.0 Procurement of Equipment and Supplies**

The establishment of an ambient  $PM_{10}$  air monitoring network requires the procurement of specialized equipment and supplies for field operations and subsequent filter analysis. Information in this section has been provided to assist the agency in selecting the proper equipment. Section 1.1 describes minimum sampling equipment necessary to conduct field operations. Recommended laboratory instrumentation is described in Section 1.2.

In addition to field operations and laboratory equipment, a data handling system (including forms, logs, files, and reporting procedures) must be developed and implemented.

It is recommended that each agency establish minimum monitoring equipment requirements and budgetary limits before the procurement procedures are initiated. Upon receipt of the sampling equipment and supplies, appropriate procurement checks should be conducted to determine their acceptability, and their acceptance or rejection should be recorded in a procurement log. Figure 1.1 is an example of such a log. This log will serve as a permanent record for procurement and provide fiscal projections for future programs. It will also help to provide the continuity of equipment and supplies. Table 1-1 lists the major equipment needed, how it should be tested, suggested acceptance limits, and actions to be taken if acceptance limits are not met.

# 1.1 Procurement Prerequisites—Field Operations

#### 1.1.1 Dichotomous Samplers

The individual sampler must meet U.S. EPA operational standards and be a model designated as a reference or equivalent method. A complete listing of minimum sampler requirements are in 40 CFR Part 50, Appendix M. Dichotomous samplers not designated as reference or equivalent methods **may not** be used for reporting data to determine attainment of the National Ambient Air Quality Standards (NAAQS) for  $PM_{10}$ . Costs for dichotomous samplers will vary with the manufacturer and the sophistication of the sampler. Basic considerations include the flow control and measurement system, maintenance requirements, reliability, and ease of operation.

					D	ate	_		Accept/	
ltem	Description	Qty	PO #	Vendor	Ord.	Rec'd	Cost	Initials	Reject	Comments
1 case filters	2 m Pore 37 mm	60	806-L187	WIZ Sapply	5/1/96	6/15/96	\$88.29	DAW	Accept	None



Equipment	Acceptance check	Acceptance limits	Action if require- ments are not met
Field operations			
Sampler	Sampler complete; no evidence of damage. Model designated as reference or equivalent method	Specifications outlined in 40 CFR Part 50, App. M	Reject sampler
Calibration QA/QC supplies	Check against NIST- traceable standards	Within accuracy limits presented in text	Adjust or reject
Laboratory operations			
Filters	Meets requirements.Filter undamaged and suitable for sampling	Specifications outlined in 40 CFR Part 50, App. M. Compatible with individual samplers	Reject filters
Filter handling materials	No evidence of damage	Allows for minimum filter damage and loss of particles	Reject materials or improve method for transporting filters
Lab equipment and instrumentation	Check against ASTM- or NIST-traceable standards	Within accuracy limits presented in text	Adjust or reject

# TABLE 1-1. ACCEPTANCE TEST AND LIMITS FOR PROCUREMENT OF EQUIPMENTAND SUPPLIES

Using only one model of sampler in a network will minimize the variety of spare parts required to keep the network in operation. An in-house inventory of general maintenance supplies and replacement parts is recommended. These include various hand tools, general all-purpose cleaner, penetrating oil, distilled water, disposable laboratory wipers, soft brush, and cotton swabs. Spare parts for the sampler may be supplied by the manufacturer or many may be purchased locally.

#### 1.1.2 Calibration Equipment

Calibration activities require specialized equipment that will not necessarily be used in routine monitoring. At a minimum, the following equipment is required:

- A thermometer capable of accurately measuring ambient air temperatures over a range of 10 to 30 °C to the nearest 0.1 °C. This thermometer should be traceable with an accuracy of 0.1 °C to a National Institute of Standards and Technology (NIST)-certified thermometer or an American Society for Testing and Materials (ASTM) thermometer.
- A barometer capable of accurately measuring barometric pressure over a range of 500 to 800 mm Hg (66 to 106 kilopascals [kPa]) to the nearest millimeter of mercury (Hg) and

referenced at least annually to a standard of known accuracy within  $\pm 5$  mm Hg. For laboratory measurements, a Fortin-type, mercury-column barometer is appropriate. For field measurements, a portable, aneroid barometer (e.g., a climber's or engineer's altimeter) is appropriate.

- Flow rate transfer standards capable of accurately measuring the total, fine, and coarse flow rates of a dichotomous sampler. Tables 2-1 and 2-2 (Section 2.0, "Calibration Procedures") present a variety of recommended transfer standards, their optimum flow ranges, and support equipment necessary for determining these flow rates. For the most commonly available commercial sampler, flow rate transfer standards capable of accurately measuring flow rates from 12.0 to 19.0 L/min and 1.4 to 1.9 L/min are required. The transfer standard calibration relationship must be referenced annually and be within ±2 percent of the NIST-traceable primary standard.
- An adapter of the correct dimensions that will connect the transfer standard outlet to the dichotomous sampler inlet and form a leak-proof seal. All interconnecting tubing should be flexible and crimp-resistant.
- Miscellaneous calibration supplies include a 9.53-mm (3/8-in.) or 6.35-mm (1/4-in.) Swagelok cap. If a soap film flowmeter is used, a rubber stopper with a tubing adapter is necessary to be able to measure flows under vacuum. The stopper may be obtained through a local scientific supply company.

## 1.1.3 QC Flow-Check Device

A QC flow check device is required for routine operation of the dichotomous sampler. Using calibrated orifice devices is a simple, accurate method for determining flow rates during routine operations.

The QC flow check devices can be fabricated in-house or ordered through a manufacturer. Figure 1.2 provides orifice device dimensions for both total and coarse flows. In the total flow range, the orifice must have an NIST-traceable calibration and be able to accurately measure flows between 12.0 and 19.0 L/min. In the coarse flow range, the orifice must have an NIST-traceable calibration and be able to accurately measure flows between 1.4 and 1.9 L/min. The calibration relationships must be referenced annually and be within  $\pm 2$  percent, of the NIST-traceable primary standard. As a minimum, both the total and coarse flows should be checked.

#### 1.1.4 Audit Equipment

The equipment needed for auditing is similar to the calibration equipment; however, the audit orifice transfer standard MUST be a different device from the one that is used for routine calibration and flow checks.



Figure 1.2. Orifice dimensions to provide approximately 1 in. of water pressure drop at 16.7 L/min (all dimensions in inches).

# 1.2 Procurement Prerequisites—Laboratory Operations

#### 1.2.1 Filter Media

No commercially available filter is ideal in all respects. The sampling program should determine the relative importance of certain filter evaluation criteria (e.g., physical and chemical characteristics, ease of handling, cost). The reference method presents basic criteria that must be met regardless of the filter type selected. These are as follows:

- Collection efficiency—Greater than 99 percent as measured by dioctylphalate (DOP) test (ASTM 2986) with 0.3-µm particles at the sampler's operating face velocity.
- Integrity— $\pm 5 \ \mu g/m^3$  (assuming sampler's nominal 24-h air sample volume), measured as the PM<sub>10</sub> concentration equivalent corresponding to the difference between the initial and final weights of a random sample of test filters when weighed and handled under simulated sampling conditions (equilibration, initial weighing, placement on inoperative sampler, removal from a sampler, re-equilibration, and final weighing), but have no air passed through them (i.e., filter blanks).
- Alkalinity—Less than 25 microequivalents/gram of filter as measured by the procedure given in "Measuring Alkalinity of Filters," which is presented as an appendix to *Quality Assurance Guidance Document 2.12* (Monitoring PM<sub>2.5</sub> in Ambient Air Using Designated Reference or Class I Equivalent Methods). The measurement is to be preceded by at least

two months' storage in a clean environment (free from contamination by acidic gases) at room temperature and humidity.

**Note**: Some filters may not be suitable for use with all samplers. Due to filter handling characteristics or rapid increases in flow resistance due to episodic loading, some filters, although they meet the above criteria, may not be compatible with the model of sampler chosen. It would be prudent to evaluate more than one filter type before purchasing large quantities for network use.

# 1.2.2 Filter Protection

Filter support cassettes (see Figure 1.3) are required for sampling with most dichotomous samplers and may be purchased through the manufacturer or a local scientific supply company. A sufficient number of cassettes must be purchased to allow insertion and removal of the filters in the laboratory. Under no circumstances should filter changes be attempted in the field.

Filter media (particularly the 37-mm [1.5-in.],  $2-\mu m$  pore size filter used with most commonly available commercial samplers) are especially delicate and easily damaged. Postsampling particle loss and filter damage will occur if proper handling procedures are not followed. To ensure the integrity of the sample, some type of protective covering is required for sample recovery and laboratory analysis. A plastic petri dish is recommended (Figure 1.3). The dish should be of comparable size (large enough to allow easy removal of the filter, yet small enough to prevent excess movement within the petri dish) and have a tight-fitting lid to prohibit damage or loss of particles during transportation to the analytical laboratory. A label can be affixed to the dish to allow proper documentation when sampling. A sufficient number of petri dishes must be

available to provide (1) protection for the filter in transportation to and from the monitoring location, and (2) storage of an exposed filter for subsequent gravimetric or chemical analysis.

# 1.2.3 Laboratory Equipment

The analytical balance must be suitable for weighing the type and size of the dichotomous filters used. The range and sensitivity are dependent upon routine tare weights and expected loadings. An analytical balance with a minimum resolution of 1 µg and a precision of 1 µg is recommended. A minimum sensitivity of  $\pm 1$  µg, and an accuracy of  $\pm 4$  µg at zero and  $\pm 2$  µg at 10 mg, is required. The balance must be calibrated at installation and recalibrated as specified by the manufacturer, but no less than once per year.





Prior to their weighing, filters must be conditioned in an environment where the mean relative humidity (RH) is between 20 and 45 percent and controlled within  $\pm 5$  percent, and mean temperature is between 15 and 30 °C and controlled within ~3 °C. Temperature and RH readings must be recorded daily, either manually or by hygrothermograph. Among the options available to ensure compliance with the reference method specifications are a sling psychrometer and a calibrated precision thermometer (capable of measuring temperatures over a range of 10 to 30 °C [283 to 303 K] to the nearest  $\pm 0.1$  °C) that is traceable with an accuracy of 0.1 °C to a NIST-certified thermometer or an ASTM thermometer.

Regardless of the filter media, all filters must be handled with nonserrated forceps and gloves. This will help eliminate interference with body oils, hydroscopic particles, and static electricity during weighing operations. These forceps can be obtained with a balance weight kit from a scientific supply company.

It is impossible to present a complete procurement package that would provide for unexpected contingencies in any monitoring network. Therefore, each agency must determine the extent of its in-house inventory and the items that should be ordered before sampling can begin. The agency must also be prepared to order any additional equipment required over and above that outlined in this section.

#### 1.2.4 Mass Reference Standards

Mass reference standards must be certified as being traceable to NIST mass standards. Additionally, they must have an individual tolerance of no more than 0.025 mg. Examples of mass reference standards that meet these specifications are American National Standards Institute/ American Society for Testing and Materials (ANSI/ASTM) Classes 1, 1.1, and 2. The mass reference standards must be recalibrated on a regular basis (e.g., yearly) at a NIST-accredited State weights and measures laboratory or at a calibration laboratory that is accredited by the national Voluntary Laboratory Accreditation Program (NVLAP), which is administered by NIST. The recalibration frequency is to be determined from records of previous recalibrations of these standards.

Note that the microbalance's resolution and precision are better than the tolerance of the most accurate classes of mass reference standards. Accordingly, the accuracy of the gravimetric analysis is limited by the tolerance of the standards rather than the balance's characteristics.

Two separate sets of mass reference standards are recommended. Working calibration standards should be used for routine permeation device weighing and should be kept next to the microbalance in a protective container. Laboratory primary standards should be handled very carefully and should be kept in a locked compartment. The working standards should be compared to the laboratory primary standards every 3 or 6 months to check for mass shifts associated with handling or contamination. The current masses of the working standards as compared to the laboratory primary standards should be recorded in a laboratory notebook and should be used to check the calibration of the microbalance.

Always use smooth, nonmetallic forceps for handling mass reference standards. The standards are handled only with these forceps, which are not used for any other purpose. Mark these forceps to distinguish them from the forceps used to handle permeation devices. Handle the standards carefully to avoid damage that may alter their masses.

# 2.0 Calibration Procedures

Before a  $PM_{10}$  monitoring program is undertaken, all sampling and analysis equipment must be properly calibrated. Careful, accurate calibrations of sampling instrumentation and associated equipment provide the backbone for any monitoring network. The requirements specified in this section may serve as initial acceptance checks. All the required data and calculations should be recorded in a calibration logbook or on calibration data sheets. A separate section of the logbook should be designated for each apparatus and sampler used in the program.

According to 40 CFR Part 50, Appendix M, the  $PM_{10}$  dichotomous sampler's flow rate must be calibrated in terms of actual volumetric flow rates (QA). The mass concentration of  $PM_{10}$  in ambient air is computed as the total mass of collected particles in the  $PM_{10}$  size range divided by the actual volume of air sampled. The mass concentration is expressed in micrograms per actual cubic meter ( $\mu g/m^3$ ).

Although the basic principle of operation for the dichotomous sampler is presented in the Introduction, more detailed information regarding calibration and operational procedures can be found in the instrument manufacturer's manual. Equipment calibration requirements are summarized in Table 2-1.

This section presents the following aspects of calibration procedures:

- A discussion of flow rate measurements and their applicability in a  $PM_{10}$  monitoring program (Section 2.1)
- Basic calibration procedures, calculations, and rotameter "set point" adjustments for the dichotomous sampler (Section 2.2)
- Recommended transfer standards and calibration equipment (Section 2.2)
- Sampler calibration frequency requirements (Section 2.3).

# 2.1 Discussion of Flow Rate Measurement and General Aspects of PM<sub>10</sub> Sampler Calibration

As discussed in the Introduction, a dichotomous sampler consists of three basic components: a specially designed inlet, a size fractionating virtual impactor, and a flow controlling system. The particle size discrimination characteristics of both the inlet and the virtual impactor depend critically on specified air velocities; a change in velocity will result in a change in the nominal particle size collected. For this reason, it is imperative that the flow rate through the sampler be maintained at a constant value that is as close as possible to the design flow rates. The design flow rates for a given sampler are specified in the sampler's instruction manual. The manual may also provide tolerance or upper and lower limits within which the sampler flows must be maintained. If the tolerance is not specified by the manufacturer, it should be assumed to be  $\pm 10$  percent. For example, if no tolerance is given and the design inlet flow rate is specified as 16.7 L/min, the acceptable flow rate range would be 15.0 to 18.4 L/min.

# **TABLE 2-1. EQUIPMENT CALIBRATION REQUIREMENTS**

Equipment	Frequency and method of measurement	Acceptance limits	Action if requirements are not met
Sampler	Calibrate with certified transfer standard upon receipt, after maintenance on sampler, and any time audits or flow checks deviate more than $\pm 7\%$ from the indicated flow rate or $\pm 10\%$ from the design flow rate.	Indicated flow rate = true flow rate ±4%.	Recalibrate.
Flow-rate transfer standard	Check upon receipt and at least at 1-yr intervals against primary standard; recalibrate or replace if damage is evident.	Indicated flow rate from previous calibration = actual flow rate $\pm 2\%$ .	Adopt new calibration curve.
On/off timer	Check at purchase and routinely on sample recovery days.	±30 min/24 h.	Adjust or repair.
Elapsed-time meter <sup>a</sup>	Compare with a standard timeplace of known accuracy upon receipt at 6-month intervals.	±2 min/24 h.	Adjust or replace time indicator to attain acceptance limits.
Analytical balance	Gravimetric test-weighing at purchase and during periodic calibration checks; use three to five standard weights covering normal range of filter weights.	Sensitivity = 1 μg Precision = 1 μg	Have balance replaced and/or recalibrated.
Relative humidity indicator	Compare with readings of wet/dry bulb psychrometer upon receipt and at 6-month intervals.	Indicator reading = psychrometer reading $\pm 6\%$ .	Adjust or replace to attain acceptance limits.
Mass reference standards	Working standards checked every 3 to 6 months against laboratory primary standards.	Standards bracket weight of filter. Individaul standard's tolerance less than 25 µg. Handle with smooth, nonmetallic forceps.	Obtain proper standards or forceps.

<sup>a</sup>Optional with a continuous flow rate recorder. <sup>b</sup>Zero and 10-µg weight checks are internal standards of the analytical balance.

## 2.2 Sampler Calibration

This section presents flow rate calibration procedures for the most common commercially available dichotomous samplers. Calibration procedures may have to be adapted for other sampler models.

The dichotomous sampler operates at a total actual flow rate of 16.7 L/min. To ensure correct fractionation of particles at the inlet, this flow rate must be maintained within  $\pm 10$  percent of 16.7 L/min. The coarse flow rate is approximately 10 percent of the total, or 1.67 actual L/min. It must also be maintained to ensure correct fractionation within the sampler's secondary separation system.

Accurate calibration data for each dichotomous sampler are essential for the following:

- To determine sampler flow rate set points
- To establish sampler flow rate control limits
- To calculate sampler flow rate during routine QC field flow checks and QA performance audits
- To calculate total sample volume for the computation of  $PM_{10}$  mass concentrations.

Calibration of the sampler rotameters must be traceable to NIST standards. A primary standard is used to calibrate a transfer standard, which in turn is used to calibrate the sampler rotameters.

Several commercially available transfer standards can be used in calibrations. Tables 2-2 and 2-3 list recommended standards, their applicable flow ranges, and the equipment necessary to perform sampler calibrations. The following are essential considerations in choosing a transfer standard for subsequent rotameter calibrations:

- The transfer standard must be traceable to NIST through the calibration procedures referenced.
- The transfer standard must be calibrated in the appropriate flow range. A minimum range of 12 to 19 L/min (total) and 1.4 to 1.9 L/min (coarse) is recommended.

Establish a calibration relationship (e.g., an equation or family of curves) such that traceability to the primary standard is accurate to within 2 percent over the expected range of ambient conditions (i.e., temperatures and pressures) under which the transfer standard will be used. Recalibrate the transfer standard periodically.

**Note:** If the transfer standard has been calibrated in terms of EPA standard conditions (25 °C or 298 Kelvin [K = °C + 273], 760 mm Hg or 101 kP<sub>a</sub>), indicated flow rates for each rotameter setting **must** be corrected to actual flow rates (Q<sub>a</sub>) to determine the sampler's set point.

As indicated in Tables 2-2 and 2-3, each transfer standard has certain characteristics. The operating agency should carefully choose the method that best utilizes equipment on hand and minimizes difficulties in establishing traceability.

<b>ND FINE FLOW RATES</b>	Calibration equation <sup>b,c</sup>	$(\Delta H_2 O) (CF) = Q_a$ where $\Delta H_2 O$ = pressure drop	(Volts) (CF) = $Q_{std}$	<u>Volume</u> = Q <sub>a</sub>	$\begin{bmatrix} \Delta H_2 O T_m \\ P_m M_m \end{bmatrix}^{1/2} (CF) = Q_a$ where $T_m = upstream absolute temperature M_m = upstream absolute pressure$	01 kPa) K). = correction factor. to the nearest ±1 mm Hg. ust be able to detect a flow change of 1% and
OCIATED EQUIPMENT-TOTAL #	Comments	Should have filtered air entering LFE. Subject to fluctuations due to temperature changes. Manometer must be used in its temperature range. Must equilibrate.	Recommended liquid-crystal display (LCD) for outdoor use. Must equilibrate in ambient conditions.	Should time through five revolutions. Repeat each timing three times.	Good only in range ΔP ≤ 8 in.	oler flow. $(Q_a)$ : where $P_a = \arctan pressure$ $P_{std} = \arctan pressure$ $T_a = \arctan pressure$ $T_{std} = 1$ $T_{std} = 1$ $T_{s$
TANDARDS AND ASS	Equipment	LFE Thermometer/barometer <sup>d</sup> Manometer <sup>e</sup> Filters Adapter	MFM Thermometer/barometer <sup>d</sup> Filters Adapter	DGM Thermometer/barometer <sup>d</sup> Stopwatch <sup>f</sup> Filters Adapter	Orifice Thermometer/barometer <sup>d</sup> Manometer <sup>e</sup> Fitters Adapter	H <sub>2</sub> O flow resistance to the samp w rate (Q <sub>std</sub> ) to actual flow rate ( ary from those presented due to the manometer range necessary the manometer range necessary ne intervals of 30 s to several m
RECOMMENDED S	Optimum flow range Q <sub>a</sub>	12.0 to 19.0 L/min	12.0 to 19.0 L/min	) 12.0 to 19.0 L/min	12.0 to 19.0 L/mim	ot cause more than 8 in. of to convert from standard flo $\mathbf{Q}_{a} = \mathbf{Q}_{stat} \begin{bmatrix} \mathbf{T}_{a} \\ \mathbf{P}_{a} \end{bmatrix} \begin{bmatrix} \mathbf{F}_{stat} \\ \mathbf{F}_{stat} \end{bmatrix}$ termining flow rates may vi- easuring temperature to the easuring temperature to the E a in. of H <sub>2</sub> O.
TABLE 2-2. F	Transfer standard <sup>a</sup>	LFE (laminar flow element)	MFM (mass flowmeter)	DGM 10 L/rev (dry gas meter	Orifice	<sup>a</sup> Transfer standard should n <sup>b</sup> Use the following equation <sup>c</sup> Calibration equations for de <sup>d</sup> Thermometer capable of m <sup>e</sup> The design or size of the Lf represent a flow resistance <sup>f</sup> Stopwatch or timer capable

TABLE 2-	<b>3. RECOMMENDED</b>	STANDARDS AND ASS	OCIATED EQUIPMENT-CO	ARSE FLOW RATE
Transfer standard <sup>a</sup>	Optimum flow range Q <sub>a</sub>	Equipment	Comments	Calibration equation <sup>b,c</sup>
LFE (laminar flow element)	1 to 2 L/min	LFE Thermometer/barometer <sup>d</sup> Incline manometer 0 t0 2.0 in., scale to the hundreths <sup>e</sup> Filters Adapter	Should have filtered air entering LFE. Subject to fluctuations due to temperature changes. Manometer must be used in its temperature range. Must equilibrate.	$(\Delta H_2O)$ (CF) = $Q_a$ where $\Delta H_2O$ = pressure drop
MFM (mass flowmeter)	1 to 2 L/min	MFM Thermometer/barometer <sup>d</sup> Filters Adapter	Recommended liquid-crystal display (LCD) for outdoor use. Must equilibrate in ambient conditions.	(Volts) (CF) = $Q_{std}$
DGM 1 L/rev (dry gas meter)	1 to 2 L/min	DGM Thermometer/barometer <sup>d</sup> Stopwatch <sup>f</sup> Filters Adapter	Should time through five revolutions. Repeat each timing three times.	$\frac{Volume}{Time} = Q_a$
Orifice	1 to 2 L/min	Orifice Thermometer/barometer <sup>d</sup> Manometer <sup>e</sup> Filters Adapter	Good only in range ΔP ≤ 4 in.	$\begin{bmatrix} \Delta H_2 O T_m \\ P_m M_m \end{bmatrix}^{1/2} (CF) = Q_a$ where $T_m = upstream absolute temperatureM_m = molecular weight of gas.$
SFFM (soap film flowmeter)	0 to 2 L/mim	SFFM Stopwatch <sup>f</sup> Plug with adapter Filters	Caution—can break. Flow in Q <sub>a</sub> . Three timings. Flow rate in terms of actual conditions.	$\frac{\text{Volume}}{\text{Time}} \begin{bmatrix} P_a - (1 - \text{RH}) P_{H_2O} \\ P_a \end{bmatrix} = Q_a$ where $\begin{array}{l} \text{RH} \\ \text{RH} \\ \text{P}_{H_2O} \\ \text{P}_{H_2O} \\ \text{Ph}_{2O} \\ \text{Ph}_{2O} \end{array} = \text{vapor pressure of water at } T_a. \end{array}$
<sup>a</sup> Transfer standard should not <sup>b</sup> Use the following equation to	cause more than 8 in. of H <sub>2</sub> O convert from standard flow rat	flow resistance to the sampler flow te $(\mathbf{Q}_{std})$ to actual flow rate $(\mathbf{Q}_a)$ :		
	$\mathbf{Q}_{\mathbf{a}} = \mathbf{Q}_{\text{std}} \begin{bmatrix} \mathbf{T}_{\mathbf{a}} \\ \mathbf{P}_{\mathbf{a}} \end{bmatrix} \begin{bmatrix} \mathbf{P}_{\text{std}} \\ \mathbf{T}_{\text{std}} \end{bmatrix}$	Ţa un	iere = actual pressure td = standard pressure (760 mm Hg or 1 = actual temperature d = standard temperature (25 °C or 298	01 kPa) K).
<ul> <li>Calibration equations for dete</li> <li><sup>d</sup> Thermometer capable of mea</li> <li><sup>e</sup> The design or size of the LFE represent a flow resistance &lt;<i>t</i></li> <li><sup>f</sup> Stopwatch or timer capable of</li> </ul>	mining flow rates may vary fr suring temperature to the nea or orifice will determine the m š in. of H <sub>2</sub> O. · accurately measuring time in	om those presented due to the trar rest ±1 °C. Barometer capable of <i>i</i> anometer range necessary and th tervals of 30 s to several minutes t	nsfer standard calibrations relationship. CF accurately measuring barometric pressure e resolution. The manometer resolution mi o nearest 0.1 s.	<sup>c</sup> = correction factor. to the nearest ±1 mm Hg. ust be able to detect a flow change of 1% and

Regardless of the transfer standard employed, a leak-tight adaptive device must be used to connect the transfer standard to the sampler inlet. Figure 2.1 illustrates such an adapter. These may be purchased commercially or fabricated in-house. Obviously, the corresponding outlet on the transfer standard will determine whether a pipe thread or tube fitting will be attached.

Tables 2-2 and 2-3 present only the basic apparatus necessary to perform calibrations. In addition to those listed, the operator will need a few miscellaneous supplies. These include a 9.53-mm (3/8-in.) Swagelok cap, 6.35-mm (1/4-in.) Swagelok cap, and hand tools.

A station logbook or calibration data sheet must be used to document calibration information. This information includes, but is not limited to, instrument and transfer standard model and serial numbers, transfer standard traceability and calibration information, ambient temperature and pressure conditions, and the collected calibration data (rotameter units versus indicated flow rate).

# 2.2.1 Precalibration System Check

Procedures for the precalibration system check are as follows:

- 1. Place a pair of filters into the dichotomous sampler filter holders. Filters used for flow rate calibrations should not be used for subsequent sampling.
- 2. Remove the sampler's inlet. Turn on the sampler and allow it to warm up to full operating temperature (at least 5 min).
- 3. While the sampler is energized, slowly close off the inlet tube with a rubber stopper or duct tape and observe the total vacuum gauge. If the sampler is equipped with an overload feature, it should shut down the system when approximately 15 in. of vacuum is reached.
- 4. If the sampler is equipped with the overload feature, disconnect. Next, perform a system leak check by opening both rotameters completely and sealing the inlet tube with a rubber stopper or duct tape. When a maximum indication on the **total** vacuum gauge is reached, shut off power to the unit, record the maximum reading on a data sheet, and observe the rate of decline in the readings of the vacuum gauges.

**Note:** Leak-free systems should indicate a vacuum of 10 to 15 in. or



Figure 2.1 Initial adapter that may be used to connect the transfer standard to the sampler's inlet tube.

more, and the rate of decline to 0 in. indication should require 60 s or more. If these conditions are not met and the control module was successfully leak-tested previously, a leak exists either in the interconnecting tubing or in the sample module.

5. If applicable, reconnect the overload feature. Conduct a pump performance check. Open the inlet tube and apply power to the unit. When stable flow is achieved, adjust both rotameter control valves to 90 percent of the rotameter scale. Observe the total vacuum gauge indication.

**Note**: Consult manufacturer's instruction manual for minimum vacuum indication. Readings lower than specified vacuum readings indicate possible pump diaphragm or reed valve problems, which should be investigated and corrected before continuing with the calibration.

#### 2.2.2 Total Rotameter Calibration

Procedures for calibrating the total rotameter are as follows:

- 1. Set up calibration system as illustrated in Figure 2.2. The inlet of the transfer standard is open to the ambient air; the outlet of the transfer standard is connected to the inlet tube of the dichotomous sampler.
- 2. Turn on the sampler and allow it to warm up to normal operating temperature (at least 5 min). If an electronic transfer standard is used, it must also equilibrate before proceeding with the calibration.
- 3. Adjust the total flow control valve to a setting representing a flow rate in the acceptable range specified for the inlet (i.e., 12 to 19 L/min). Adjust the coarse flow control valve to indicate a nominal flow of 1.67 L/min.
- 4. Read the following parameters and record them on a data form (Figure 2.3) or in a logbook:
  - Ambient temperature (T<sub>a</sub>), K
  - Barometric pressure (P<sub>a</sub>), mm Hg or kP<sub>a</sub>
  - Transfer standard (TS) readings, volts,  $\Delta H_2O$ , timings, etc.



Figure 2.2. Calibration assembly and dichotomous sampler with transfer standard connected.

Dicho	tomous Samp	ler Calibratior	n Data Sheet	
Station Location Raleigh	, NC Date	9/3/97 Unu	sual Conditions	none
Sampler Model 244	E S/N	619	EPA #	387265
P <sub>a</sub> 748 mm Hg P <sub>s</sub> 751	mm Hg T <sub>a</sub>	17 °C or	K T <sub>s</sub>	20 °C or K
Leak check, maximum vacuum	19 in.	Notes	overload feat	ure disconnected
Total Trans. Std. Model	MFM S/N	2913	Cal. Date	7/1/89
Q <sub>a</sub> Cal. relations	hip m = 0.942	b = 0.00	r = 0.999	
Coarse Trans. Std. Model	SFFM S/N	<i>V</i> K1	Cal. Date	12/11/88
Q <sub>a</sub> Cal. relations	hip m = 1.0	p = 0	r=no c	correction (1000 cc)
Total Transfe Cal. Indica Point (TS) (	er Std. ation F vdc)	TQ <sub>a</sub> Iow Rate (L/min)	Rotameter Response (I)	Corr. Response I (T <sub>a</sub> /P <sub>a</sub> ) <sup>1/2</sup> (AC)
90% 20.6	60	19.4	15.3	9.53
75% 19.0	07	17.9	14.2	8.84
60% 16.	17	15.8	13.3	8.28
40% 15.	57	14.7	12.4	7.72
20% 14.	57	13.7	11.7	7.29
Coarse Transfe Cal. Indica Point (TS) (	er Std. ation F min)	CQ <sub>a</sub> low Rate (L/min)	Rotameter Response (I)	Corr. Response I (T <sub>a</sub> /P <sub>a</sub> ) <sup>1/2</sup> (AC)
90% 0.44	97	2.01	12.2	7.60
75% 0.58	89	1.70	10.5	6.54
60% 0.65	24	1.60	9.5	5.92
50% 0.6	19	1.47	9.0	5.60
20% 0.7	12	1.40	8.5	5.29
Sampler Cal. Relationship ( $Q_a$ , x Total: m = 0.379 b = 2.15 Today's TSP 13.6 Seasonal TSP 13.6 TSP = {[m (16.7) + b] [( $P_a$ or $P_s$ / CSP = {[m (16.7) + b] [( $P_a$ or $P_s$ / Operator <i>Robert Murdoch</i>	x-axis; corrected r = 0.995 Today's CS Seasonal C T <sub>a</sub> or T <sub>s</sub> ) <sup>1/2</sup> ]} T <sub>a</sub> or T <sub>s</sub> ) <sup>1/2</sup> ]}	recorder respon Coarse: m = P <u>10.1</u> SP <u>10.1</u>	se, AC, y-axis). 3.806 b =(	0.04 r = <u>0.995</u>

#### Figure 2.3. Example dichotomous sampler calibration data sheet.

• Sampler total rotameter indication (I), arbitrary units.

**Note**: Rotameter settings for subsequent sampling periods may need to be adjusted to account for day-to-day variations in  $T_a$  and  $P_a$ . When such adjustments are necessary, they should be based on nearby daily measurements of  $T_a$  and  $P_a$  if possible. However, the seasonal average temperature ( $T_s$ ) and the seasonal average pressure ( $P_s$ ) may be used to avoid daily rotameter adjustments. Figure 2.3 includes a procedure for calculating the seasonal total rotameter set point (TSP) and the seasonal coarse rotameter set point (CSP).

5. Repeat procedure for at least two additional rotameter settings representing flow rates in the acceptable range for the inlet. For each calibration point, record the rotameter indication and corresponding transfer standard output.

## 2.2.3 Coarse Rotameter Calibration

- 1. Turn off the sampler, disconnect the fine flow vacuum line (9.53-mm [3/8-in.] o.d. line), and cap the fine flow outlet port with a 9.53-mm (3/8-in.) Swagelok cap (see Figure 2.4). This step keeps the fine flow line open to the vacuum pump. It is recommended that a particle-free filter be attached to the detached fine flow line to prevent particles from entering the system. Install the coarse flow rate transfer standard.
- 2. Energize the sampler and the transfer standard (if electronic). Allow both to warm up again to full operating temperature.
- 3. Adjust the coarse rotameter flow-control valve to an approximate value of 90 percent of the rotameter scale. Adjust the total flow control valve to indicate a nominal flow of 16.7 L/min.
- 4. Read the following parameters and record them on a data form (Figure 2.3) or in a logbook:
  - Ambient temperature (T<sub>a</sub>) if variation has occurred, K
  - Barometric pressure (P<sub>a</sub>) if variation has occurred, mm Hg or kPa
  - Transfer standard (TS) readings, volts,  $\Delta H_2 O$ , timings, etc.
  - Sampler coarse rotameter indication (I), arbitrary units.
- 5. Repeat procedure for rotameter settings representing flow rates of 75, 60, 40, and 20 percent of the established operating range (1.4 to



Figure 2.4. calibration assembly and dichotomous sampler set up to calibrate the coarse flow rotameter.

1.9 L/min). For each calibration point, record the rotameter indication and corresponding transfer standard output.

6. Turn off sampler, and reconnect the fine flow line and the sampler's inlet.

### 2.2.4 Calibration Calculations

Gather together all the calibration data, including the transfer standard calibration information and the dichotomous 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 have already been obtained.

- 1. Verify that the transfer standard calibration equation is current and traceable to an acceptable primary standard.
- 2. Calculate  $Q_a$  for each calibration point as determined by the transfer standard calibration equation.

**Note**: It may be necessary to correct the indicated transfer standard flow rates from  $Q_{std}$  to  $Q_a$ . This can be accomplished using Equation (1):

$$Q_a = Q_{std}(T_a/P_a)(P_{std}/T_{std})$$
(1)

where

 $Q_a$  = flow rate at actual conditions, L/min

Q<sub>std</sub> = flow rate corrected to standard temperature and pressure (25 °C, 298 K; 760 mm Hg, 101 kPa), L/min

 $T_a$  = ambient temperature, K (K = °C + 273)

 $P_a$  = ambient barometric pressure, mm Hg or k $P_a$ 

 $P_{std}$ ,  $T_{std}$  = standard barometric pressure and temperature, respectively.

3. Calculate and record the total and coarse rotameter actual corrections (AC) for each calibration point as

$$AC = I(T_a/P_a)^{1/2}$$
(2)

where

AC = actual correction

- I = rotameter response, arbitrary units
- $T_a = ambient temperature, K$
- $P_a$  = ambient barometric pressure, mm Hg or k $P_a$ .
- 4. On a sheet of graph paper, plot the sampler-corrected total rotameter units (y-axis) versus the corresponding calculated transfer standard total flow rates (x-axis) to obtain the dichotomous sampler total flow rate calibration relationship.

5. Repeat Step 4, plotting corrected coarse rotameter units vs. the corresponding calculated coarse flow rates.

Because the determination of the sampler's average operational flow rate  $(Q_a)$  during a sample period depends on the ambient average temperature and pressure, use of 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  $y = AC = I(T_a/P_a)^{1/2}$  and  $x = Q_a$  so that the model is given by:

$$AC = m[Q_a(transfer standard)] + b.$$
(3)

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 0.5 L/min for total or 0.05 L/min for coarse rotameter calibrations from the value predicted by the regression equation. Plot the regression line on the same graph paper that has the individual calibration points.

6. For subsequent sample periods, the sampler's average actual operational flow rate  $TQ_a$  or  $CQ_a$  is calculated from the calibration slope and intercept using Equation (5):

$$\overline{\mathrm{TQ}_{\mathrm{a}}}$$
 or  $\overline{\mathrm{CQ}_{\mathrm{a}}} = 1/\mathrm{m}[\overline{\mathrm{I}}(\mathrm{T}_{\mathrm{av}}/\mathrm{P}_{\mathrm{av}})^{1/2} - \mathrm{b}]$  (4)

where

 $\overline{TQ_a}$ ,  $\overline{CQ_a}$  = sampler total or coarse average flow rate, actual L/min

 $\overline{I}$  = average total or coarse rotameter response, arbitrary units

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

 $P_{av}$  = average ambient barometric pressure for the run day, mm Hg or k $P_a$ 

m = slope of the total or coarse flow rate calibration relationship

b = intercept of the total or coarse flow rate calibration relationship.

**Note**: The expression  $[\bar{I}(T_{av}/P_{av})^{1/2}]$  is the "y" term of linear regression equation: y = mx + b, or x = (y - b)/m.

**Note**:  $T_{av}$  and  $P_{av}$  readings may be recorded onsite or from a nearby U.S. National Weather Service station 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 elevation of the monitoring site and that of the airport. If ambient temperature and pressure readings are not available, seasonal average temperature ( $T_s$ ) and barometric pressure ( $P_s$ ) 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 °C and 40 mm Hg.

#### 2.2.5 Rotameter Set Point Adjustment Procedure

1. Calculate and record on the sampler's calibration data sheet total and coarse rotameter set points using temperatures and pressures for the calibration date and the season.

$$\Gamma SP = \{ [m(16.7) + b](P/T)^{1/2} \}$$
(5)

where

TSP = total rotameter set point, arbitrary units

16.7 = total flow rate, L/min

- P = barometric pressure, mm Hg or kPa (ambient or seasonal)
- T = temperature, K (ambient or seasonal)
- m = slope of the total flow rate calibration relationship
- b = intercept of the total flow rate calibration relationship.

$$CSP = \{ [m(1.64) + b] (P/T)^{1/2} \}$$

(6)

where

- CSP = coarse rotameter set point, arbitrary units
- 1.67 = coarse flow rate, L/min
  - P = barometric pressure, mm Hg or kPa (ambient or seasonal)
  - T = temperature, K (ambient or seasonal)
  - m = slope of the coarse flow rate calibration relationship
  - b = intercept of the coarse flow rate calibration relationship.

Adjusting the sampler rotameter to seasonal average conditions will help minimize data loss caused by exceeding the manufacturer's design condition specifications.

- Energize the sampler and allow it to warm up to operating temperature (3 to 5 min).
- Following the manufacturer's instructions, adjust the total rotameter until the sampler response indicates the total flow rate set point (TSP) as calculated for the calibration date.
- Following the manufacturer's instructions, adjust the coarse rotameter until the sampler response indicates the coarse flow rate set point (CSP) as calculated for the calibration date.
- Verify that the sampler will maintain these flow rates for at least 10 min. Turn off the sampler.
- The sampler can now be prepared for the next sample run day.

# 2.3 Sampler Calibration Frequency

To ensure accurate measurement of the  $PM_{10}$  concentrations, calibrate the sampler upon installation and then recalibrate it as follows:

• At least annually.

- After any repairs that might affect sampler calibration.
- If the field calibration flow check results exceed QC limits (±10 percent from the sampler's indicated flow rate).
- Whenever an audit indicates that the sampler is out of calibration (±10 percent from the sampler's indicated flow rate).

# 3.0 Field Operations

# 3.1 Siting Requirements

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  $PM_{10}$  monitoring project is to select a site where the collected sample mass is representative of the monitoring area.

Spatial and temporal scale considerations are important in dichotomous sampler siting. Spatial scales may range from a small (0.1- to 0.5-km<sup>2</sup>) area to large regional areas exceeding tens of hundreds of square kilometers. Whether the potential impact of particulate pollution is generated by a local or general source category will affect the decision on the size of the spatial monitoring scale. In addition, the siting of the samplers within a monitoring network should reflect whether the expected impact will be limited to a small area (a few city blocks) or extend to larger areas (metropolitan or rural).

With regard to the temporal scale, interest focuses on either an annual geometric mean concentration or a 24-h average concentration. Because siting of a dichotomous sampler requires that consideration be given to prevailing wind direction, a sampler sited for monitoring trends in air quality over a period of a year will not necessarily be ideal for measuring 24-h concentrations. Thus, the choice of temporal scale will also affect the sampler location.

Although spatial and temporal scales must be considered in site selection, the following guidelines should be observed regardless of the scale:

- The dichotomous sampler must have unobstructed air flow for a minimum of 2 m in all direction.
- The sampler inlet should be placed at a height of 2 to 15 m above ground level.
- If a dichotomous sampler is collocated with any other particulate sampler, the minimum spacing between sampler inlets must be 2 m and the maximum spacing must be 4 m. All inlet heights should be within 1 vertical meter of one another.

Complete siting requirements are outlined in 40 CFR Part 58, Appendix E. (See also Part 1, Section 6 of this volume of the handbook.)

Additional factors must be considered in determining where the actual sampler will be deployed. These include accessibility under all weather conditions, availability of adequate electricity, and security of the monitoring equipment.

A dichotomous sampler used for routine sampling must be situated where the operator can reach it safely regardless of 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, sample installation and recovery, flow checks, and audits) involves transporting supplies and equipment to and from the monitoring site. A dichotomous sampler will require a minimum continuous operating current of 3 to 5 A (120 V a.c., 60 Hz) and may require a higher startup current, which necessitates a slow-blow fuse. Although most dichotomous samplers are equipped with timers, there is often no recording device provided to indicate short-term power interruptions. This lack necessitates a stable power source for the monitoring site.

The security of the sampler itself depends largely 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.

## 3.2 Sampler Installation Procedures

- 1. On receipt of a dichotomous sampler from the manufacturer, visually inspect the sampler to ensure that all components are accounted for. Compare equipment delivered with the enclosed packing slip. Notify the manufacturer immediately of any missing or damaged equipment.
- 2. Before transporting the sampler to the field site, perform a quick laboratory check to determine if the sampler is operational. Energize the sampler and observe rotameter responses, vacuum gauges, and pump performance.
- 3. Carefully transport the sampler to the monitoring site.
- 4. Bolt down the sampling module to a secure mounting surface.
- 5. Install the control module. This module can be bolted down adjacent to the sampling module (no closer than 2 m), or it can be located remotely (e.g., inside a monitoring station). It is recommended that the control module be no more than 10 to 15 m away from the sampling module to avoid a pressure drop along the flow lines.
- 6. Connect the vacuum lines between the sampling module and the control module. First, hand-tighten the nuts on the tube connectors as much as possible, and then wrench-tighten them 1-1/4 revolutions. Be careful not to cross-thread the fittings.
- 7. Check all tubing for crimps, cracks, or breaks.
- 8. Plug the power cord into a line voltage outlet. The use of waterproof interlocking electrical connectors is recommended to ensure operator safety and to avoid shorts and/or power interruptions. Do not allow any electrical connections to be submerged during periods of inclement weather.
- 9. Perform a multipoint flow rate calibration as described in Section 2.

# 3.3 Sampling Operations

Sampling operations provided here are specific to one type of commercially available dichotomous sampler. Because operational procedures may vary among sampler models, the manufacturer's instrument manual should be consulted before the sampler is put into operation. Sampling procedure checks are summarized in Table 3-1.

Procedure	Frequency and method	Requirements	Action if require- ments are not met
Filter installation	Visually check each filter. Designate as coarse or fine on petri dish. Install filters one at a time. Hand-tighten knurled rings.	Filters are provided with ID numbers. Filters are tare- weighed and undamaged.	Void the filters; install substitute filter.
Sample validation and documentation	Visually check each sample and sample data sheet for completeness.	Sampling data, filter and sampler ID, station location, flow rates, sample time, and unusual conditions recorded on data sheet. Petri dishes marked to indicate coarse or fine filter.	Complete or correct the documentation; if unavailable, void the sample.
Post-sample inspection	Visually check each sample for tears, missing places, or leakage.	No evidence of filter damage or sampler malfunction.	Void the sample; correct the case of the malfunction.
Flow checks	Check flow rate at least monthly.	Sampler flow rate must be within $\pm$ 7% of the specified flow rate.	Determine cause of flow problem and correct. Calibrate the sampler.

# TABLE 3-1. SAMPLING PROCEDURE CHECK

#### 3.3.1 Filter Installation

Care must be taken to ensure that clean filters are not damaged before they are installed in a dichotomous sampler. Filter cassettes should be kept in their protective petri dishes (see Figure 1.3), and any damaged filters must be discarded.

The procedure used to install filters in a dichotomous sampler is presented here. Each dichotomous sampler is equipped with two filter holders, and the petri dish should be marked to indicate which filter will be used for coarse particle sampling, which will be used for fine particle sampling, the filter and sampler ID number, and the sampling run date.

- 1. Switch mechanical or digital timer to OFF.
- 2. The coarse-particle filter holder is the one with the 6.35-mm (1/4-in.) o.d. tubing, and the fine-particle filter holder is the one with the 9.53-mm (3/8-in.) o.d. tubing. As shown in Figure 3.1, the filter holders can also be distinguished by the fact that the coarse-particle filter holder is on the center line of the virtual impactor head and aerosol inlet, whereas the fine-particle filter holder is offset. Unscrew (by hand) the knurled filter-holder assembly underneath the receiver tube assembly. Install each cassette containing the preweighed filters in its respective filter holder. Do not attempt to install both filters simultaneously, as this could cause damage and/or transpose the coarse and fine filters. This transposition of filters is a common error found in the operation of dichotomous samplers. The lower half of the filter cassette, which goes over the screen, is also the side with the shorter distance (approximately 2.0 mm) to the filter surface. Each filter holder has an O ring that seals the

filter holder to the virtual impaction assembly. Visually ascertain that the O rings are present and secure. Do not sample without these O rings installed, as the system will no longer be leak-free. Tighten both knurled filter holder nuts by hand, making sure the nuts are not cross-threaded. Record on a data sheet, similar to Figure 3.2, the fine and coarse filter ID numbers.

- 3. Open the front cover of the control module by turning the knob latch counterlockwise. The cover is released by turning the indicator one-quarter turn counterclockwise and is locked by reversing this process.
- 4. Switch the mechanical or digital timer to ON. If the sampler has a digital timer/ programmer equipped with a POWER switch, turn on the vacuum pump. Allow the pump to run for at least 5 min to establish operating temperature conditions. While the sampler equilibrates, record on





the data sheet station documentary information (i.e., location, Aerometric Information and Retrieval System [AIRS] designation, sampler model, and serial number) and the run date of the sample. The sampler's calibration relationships and the total and coarse set points (TSP and CSP) should be recorded. Set point calculations are presented in Subsection 2.

- 5. Set the total flow rate by adjusting the rotameter to the calculated TSP value. Observe and record the total vacuum gauge indication. The vacuum gauge should show a pressure drop  $(\Delta P)$  of approximately 1 to 2 in. Hg for a 2-µm pore filter.
- 6. Set the coarse flow rate by adjusting the rotameter to the calculated CSP value. Observe and record the coarse vacuum gauge indication; it should read approximately zero. Turn off the sampler.
- 7. The sampler is now ready to sample. Set the master timer (according to the manufacturer's instructions) to energize the sampler for the next sampling period. Reset the elapsed time indicator to zero.
- 8. Close the front cover of the control module and visually inspect the monitoring site to ensure that all sampling components (sampling inlet and control module) are in readiness for the next run day.

# 3.3.2 Filter Recovery Procedure

1. After sampling, record the elapsed-time indicator value and energize the sampler. Allow the sampler to warm up to operating temperature and record the final total and coarse rotameter

Dichotomous Sampler Field Data Sheet
Station Location Cary, NC Date 12/1/97 AIRS Number 372346789
Sampler Model         244E         S/N         619         EPA #         387265
Filter ID Numbers:     Fine:     BR549     Coarse:     BR550
Total Cal. Relationship: $m = 0.379$ $b = 2.15$ $r = 0.995$
Coarse Cal. Relationship: $m = 3.806$ $b = -0.04$ $r = 0.995$
Vacuum Gauge Indications: Total Initial <u>1.5</u> Total Final <u>1.6</u> Coarse Initial <u>0</u>
Coarse Final 0
$P_{av}$ <u>741</u> mmHg $T_{av}$ <u>23</u> °C <u>296</u> K Elapsed Time Sampled <u>1445</u> min
TSP* 13.55 Final Total 13.5 Ave. Total $(\overline{I})$ 13.5 TO 16.84 L/min
$CSP^*$ 10.08 Final Coarse 10.0 Ave Coarse ( $\overline{I}$ ) 10.0 $\overline{CO}$ 1.67 L/min
$\frac{10.00}{10.00} = 10.00 = 10$
$[Q_a, CQ_a = 1/m [I(I_{av}/P_{av}))^{m/2} - b]$
Total Act. Volume $(TV_a) = TQ_a X$ min sampled = <u>24334</u> L $FQ_a = \underline{15.17}$ L
Coarse Act. Volume ( $CV_a$ ) = $CQ_a X$ min sampled = <u>2413</u> L $FQ_a = TQ_a - CQ_a$
Comments: Grass fire in adjacent field; a lot of smoke blowing away from
the sampler while on-site
Operator Lisa Wall
Laboratory Calculations
Filter Weights:
Fine: Gross Weight (Wg) 100.136 mg Coarse: Gross Weight (Wg) 105.067 mg
Tare Weight (Wt) 99.211 mg Tare Weight (Wt) 104.413 mg
Net Weight (Mf) 0.925 mg Net Weight (Mc) 0.654 mg
$PM_{10} \text{ Concentration} \qquad \frac{64.89}{\mu g/m^3}  \mu g/m^3 \qquad \mu g/m^3 = (Mf + Mc)(10^{\circ})/1 V_a$
*Total or coarse set points, initial rotameter response.



readings and the final total and coarse vacuum gauge indications on the data sheet. Turn the sampler off.

- 2. Reverse the filter installation procedure and remove each filter one at a time. Put the filter cassettes in their original marked plastic petri dishes. Verify that filter ID numbers match numbers recorded on the data sheet.
- 3. Calculate and record the total and coarse average rotameter readings as:

$$\overline{I} = (TSP \text{ or } CSP + IF)/2$$
 (7)

where

 $\overline{I}$  = average total or coarse rotameter response, arbitrary units

TSP, CSP = total or coarse rotameter set points, arbitrary units IF = indicated final total or coarse rotameter response, arbitrary units.

- 4. Record the average ambient temperature  $(T_{av} [K])$  and barometric pressure  $\sim P_{av}$  (mm Hg or  $kP_a$ )] for the run day on the field data sheet.
- 5. Calculate and record the total and coarse average actual flow rates (TQ<sub>a</sub> and CQ<sub>a</sub>), as determined by the sampler's calibration relationships.

$$\overline{\mathrm{TQ}}_{\mathrm{a}}$$
 or  $\overline{\mathrm{CQ}}_{\mathrm{a}} = 1/\mathrm{m}[\bar{\mathrm{I}}(\mathrm{T}_{\mathrm{av}}/\mathrm{P}_{\mathrm{av}})^{1/2} - \mathrm{b}]$  (4)

where

 $\overline{TQ_a}$ ,  $\overline{CQ_a}$  = sampler total or coarse average flow rate, actual L/min

 $\overline{I}$  = average total or coarse rotameter response, arbitrary units

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

- $P_{av}$  = average ambient pressure for the run day, mm Hg or kPa
- m = slope of the dichotomous sampler total or coarse calibration relationship
- b = intercept of the dichotomous sampler total or coarse calibration relationship.

Note: Refer to Section 5 for a description of  $T_{av}$  and  $P_{av}$  measurements.

- 6. Calculate the actual fine flow rate by subtracting the calculated  $Q_a$  coarse from the  $Q_a$  total, and record.
- 7. Observe conditions around the monitoring site; note any activities that may affect filter particle loading (paving, mowing, fire) and record this information on the field data sheet.

#### 3.3.3 Sample Validation

The following criteria have been established to assist the operator in determining whether or not a sample is valid.

#### 1. Timing

- All samplers must turn ON and OFF within 1/2 h of midnight.
- All samplers must operate for at least 23 but not more than 25 h (1,330 to 1,500 min).
- 2. Flow Rates

The average flow rates must be within 10 percent of 16.7 L/min (total) and 1.67 L/min (coarse) at actual conditions. If these limits are exceeded, investigate the cause. Use the following criteria as a basis for sample invalidation:

- Decreases in flow rate during sampling (due to mechanical failure) of more than 10 percent from the initial set point require a field calibration check (Section 3.4). If the sampler's calibration check indicates that the sampler flow was not within  $\pm 10$  percent of the designed flow, the sampler should be invalidated.
- If the sampler flow rate decreases because of heavy particulate loading on the filter, a postsampling check of the vacuum gauges will indicate increased vacuum. These filters should not be invalidated because they may indicate an episodic situation.
- Changes in flow rate calibration of more than 10 percent, as determined by a field calibration check, will invalidate all samples collected back to the last acceptable flow rate check. Recalibrate the sampler.
- 3. Filter Quality
  - Any filter that is obviously damaged (i.e., is torn, frayed, or has pinholes) should be invalidated.

#### 3.3.4 Sample Handling

#### 3.3.4.1 Handling of a Valid Sample

- 1. Calculate the total, coarse, and fine flow rates and complete the data sheet.
- 2. Promptly deliver the filter cassettes in their protective petri dish, accompanied by the completed data sheet, to the analytical laboratory.

#### 3.3.4.2 Handling of an Invalid Sample

- 1. Complete as much of the data sheet as possible and explain any omissions.
- 2. Mark "VOID" on the data sheet accompanying the filter and record in the site logbook.
- 3. **Do not discard** the filter.
- 4. Promptly deliver the filter cassettes in their petri dish and the data sheet to the analytical laboratory, where a final decision on sample validity will be made.

#### 3.3.4.3 Handling of a Questionable Sample

If uncertain whether or not a sample should be voided:

- 1. Complete as much of the data sheet as possible and explain any factors that may affect the sample validity.
- 2. Put a question mark in the upper right corner of the data sheet.
- 3. Record as "Questionable" in the site logbook.
- 4. Promptly deliver the filter cassettes in their petri dish and the data sheet to the analytical laboratory, where a final decision on sample validity will be made.

# 3.4 Operator's QC Field Calibration Check Procedure

For dichotomous samplers, a field calibration check of the total and coarse flow rates is recommended after each month of operation. The purpose of this check is to track the sampler calibration stability. Control charts presenting flow check data (indicated vs. observed) should be maintained. These charts provide a quick reference of instrument flow rate drift patterns and will indicate when flow limits ( $\pm 10$  percent variation from the indicated or design condition flow rate) have been exceeded. The field check is made by installing a measuring device (which is traceable to NIST and is calibrated within the range of the total or coarse flow rate) on the inlet of the sampler.

Calibration checks of the sampler flow rate require that the instrument be running. The following flow check procedures are specific to an orifice device. A variety of transfer standards may be used with this same procedure; however, necessary apparatus and subsequent calculations to determine the sampler's flow rates will vary.

#### 3.4.1 Field Check Apparatus

The following equipment is required for a field calibration check:

- A thermometer capable of accurately measuring ambient air temperatures over a range from 10 to 30 °C to the nearest 0.1 °C. This thermometer should be traceable with an accuracy of 0.1 °C to a NIST-certified thermometer or an ASTM thermometer.
- A barometer capable of accurately measuring ambient barometric pressure to the nearest ±1 mm Hg and referenced to an NIST or ASTM barometer within ±5 mm Hg at least annually
- Two calibrated orifice devices and calibration relationships (one for total and one for coarse)
- The sampler's calibration information
- Two clean flow check filters
- Dichotomous sampler flow check data sheet (Figure 3.3) or logbook.

#### 3.4.2 **Procedure for Field-Calibration Check**

 Insert clean filters (designated "flow check filters") into both the fine and coarse filter holders of the sampler as described in the operating procedure in Section 3.3.1. Flow check filters should never be used for subsequent sampling because particles larger than 10 µm can impact on the filter when the inlet is removed and bias the sample.

	Dichoton	nous Sampler	Flow Check F	)ata Sheet		
Station Location	Cary, NC	Date 1	2/2/97 AIRS I	Number 3723	46789	
- Sampler Model	244E	S/N	619	EPA #	3872	265
P <sub>a</sub> 742   mm Hg	T <sub>a</sub> 19 °C	С 292 K	Unusual Co	onditions none		
Orifice S/N 317 (	(Total) Orif	ice S/N 316 (c	oarse) (	Orifice Calibrati	on Date	10/1/89
Orifice Q <sub>a</sub> (Total)	Calibration Relat	tionship:	m = 24.69	91 b = +0.8	9 r=	0.999
Orifice Q <sub>a</sub> (Coarse	e) Calibration Re	lationship:	m = 2.493	3 b = +0.0	f r=	0.999
Sampler Total Ca	libration Relation	iship:				
m = <u>0.379</u> b =	+2.15 r =	0.995 Desig	gn TQ <sub>a</sub> 16.7	L/min, Ta	rget TSP	13.6
Sampler Coarse (	Calibration Relati	onship:		=		
m = 3.806 b =	-0.04 r =	0.995 Desig	n CQ <sub>a</sub> _ 1.67	L/min, Ta	arget CSP	10.1
	Orifice	Orifice Flow	Observed	Sampler**	QC Diff	erence***
Flow Rate Description	ΔH <sub>2</sub> O (in.)	Rate* (L/min)	Rotameter Reading	TQ <sub>a</sub> or CQ <sub>a</sub> (L/min)	L/min	%
Total Flow	0.95	15.99	13.6	16.70	0.71	4.4
Coarse Flow	0.97	1.58	10.1	1.67	0.09	5.7
*Orifice TQ <sub>a</sub> or 0 **Sampler TQ <sub>a</sub> or **** <b>QC % Differen</b>	$CQ_a = m[(\Delta^{-1}_2O)]$ r CQ_a = 1/m [(TS ce = [100] [(Sa	(T <sub>a</sub> /P <sub>a</sub> )] <sup>1/2</sup> +b SP or CSP) (T <sub>a</sub> /F mpler TQ <sub>a</sub> or C	$(P_a)^{1/2} - b]$ $(Q_a) - Orifice Flow Bate$	ow Rate		
Operator <i>Lisa U</i>	lall			J		



- 2. Turn on the sampler and allow it to warm up to operating temperature (approximately 5 min).
- 3. Read and record the following parameters on the sampler flow check data sheet (Figure 3.3):
  - Ambient temperature  $(T_a)$ , °C and K
  - Ambient barometric pressure  $(P_a)$ , mm Hg or kPa
  - Sampler S/N and model
  - Orifice S/Ns and calibration relationships
  - Date, location, and operator's signature
  - Sampler rotameter's target flow rates (16.7 and 1.6 L/min) and target set points (TSP, CSP).
- 4. Adjust both the total and coarse rotameters to their respective calculated set points (TSP, CSP).
- 5. Remove the inlet from the sampler, replace it with the flow check orifice device, and recheck the rotameter set points.
- 6. Observe the  $\Delta H_2O$  across the total flow orifice by reading the manometer deflection (at the bottom of the meniscus), and determine the corresponding flow rate from the orifice calibration data. Record both values (manometer deflection and corresponding flow rate) on the flow check data sheet. Using the sampler's calibration relationship, calculate the sampler's indicated total **actual** flow rate (TQ<sub>a</sub>) and record.
- 7. Turn the sampler off. Disconnect the fine flow vacuum line and cap the fine flow outlet port with a 9.53-mm (3/8-in.) Swagelok cap. This opens the fine flow to the vacuum pump. It is recommended that a particle-free filter be attached to the detached line to prevent particles from entering the system. Install the coarse flow rate orifice and turn the sampler on.
- 8. Observe the  $\Delta H_2O$  across the coarse orifice by reading the manometer deflection and determine the corresponding flow rate from the orifice calibration data. Record both values on the flow check data sheet. Using the sampler's calibration relationship, calculate indicated coarse **actual** flow rate (CQ<sub>a</sub>) and record.
- 9. Using the above information, calculate the QC percentage difference as:

QC % Difference = [100] 
$$\frac{(\text{Sampler TQ}_a \text{ or } \text{CQ}_a) - \text{Orifice flow rate}}{\text{Orifice flow rate}}$$
(8)

- 10. If the sampler flow rate is within 90 to 110 percent of the 16.7 L/min or 1.67 L/min flow rate, the sampler is operating properly. If these limits are exceeded, investigate and correct any malfunction. If necessary, recalibrate the sampler before sampling is resumed.
- 11. Turn off the sampler, remove the orifice device, replace the inlet, and reconnect the fine flow vacuum line.
- 12. Remove the filters from both fine and coarse filter holders.

13. Set up the sampler for the next sampling period according to the operating procedure in Section 3.3.1.

## 3.5 Documentation

The responsible persons should record the following information on the filter petri dish, the field data sheet, and in the logbook.

#### 3.5.1 Operator Who Starts the Sample

- 1. Mark on the filter petri dish:
  - Sampler ID number
  - Filter number
  - Sample date
  - Designation (e.g., whether it is a coarse [C] or fine [F] filter).
- 2. Mark on the field data sheet and record in the logbook:
  - Site designation and location
  - Sampler ID number
  - Filter ID number
  - Sample date
  - Initial flow rates and rotameter readings; initial temperature and barometric pressure, if required
  - Unusual conditions that may affect the results (e.g., subjective evaluation of pollution that day, construction activity, weather conditions)
  - Signature.

#### 3.5.2 Operator Who Removes the Samples

- 1. Mark on the field data sheet and record in the logbook:
  - Elapsed time of the sample run
  - Final flow rates and rotameter readings; final temperature and barometric pressure, if required
  - Existing conditions that may affect the results
  - Explanations for voided or questionable samples
  - Signature.

# 4.0 Filter Preparation and Analysis

The accuracy of a  $PM_{10}$  sampling program depends on several factors. A primary consideration is the analyst's attention to detail and balance technique. This section offers guidelines to enhance the accuracy of laboratory operation and hence the mass concentration determinations of  $PM_{10}$ .

Balance accuracy, precision, calibration requirements, and recommended filter media are outlined in Section 1. Laboratory activities are summarized in Table 4-1.

Activity	Method and frequency	Requirements	Action if require- ments are not met
Handling	Observe handling procedure.	Use nonserrated forceps and nylon gloves. Place filters in protective petri dishes and number sequentially.	Improve technique
Integrity check	Visually check each filter.	No pinholes, tears, etc.	Discard filter
Identification	Visually check each filter number assigned.	Fileter ID must be legible and recorded on the laboratory data/coding form and on the petri dish.	Identify properly
Equilibration (tare and gross desiccation)	Observe and record equilibration room or chamber conditions. Observe the minimum equilibrium period for each sample.	Equilibration in controlled environment for >24 h; RH between 20 and 45% with <±5% variation and tempera- ture between 15 and 30 °C with <±3% variation.	Repeat equilibration
Tare weighing procedure	Observe weighing procedure; perform all internal QC procedures.	Indicated filter weight deter- mined to nearest $\pm 1 \mu g$ . Begin weighing within 30 s after removing from equilibration chamber.	Reweigh after re- equilibration
Postsampling inspection, documentation, and verification	Visually check all samples and documentation.	No pin holes, tears, etc.; complete documentation; no evidence of malfunction or sample loss.	Void the affected samples; report to supervisor
Gross weighing procedure	Observe filter weighing procedures; perform all internal QC procedures.	Indicated weight obtained to nearest $\pm 1 \ \mu$ g; begin weighing within 30 s after removing from equilibration chamber.	Reweigh after re- equilibration

# TABLE 4-1. FILTER PREPARATION AND ANALYSIS CHECKS

# 4.1 Filter Handling

It is recommended that enough filters to last at least for a l-month sampling period be numbered and weighed at one time. During weighing, analysts must wear nylon gloves and handle the filters carefully with nonserrated forceps. This reduces the potential effect from body moisture or oils contacting the filters and subsequently affecting measured weights, and it also restricts static electricity interference. Filters should be packed so that each is encased in a filter cassette and a petri dish for convenience in filter weighing, transportation, and storage (see Figure 1.3). A label should be attached to the dish that identifies the filter number. This label will also be used at the time of sampling to identify sample date, size fraction (coarse/fine), and sampling site. To improve filter inventory control, care should be taken to stack the filters in the box in numerical order so that the operator will use the proper filter first.

If samples are to be mailed, the field operator should be supplied with reinforced envelopes or some other method (in addition to the petri dish) of protecting the exposed filters during their delivery to the analytical laboratory.

# 4.2 Filter Integrity Check

All filters must be visually inspected for defects before the initial weighing. A filter must be rejected if any defects are found. Batches of filters containing a high number of defects should be returned to the supplier.

Specific defects to look for are:

- Pinhole—A small hole appearing (1) as a distinct and obvious bright point of light when examined over a light table or screen, or (2) as a dark spot when viewed over a black surface.
- Separation of ring—Any separation or lack of seal between the filter and the filter border reinforcing ring.
- Chaff or flashing—Any extra attached residual material on the reinforcing, polyolefin ring, or heat seal area that would prevent an airtight seal when the ring is placed under compression.
- Loose material—Any extra loose material or dirt particles on the filter that require removal by brushing prior to weighing.
- 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.

## 4.3 Filter Equilibration

Filters must be equilibrated in a conditioning environment for at least 24 h before being weighed. Relative humidity (RH) should be held constant at a mean value between 20 and 45 percent, with a variability of not more than  $\pm 5$  percent. Temperature should be held constant with a mean value between 15 and 30 °C, with a variability of not more than  $\pm 3$  °C. An air conditioned room may be used for equilibration if it can be maintained at this RH and in this temperature range while the filters are equilibrating. RH and temperature must be checked and recorded on equilibration days (either manually or by hygrothermograph) to ensure compliance with these guidelines. Equilibration chamber malfunctions, discrepancies, and maintenance activities also should be recorded in the equilibration chamber or laboratory logbook.

Filters should be conditioned in their protective petri dishes with the lids (on which the filter ID is recorded) removed and placed beneath the bottom half of the petri dish. Placing the lid beneath the bottom half will make certain that no mixup occurs.

# 4.4 Initial Weighing Procedures (Tare Weight)

This section presents procedures specific for a common commercially available analytical balance. Calibration, QC checks (and acceptable tolerances) and operational procedures may have to be adapted to other analytical balance models.

Filters must be weighed on a microbalance with a minimum resolution of 0.001 mg and a precision of  $\pm 0.001$  mg (1 µg). Each balance used in the weighing procedures must be identified by a balance number. The procedures follow.

**Note**: Make sure that the balance has been calibrated (at least annually) and maintained according to manufacturer's recommendations. If out of calibration, have the balance calibrated according to manufacturer's directions.

- 1. Turn on the balance and allow it to warm up for at least 15 min. If it is used daily, leave it on at all times.
- 2. Zero the balance according to manufacturer's directions.
- 3. Have the QC supervisor perform the "standard" filter QC check (Section 4.5.2) to increase the validity of subsequent tare weight values.
- 4. If filters must be weighed outside the conditioning chamber, use caution to avoid interference with ambient hydroscopic particles and begin the weighing procedure within 30 s. Weigh the filter according to manufacturer's directions, making sure a stable reading is obtained.
- 5. Place the tared filter, with the reinforcing ring side up, in a comparably sized petri dish.
- 6. It is recommended that each balance be assigned a block of filter numbers to be used sequentially. Assign a filter ID number and take care to avoid duplication or missed numbers.

7. Record the assigned filter number on the petri dish, leaving sufficient room for one more letter designating size fraction (F for fine or C for coarse) to be written following the number.

**Suggestion**: The operator may decide to include three additional digits on the petri dish label to represent the tare weight of the filter (e.g., 101, 99, 105).

- 8. Record the balance number, the assigned filter number, and the tare weight on the laboratory data form (Figure 4.1). Number each form sequentially in the upper right corner.
- 9. Perform regular QC checks as detailed in Section 4.5.
- 10. Install the filter into a cassette and return the filter/cassette assembly to its individual petri dish.

# 4.5 Internal QC

#### 4.5.1 Analyst QC

After every tenth weighing, the analyst should recheck the zero and calibration of the balance and record these check values on the Laboratory Internal Quality Control Log (Figure 4.2). (The zero and 10-mg weight checks are internal standards of the analytical balance.) Zero QC checks within 4  $\mu$ g of true zero and calibration QC checks within 2  $\mu$ g of 10 mg are acceptable. Larger discrepancies should be corrected immediately. When QC checks are unacceptable, the previous 10 filters must be reweighed. Any filter weight outside of the normal range of 80 to 110 mg must be investigated immediately.

**Note**: An electrostatic charge will prevent a microbalance from operating properly. Static charge is the accumulation of electrical charges on the surface of a nonconductive material. Common symptoms of this problem include noisy readout, drift, and sudden readout shifts. To reduce static charge within the balance, it may be necessary to place a radioactive ionizing unit (i.e., Polonium 210) in the weighing chamber. It may also be necessary to pass the filters over an ionizing unit before they are weighed. For more information about static and how to minimize its effects, see the Technical Note, "Static Control for Balances," prepared by Cahn Instruments, Inc.

#### 4.5.2 Supervisory QC Procedures

- 1. Keep a bound QC notebook. These notebooks must contain all QC data, including the balance calibration and maintenance information, internal routine QC checks, and independent audits. It is recommended that control charts be maintained on each balance and included in this notebook. These charts may indicate any excess drift that could flag an instrument malfunction.
- 2. For cross-checking, reference all QC data on the Quality Control Log Form to the laboratory QC notebook.
- 3. At the beginning of each weighing day, after the analyst has completed the zeroing and calibration checks of the balances, tare weigh one arbitrarily selected filter from a set of "standard" filters (10 percent of the total number of filters to be weighed). Because these

			Analyst			
Balance Number			QC Supe	ervisor		
Initial Filter Weighing	Date			RH	Ter	np
Final Filter Weighing	Date			RH	Ter	np
Sampler Number	Filter Number <sup>a</sup>	Initial (m	Mass ig)	Fin	al Mass (mg)	Net Mass Filter Loading (mg)
						>

## Figure 4.1. Example laboratory data form.

lter Lot Number	W9706	Analyst Jennifer Arm	nstrong	
alance Number	A44603	QC Supervisor Bob Vanderpool		
Analysis Date	Zero (Tare) Check Weight (mg)	Zero Check Weight (mg)	Cal Check Weight (mg)	
6/10/97	0.000	0.001	10.001	
6/12/97	0.000	0.002	10.000	
			/	
Filter Number	Original Weight (mg)	Reanalysis Weight (mg)	±20 μg (Yes/No)	
6428	91.628	91.630	Yes	
6429	98.290	98.285	Yes	
6427	95.617	95.730	No	
6427 (repeat)	95.617	95.620	Yes	

Figure 4.2.	Example	laboratory	internal	QC	log.
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filters represent a repetitive QC check, do not use them for subsequent sampling. These weights must be repeatable for each balance to within  $20 \ \mu g$  of the original value. If not, the balance performance is unacceptable; troubleshoot and reweigh the filters as necessary. If more than one balance is used, take care that the filter is weighed on the same balance that determined the original tare value. Unless this procedure is adhered to, many samples may have to be invalidated.

- Reweigh five to seven exposed and unexposed filters per balance each day of operation. Weights should be within ±20 μg of original values; if not, troubleshoot and reweigh. Because of the loss of volatile components, no limits are set for exposed filters. Record all data on the Quality Control Log Form and in the QC notebook.
- 5. Certify acceptability of filter weights and data completeness daily on the laboratory data/coding forms and initial. When bound, these serve as a laboratory notebook. Sign each completed form.

# 4.6 Postsampling Documentation and Inspection

Upon receipt of the sample from the field, the analyst should follow this procedure:

- 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 and 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.
- 2. If the exposed filter was packaged for shipment, remove the filter from its protective petri dish and examine the petri dish. If sample material has been dislodged from a filter, recover as much as possible by brushing it from the petri dish onto the deposit on the filter with a soft camel's hair brush.
- 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.
- 4. Remove the filter from both the petri dish and the filter cassette. The filters must be handled with clean, nonserrated forceps; they must not be touched by the hands. Inspect the filters for any damage that may have occurred during sampling. Reject the filter for mass concentration determination or any additional analysis if defects are found.
- 5. Return filters with no defects to their original petri dish and forward to the laboratory. File the data sheets for subsequent mass concentration calculations.
- 6. Return defective filters with the type of defect (or combination of defects) to their original petri dish, labeled by defect type(s), and submit to laboratory supervisor for final approval of filter validity.

# 4.7 Final Weighing Procedure (Gross Weight)

- 1. Group filters according to their recorded balance numbers. (Filters should be separated initially by balance ID number; this will lower the incidence of balance error that would occur if different balances were used for tare and gross weights.) Reweigh each filter on the same balance on which its tare weight was obtained.
- 2. In an environmentally controlled area, open the petri dish, making certain that the lid (with the filter ID inscribed) is placed beneath the bottom and that no mixup occurs.
- 3. Cover the open petri dish with a clean laboratory paper towel and place it in the conditioning environment. Allow the filter to equilibrate according to procedures outlined in Section 4.3.
- 4. Repeat Steps 1 through 5 of the dichotomous filter tare weighing procedure (Section 4.4).
- 5. Perform the internal QC checks described in Section 4.5 to ensure validity of reweighing.
- 6. Record the indicated gross weight on the laboratory data/coding form.
- 7. If the dichotomous filter is not to receive additional analysis, place it back into the corresponding petri dish. Deliver weighed filters to the sample custodian for archiving.
- 8. If the filter is to receive further analysis, return it to the petri dish and note on the petri dish what additional analyses are required. Place an asterisk after the gross weight column on the laboratory data/coding form to indicate that the filter requires additional analysis. Carefully place each filter thus packaged in a box, and deliver to the sample custodian who will forward it to the laboratory responsible for the additional analysis.

# 4.8 Calculation of Net Mass Filter Loading

The gross weight minus the tare weight of a dichotomous filter is the net mass of the particulate for that filter. Each calculation of this process must be independently validated. Refer to Section 5 for information regarding the calculation of  $PM_{10}$  mass concentration.

# 5.0 Calculations, Validations, and Reporting PM<sub>10</sub> Data

Measurements of  $PM_{10}$  mass concentration in the atmosphere that are used to determine attainment of the NAAQS for particulate matter must be expressed in units of micrograms per actual cubic meter ( $\mu g/m^3$ ) of air. This section presents the calculations required to compute and report ambient  $PM_{10}$  concentrations. A summary of all calculation formulas and associated symbols is given in Table 5-1.

Particle size discrimination by inertial separation requires that specific air velocities be maintained in the sampler's air inlet system. These design velocities are obtained when a specified "design flow rate" is maintained. The design flow rate is specified as an actual flow rate  $(TQ_a \text{ and } CQ_a)$  measured at existing conditions of temperature  $(T_a)$  and pressure  $(P_a)$ .

The sampler's operational flow rate (i.e., the actual flow rate when the sampler is operating normally to collect a  $PM_{10}$  sample) should, of course, be very close to the design flow rate. All  $PM_{10}$  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. For determining the average sampler flow rate over a sample period, use of average temperature ( $T_{av}$ ) and average barometric pressure ( $P_{av}$ ) over the sample period is recommended. 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.

 $T_{av}$  and  $P_{av}$  readings may be recorded onsite or estimated from data obtained from a nearby U.S. National Weather Service Forecast Office or 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 that the actual temperature and barometric pressure at the site can be reasonably represented by such averages. It is therefore recommended that seasonal average temperature and pressure values ( $T_s$  and  $P_s$ ) for the site 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.

The calculations presented in this section assume that the sampler has been calibrated in actual flow rate units ( $TQ_a$  and  $CQ_a$ ) 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  $P_{av}$  in Equation (4).

Calculation	Formula	Equation	Sectior
Conversion of flow rates from standard to actual conditions	$Q_a = Q_{std} (T_a/P_a) (P_{std}/T_{std})$	1	2.2.4
Total and coarse rotameter actual corrections	$AC = I \left( T_a / P_a \right)^{1/2}$	2	2.2.4
Rotameter calibration, actual correction vs. transfer standard flow, actual conditions	AC = m[Q <sub>a</sub> (transfer standard)] + b	3	2.2.4
Slope, intercept, and linearity of total and coarse flow rate calibration	$\overline{TA_a}$ or $\overline{CQ_a}$ = 1/m [ $\overline{I}(T_{av}/P_{av})^{1/2}$ - b	4	2.2.4
Total rotameter set point response	TSP = {[m(16.7) + b] $(P_a/T_a)^{1/2}$ }	5	2.2.4
Coarse rotameter set point response	$CSP = \{[m(1.67) + b] (P_a/T_a)^{1/2}\}$	6	2.2.4
Total and coarse average rotameter readings	$\overline{I}$ = (TSP or CSP + IF)/2	7	3.3.2
QC percentage difference	% Difference = [100] $\frac{TQ_a \text{ or } CQ_a - \text{ Orifice flow rate}}{\text{Orifice flow rate}}$	8	3.4.2
Total volume of air sampled	$V = (\overline{TQ_a})t$	9	5.1.2
Total mass concentration	$PM_{10}$ total mass concentration = $\frac{(Mf + Mc)(10^6)}{(V)}$	10	5.1.2
Concentration of fine particle fraction	$[F] = \frac{Mf(10^6)}{(FQ_a)t}$	11	5.4
Concentration of coarse particulate fraction	$[C] = \frac{Mc(10^{6}) - [F](\overline{CQ_{a}})t}{(\overline{TQ_{a}})t}$	12	5.4
Percentage difference between sampler-indicated and audit-measured flow rates	% Difference = [100] $rac{Q_{a}(sampler) - Q_{a}(audit)}{Q_{a}(audit)}$	13	7.1.4

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#### 5.1 Calculations

#### 5.1.1 Flow Rate Calculations

The total and coarse flow rates are calculated by first averaging the sampler's initial rotameter set points (TSP or CSP) and final indicated rotameter responses (IF).

$$\bar{I} = (TSP \text{ or } CSP + IF)/2$$
(7)

where

 $\overline{I}$  = average total or coarse rotameter response, arbitrary units

TSP, CSP = total or coarse rotameter set points, arbitrary units

IF = indicated final total or coarse rotameter response, arbitrary units.

These values are then applied to the sampler's total or coarse calibration relationship.

$$\overline{\mathrm{TQ}}_{a}$$
 or  $\overline{\mathrm{CQ}}_{a} = 1/m[\overline{\mathrm{I}}(\mathrm{T}_{av}/\mathrm{P}_{av})^{1/2} - \mathrm{b}]$  (4)

where

#### 5.1.2 PM<sub>10</sub> Concentrations Calculation

The reporting of total  $PM_{10}$  mass concentration data requires the calculation of the total volume of air sampled (Equation [9]) and the computation for total mass concentration (Equation [10]).

$$V = (\overline{TQ_a}) t$$
(9)

where

V = total sample volume in standard volume units, m<sup>3</sup>

 $\overline{TQ_a}$  = total flow rate corrected to standard conditions, m<sup>3</sup>/min

t = elapsed total sampling time, min.

$$PM_{10} = \frac{(Mf + Mc)(10^6)}{(V)}$$
(10)

where

 $PM_{10} = mass \text{ concentration of } PM_{10}, \mu g/m^3$ 

- Mf = net mass of particulate of the fine filter, mg
- Mc = net mass of particulate of the coarse filter, mg
- $10^6$  = conversion factor for mg to µg and L to m<sup>3</sup>
  - V = total sample volume, L.

# 5.2 Calculation Validation

Data necessary to compute the mass concentration of  $PM_{10}$  originate from two main sources—field operations and laboratory operations—and must be validated. The validation procedure ensures that all reported data are accurate relative to the overall scope of the QA program. When the final mass concentration of  $PM_{10}$  in a sample has been computed, the validation procedure will not only check these computations, but will also aid in flagging questionable mass concentrations (i.e., extremely high or low values). Should a mass concentration approach the primary or secondary ambient air quality standard, this validation procedure will provide checks for all preliminary field and laboratory operations. The steps of the calculation validation procedure are as follows:

- 1. Gather the following data for each sample:
  - Total sampling time and the average total flow rate corrected to standard conditions, minutes, L/min.
  - Tare, gross, and net weights of both coarse and fine filters, mg.
- 2. Compute the total mass concentration of  $PM_{10}$  for seven samples per 100 (minimum of four per lot) as specified in Section 5.1.1 or 5.1.2. These suggested starting frequencies may be altered, based on experience and data quality. Decrease the frequency if past experience indicates that data are of good quality, or increase it if data are of poor quality. It is more important to be sure that the validation check is representative of the various conditions that may influence data quality than to adhere to a fixed frequency. If calculation errors are found, all values in that sample lot should be recalculated.
- 3. Scan all total mass concentration values, note those that appear excessively high or low, and investigate. Repeat Step 2 for these samples. Compare validated  $PM_{10}$  concentration to the originally reported value. Correct any errors that are found, initial them, and indicate the date of correction.
- 4. If all mass concentration computations appear correct and questionably high or low value(s) still exist, review all raw data (i.e., sample time, average actual total flow rate and its subsequent correction to standard conditions, and total net particle mass for coarse and fine filters) for completeness and correctness.

# 5.3 Data Reporting

The primary standards for particulate matter in the ambient air are based on the measured mass concentration of  $PM_{10}$ . Information on reporting and interpretation of  $PM_{10}$  data with respect to the attainment of these standards is covered in 40 CFR Part 50, Appendix K.

# 5.4 Additional Calculations

This section outlines the procedures and computations necessary to calculate the mass concentration for the fine and coarse particle fractions of a dichotomous sample. The following calculations are not required to determine attainment of the primary or secondary standards; rather, they are supplemental and may not be necessary. The dichotomous sampler is designed to fractionate a total  $PM_{10}$  sample into two discrete size ranges (fine particles [less than 2.5 µm] and coarse particles [less than 10 µm, but greater than 2.5 µm]). **Note**: A correction is required for the relatively small mass of fine particles collected on the coarse filter, as illustrated in Equation (12).

First determine the concentration of the fine particle fraction as calculated by Equation (11).

$$[F] = \frac{Mf(10^6)}{(\overline{FQ_a})t}$$
(11)

where

- [F] = mass concentration of fine particles,  $\mu g/m^3$
- Mf = net mass of particulate on fine filter, obtained gravimetrically, mg
- $10^6$  = conversion factor for mg to µg and L to m<sup>3</sup>
- $\overline{FQ_a}$  = average fine flow rate corrected to standard conditions and calculated as

 $\overline{TQ_a} - \overline{CQ_a}, L/min$ 

t = elapsed total sampling time, min.

The calculation to determine the concentration of the coarse particulate fraction is presented in Equation (12).

$$[C] = \frac{Mc (10^6) - [F] (\overline{CQ_a}) t}{(\overline{TQ_a}) t}$$
(12)

where

[C] = mass concentration of coarse particles,  $\mu g/m^3$ 

Mc = net mass of particulate on coarse filter, obtained gravimetrically, mg

- $10^6$  = conversion factor for mg to µg and L to m<sup>3</sup>
- [F] = mass concentration of fine particles as determined by Equation (11),  $\mu g/m^3$

- $\overline{CQ_a}$  = average coarse flow rate corrected to standard conditions, L/min
- $\overline{TQ_a}^a$  = average total flow rate corrected to standard conditions, L/min
  - t = elapsed total sampling time, min.

# 6.0 Maintenance

Preventive maintenance is defined as a program of positive actions aimed to prevent failure of monitoring and analytical systems. The overall objective of a routine preventive maintenance program is to increase measurement system reliability and to provide for more complete data acquisition.

This section outlines general maintenance procedures for a specific commercially available dichotomous sampler. For more complete information on a particular sampler, or on laboratory equipment maintenance, refer to the manufacturer's instruction manual for the individual instrument.

Records should be maintained for the maintenance schedule of each dichotomous sampler. Files should reflect the history of maintenance, including all replacement parts, suppliers, cost expenditures, and an inventory of on-hand spare equipment for each sampler.

# 6.1 Maintenance Procedures

## 6.1.1 Recommended Supplies for Maintenance Procedures

An alcohol-based general-purpose cleaner, cotton swabs, a small soft-bristle brush, paper towels, distilled water, and miscellaneous hand tools are required maintenance supplies for dichotomous samplers. A compressed air source is recommended, but not required.

## 6.1.2 Sampling Module

The sampling module of the dichotomous sampler consists of the sampler inlet and the virtual impaction assembly. Figure 6.1 shows a disassembled sampling inlet, and Figure 6.2 illustrates the virtual impactor assembly. All parts are sealed with O rings.

## CAUTION: SOME PM<sub>10</sub> DICHOTOMOUS SAMPLER INLETS SHOULD NOT BE DIS-MANTLED. CHECK WITH MANUFAC-TURER'S SPECIFICATIONS BEFORE PROCEEDING.

To dismantle the sampler inlet:

- Mark each assembly point of the sampler inlet with pen or pencil to provide "match marks" during reassembly.
- Disassemble the unit in accordance with manufacturer's instructions, taking care to retain all O rings and miscellaneous parts.



Figure 6.1. Dichotomous sampler inlet.

**Note**: If the assembly screws appear frozen, the application of penetrating oil or commercial lubricant will make removal easier.

- Clean all interior surfaces with the general-purpose cleaner or compressed air source, paying particular attention to small openings and crevices. Cotton swabs and/or a small brush would be most helpful in these areas. Completely dry all components.
- Reassemble the unit in accordance with the previously scribed match marks. Take particular care to ensure that all O ring seals are properly sealed and that all screws are uniformly tightened.

The O rings in the aerosol inlet should be removed periodically and conditioned with vacuum grease. This will inhibit breakdown and fraying of the O ring caused by friction on the inlet tube. The bug screen protecting the aerosol sampler inlet should be cleaned periodically during the summer months. The bug screen is exposed for cleaning by pulling the sampler inlet off the receiver tube assembly. An O ring in the sampler inlet acts as the seal. Many samplers are equipped with an inlet that also has a primary water trap on the exterior of the unit. If this trap is glass, care should be taken not to crack or break it, as the sampler will not maintain adequate vacuum during operation. The glass trap may either be replaced with a plastic jar or wrapped with insulating tape to minimize the danger of accidental breakage.

**Virtual Impactor Assembly**—Internal particulate deposits accumulate primarily on the outer and inner surfaces of the tip





(closest to sampler inlet) of the inlet tube. Thus, the inlet tube should be inspected periodically for such particulate deposits and cleaned as required. An inlet tube cleaning schedule of every 3 to 4 months is typical; the remaining inner surfaces should be cleaned every 6 to 12 months. Use alcohol or water and a soft-bristle brush for cleaning.

Examine sample module vacuum tubing periodically for crimps, cracks, or breaks and replace as necessary. Examine connecting fittings for cross-threading, and replace fittings if necessary.

#### 6.1.3 Control Module

## CAUTION: UNPLUG THE POWER CORD FROM ITS RECEPTACLE BEFORE REMOVING OR OPENING THE FRONT PANEL OF THE DICHOTOMOUS CONTROL MODULE.

#### **Control Module Cleaning Procedures**—

- 1. Remove or open the front panel and blow out loose dust and dirt if compressed air is available. Wipe down all surfaces with the general-purpose cleaner and towels.
- 2. Make note of any obvious problems in the unit and take action to correct them before completion of cleaning. Refer to the manufacturer's instruction manual.
- 3. Check rotameters for cleanliness. If they are dirty and/or contain water, they must be removed and cleaned. (If water is found, the interior of the vacuum pump may be damaged. It should be opened for inspection and possible repair.) To clean the rotameters, take the following steps:
  - Remove the tubing from the total rotameter output port and any other connecting tubing that may prove too inflexible to allow removal of the rotameters.
  - Remove the screws securing the rotameter assembly to the front panel.
  - Slip the assembly back from the front panel enough to gain access to the Allen screws in the top of each rotameter and remove the protective cover.
  - While holding the glass rotameter with one hand, loosen the Allen screws just enough to allow removal of each of the graduated glass tubes.
  - Clean the two rotameters with an alcohol-based cleaner and rinse thoroughly in distilled water. For proper cleaning of the unit, the float and its retainers also should be removed. The retainers are easily removed with the aid of a wire hook fashioned from a paper clip.
  - Allow the tubes to dry thoroughly and reassemble.
- 4. Remove and clean all filter jars. Check each for possible cracks and replace if necessary. Should a filter jar become cracked or loosened, the dichotomous sampler will not maintain an adequate vacuum during system leak tests. Be certain that each filter jar is tightened and sealed properly. Clean or replace any dirty filter elements. These elements may become dirty in routine operation or if the sampler is inadvertently energized without sample filters installed.
- 5. Clean the cooling fan's blades and housing with compressed air or a small brush. Check the housing for any dirt that could cause the fan to lock up.
- 6. Clean exterior surfaces of the vacuum pump; be sure that all cooling vents are open. Take care that fluids do not run inside the pump. Check all mounting brackets to ensure that they are tight and in good condition.

**Vacuum Pump**—It is recommended that the diaphragm and the flapper valves of the vacuum pump be replaced routinely (at l-year intervals) or if a sudden reduction in sampler vacuum occurs and a leak check indicates there are no other obvious leaks in the system.

To replace the diaphragm, remove the finned head of the pump by removing the head screws. Remove the screws that hold down the diaphragm and replace the diaphragm. Wipe down the head and interior valves to remove any small particles of diaphragm rubber that might be present. To reassemble, reverse the procedure, making sure that the screw clearance cavity in the plate is lined up under the intake valve screw heads. All head screws must be tightened evenly. Diaphragms are often available through local suppliers or they may be purchased through the manufacturer.

When all cleaning and routine maintenance operations have been completed, close the control module, reassemble and connect the sample module, and recalibrate the instrument (if necessary). Refer to Section 2 for calibration procedures.

# 6.2 Refurbishment of Dichotomous Samplers

Dichotomous samplers that have been operated in the field for extended periods may require major repairs or complete refurbishment. In these cases, the manufacturer's instrument manual must be referred to before work is undertaken. A dichotomous sampler that has been subject to major repairs or refurbishment must be leak-checked and calibrated prior to sample collection.

# 7.0 Auditing Procedures

The operating agency must perform QA audits and process evaluations to determine the accuracy of the  $PM_{10}$  monitoring system and, hence, the data it produces. The primary goal of an auditing program is to identify system errors that may result in suspect or invalid data. The efficiency of the monitoring system (i.e., labor input vs. valid data output) is contingent upon effective QA activities. This true assessment of the accuracy and efficiency of the  $PM_{10}$  measurement system can only be achieved by conducting an audit under the following guidelines:

- Without special preparation or adjustment of the system to be audited.
- By an individual with a thorough knowledge of the instrument or process being evaluated, but not by the routine operator.
- With accurate, calibrated, NIST-traceable transfer standards that are completely independent of those used for routine calibration and QC flow checks.
- With complete documentation of audit information for submission to the operating agency. The audit information includes, but is not limited to, types of instruments and audit transfer standards, instrument model and serial numbers, transfer-standard traceability, calibration information, and collected audit data.

The audit procedures described in this section produce a quantitative estimate of a  $PM_{10}$  sampler's performance: the audit flow rate percentage difference. The audit flow rate percentage difference determines the accuracy of the sampler's indicated flow rate by comparing it with a flow rate from the audit transfer standard.

An independent observer should be present for the audit, preferably the routine operator of the sampling equipment. This practice not only contributes to the integrity of the audit, but also allows the operator to offer any explanations and information that will help the auditor to determine the possible causes of discrepancies between audit-standard values and the sampling equipment values.

To determine whether differences in flow rate (between audit flow and sampler flow) are a result of sampling equipment malfunction or operator technique, an auditor may request permission to observe the routine field flow check procedure performed by the operator.

Audits and evaluations of the following individual portions of the total  $PM_{10}$  measurement system are detailed in this section:

- Flow rate performance audit
- System audit of data processing
- Analytical process system evaluation.

These audits and evaluations are summarized in Table 7-1. Refer to Part 1, Section 15, of this volume of the handbook for detailed procedures for systems audits.

Procedure	Frequency and/or method	Acceptance limits	Action if require- ments are not met
Flow rate audit	Once each quarter—PSD monitoring; once per year— SLAMS.	Percentage difference between the sampler- indicated flow rates and the audit-measured flow rate is within ±10%.	Recalibrate before resuming sampling; if difference exceeds $\pm 10\%$ , double-check calculations. If differ- ence still exceeds $\pm 10\%$ , invalidate since last calibration or valid flow check.
Analytical evaluation			
Filter weighing	Perform seven audits/100 filters, or four audits/≤50 filters, use microbalance, condition filters for at least 24 h before weighing.	Audit weight = original weight ±20 mg for unexposed filters; no limit for exposed.	Reweigh all filters in the lot.
Balance	Observe weighing technique. Review balance maintenance and calibration log.	Balance maintained and calibrated at least annually.	Reweigh all filters; calibrate balance.
Data processing audit	Independently repeat calculation of $PM_{10}$ concentration from data record for seven samples per 100 (minimum of four per lot).	Audit concentration agrees with original report concentra- tion within round-off error.	Recheck all calculations.
Systems audit	At beginning of a new monitoring system and periodically as appro- priate, observe procedures and use checklist.	Method described in this section and Part 1, Section 15, of this Handbook.	Initiate improved methods an/or training programs.

#### **TABLE 7-1. AUDITING REQUIREMENTS**

Proper implementation of an auditing program serves a twofold purpose: to ensure the integrity of the data and to assess the accuracy of the data. Additional information on assessing the accuracy of the data is given in 40 CFR, Part 58, Appendix A (Quality Assurance Requirements for State and Local Air Monitoring Stations [SLAMS]).

# 7.1 Flow Rate Performance Audit

The following section presents audit procedures specific to commercially available dichotomous samplers that operate at an actual total flow of 16.7 L/min and a coarse flow of 1.67 L/min. Audit techniques may vary between different models of samplers due to differences in required flow rates and the sampler's sampling configuration.

The dichotomous sampler flow rate audit method involves using two transfer standards. One is calibrated in the flow range of the total and fine flow rates and the second is calibrated within the range of the coarse flow rate. This enables the auditor to measure the critical flow rates directly without compounding transfer standard error through subtraction. Obviously, the optimum audit method would incorporate one transfer standard calibrated over the entire range of the sampler's accepted flow limits (1.5 L/min to 18.4 L/min). Accuracy over this flow range is difficult to achieve within acceptable limits. Consequently, it is recommended that audits be conducted using transfer standards within specific ranges to measure the sampler's indicated flow rates.

Since the accurate measurement of  $PM_{10}$  mass concentration is dependent upon flow rates under actual conditions, the auditor must also audit in terms of actual conditions. If the audit transfer standard's calibration data have been corrected to EPA reference conditions (298 K, 760 mm Hg or 101 kPa), a conversion must be calculated to adjust the standard L/min flow rate ( $Q_{std}$ ) to an actual L/min flow rate ( $Q_a$ ).

# 7.1.1 Audit Apparatus

Any type of flow rate transfer device acceptable for use in calibration of dichotomous samplers may be used as the audit flow rate reference standard; however, the audit standard **must** be a different device from the one used to calibrate the sampler. The audit standard must be calibrated

against a primary standard traceable to the NIST. Refer to Section 2, Tables 2-2 and 2-3, which reference flow rate transfer standard calibration procedures. Assemble the audit apparatus as indicated in Figures 7.1 through 7.3. In addition to that which is presented in the tables, a few miscellaneous supplies are required. These include a 9.53-mm (3/8-in.) Swagelok cap, 6.35-mm (1/4-in.) Swagelok cap, hand tools, and an adapter to connect the transfer standard outlet to the sampler inlet.

An audit data sheet similar to Figure 7.4 must be used to document audit information. This information includes, but is not limited to, sampler and audit transfer standard type, model and serial numbers, transfer standard traceability and calibration information, ambient temperature and pressure conditions, and collected audit data.



Figure 7.1. Audit assembly and dichotomous sampler set up to audit total flow (TQ<sub>a</sub>).

#### 7.1.2 Total Flow Rate Audit Procedures

- 1. Instruct the operator to install new filters in both the fine and coarse filter holders and energize the sampler. Filters used for flow rate audits should not be used for sampling.
- 2. Instruct the operator to adjust the rotameter flow control valves to set the total and coarse rotameters to their operational set points for routine sampling. These set points should correspond to the calculated set points (TSP, CSP) determined by the sampler's calibration relationship.
- 3. Allow the sampler to warm up for a minimum of 5 min while maintaining the proper total and coarse rotameter set points.
- 4. Complete the top half of the data sheet with the required information, including ambient temperature  $(T_a)$  and ambient barometric pressure  $(P_a)$ . Record both the TSP and CSP values and the corresponding flow rates.
- 5. Remove the sampler inlet and replace with the transfer standard adaptive device (see Figure 2.1).
- 6. Connect the adapter to the transfer standard outlet with flexible tubing, being careful not to crimp the tubing. If the



Figure 7.2. Audit assembly and dichotomous sampler set up to audit fine flow (FQ<sub>a</sub>).



Figure 7.3. Audit assembly and dichotomous sampler set up to audit coarse flow (CQ<sub>a</sub>).

	Dich	otomous Sar	mpler Audit D	Data Sheet		
Station Location	Cary, N	C Date	12/11/97	AIRS Number	372346789	
Station Address	101 N. Harris	son Ave.		Time 13	330	
Sampler Model	244E	S/N	619	EPA #	3872	.65
P <sub>a</sub> mm H	g T <sub>a</sub> _18_	°C or 291 K	Unusua	al Conditions n	one	
Audit Transfer S	Standards:					
Total and Fine:	Model Orifi	ce S/N 321	Coarse:	Model Or	rifice S/N	322
Transfer Standa	rds Calibration F	Relationships:				
Total and Fine:	m = 23.45	1 b = +1.1	0 r = 0	.999 Last C	al. Date	9/3/97
Coarse:	m = 2.393	b = +0.0	1 r = 0	.999 Last C	al. Date	9/3/97
Sampler Calibra	tion Relationshi	DS:				
Total: m =	0.379 b =	2.15 r =	0.999 Desigi	n TQ <sub>a</sub> 16.7	L/min, TSP	13.6
Coarse: m =	3.806 b =	0.04 r =	0.999 Desig	n CQ <sub>a</sub> 1.67	L/min, CSP	10.1
						·
			Observed	Q	Audit Diff	rencer
Flow Type	Transfer Std. Indication (TS)	Q <sub>a</sub> (audit) (L/min)	Observed Rotameter Reading	Q <sub>a</sub> (sampler) (L/min)	Audit Diff	erence <sup>^</sup>
<b>Flow Type</b> Total	Transfer Std. Indication (TS) 1.10	Q <sub>a</sub> (audit) (L/min) 17.48	Observed Rotameter Reading 13.6	Q <sub>a</sub> (sampler) (L/min) 16.7	Audit Diff	~erence^ % _4.5
Flow Type Total Fine	Transfer Std. Indication (TS) 1.10 0.95	<b>Q<sub>a</sub> (audit)</b> (L/min) 17.48 15.35	Observed Rotameter Reading 13.6 12.6	Q <sub>a</sub> (sampler) (L/min) 16.7 15.03	Audit Diff L/min -0.78 -0.32	<b>erence</b> * % 4.5 -2.1
Flow Type Total Fine Coarse	Transfer Std.           Indication (TS)           1.10           0.95           1.30	Q <sub>a</sub> (audit) (L/min) 17.48 15.35 1.71	Observed Rotameter Reading 13.6 12.6 10.1	Q <sub>a</sub> (sampler) (L/min) 16.7 15.03 1.67	Audit Diff L/min -0.78 -0.32 -0.04	-4.5 -2.1 -2.3
Flow Type Total Fine Coarse	Transfer Std. Indication (TS) 1.10 0.95 1.30 * Audit %	Q <sub>a</sub> (audit) (L/min) 17.48 15.35 1.71 Difference = [1	Observed Rotameter Reading 13.6 12.6 10.1 00] $\left[ \frac{(Q_a(sample))}{Q_a} \right]$	$Q_a$ (sampler) (L/min) 16.7 15.03 1.67 ler) - $Q_a$ (audit) a (audit)	Audit Diff	%       -4.5       -2.1       -2.3

#### Figure 7.4. Example dichotomous sampler audit data sheet.

transfer standard is electronic, it must equilibrate to operating conditions. A warmup time of at least 5 min is recommended.

- 7. Recheck rotameter settings; if different from designated set points, record new value and the corresponding flow rate as determined by the sampler's calibration relationship.
- 8. Record on the audit data sheet the transfer standard (TS) readings (volts,  $\Delta H_2O$ , timings, etc.).

#### 7.1.3 Fine Flow Rate Audit Procedures

- Turn the sampler off and disconnect the coarse-flow 6.53-mm (1/4-in.) line. Cap the coarse flow outlet port located beneath the dichotomous sampler filter holder with a 6.53-mm (1/4-in.) Swagelok cap. This opens the coarse line to the vacuum pump. To prevent particle entrapment within the system, it is recommended that a particle-free filter be attached to the line.
- 2. Turn the sampler on and check the rotameter set points. If variation has occurred since the total flow rate audit, record the total and coarse rotameter units and their corresponding flow rate values determined from the sampler's calibration. A small flow imbalance occurs when the coarse line is disconnected; this may cause rotameter fluctuations.
- 3. Record on the audit data sheet the TS readings (volts,  $\Delta H_2O$ , timings, etc.).

#### 7.1.4 Coarse Flow Rate Audit Procedures

- 1. Turn the sampler off and exchange the total and fine flow-rate transfer standard for the coarse flow transfer standard. If necessary, allow this transfer standard to equilibrate to ambient conditions (at least 5 min).
- Reconnect the coarse flow line and disconnect the fine flow 9.53-mm (3/8-in.) line. Cap the fine flow outlet port located beneath the dichotomous sampler filter holders with a 9.53-mm (3/8-in.) Swagelok cap. This opens the fine line to the vacuum pump. To prevent particle entrapment within the system, it is recommended that a particle-free filter be attached to the line.
- 3. Turn the sampler on and check rotameter set points. If variation has occurred since the total flow rate audit, record the total and coarse rotameter units and their corresponding flow rate values determined from the sampler's calibration. A small flow imbalance occurs when the fine line is disconnected; this may cause rotameter fluctuations.
- 4. Record on the audit data sheet the TS readings (volts,  $\Delta H_2O$ , timings, etc.).

#### 7.1.5 Audit Data Calculations

 Calculate and record the audit total, fine, and coarse flow rates by using the calibration curve accompanying the transfer standard. Record these values to the nearest 0.01 L/min (e.g., 1.67 L/min) on the audit data sheet. **Note**: It may be necessary to correct audit flow rates to actual conditions. If a soap film flowmeter has been used to determine the coarse flow rate, no water vapor corrections are necessary for this audit flow.

$$Q_a = Q_{std}(T_a/P_a) (P_{std}/T_{std})$$
(1)

where

 $Q_a$  = flow rate at actual conditions, L/min

- Q<sub>std</sub> = flow rate corrected to standard temperature and pressure (25 °C, 298 K; 760 mm Hg or 101 kPa), L/min
  - $T_a$  = ambient temperature, K (K = °C + 273)
- $P_a$  = ambient barometric pressure, mm Hg (or k $P_a$ )

 $P_{std}$ ,  $T_{std}$  = standard barometric pressure and temperature, respectively.

- 2. Instruct the operator to calculate (using the sampler's calibration relationship) the corresponding sampler flow rates and record.
- 3. Determine the percentage difference between the sampler-indicated flow rates and the audit-measured flow rates as:

% Difference = [100] 
$$\left[ \frac{Q_a(\text{sampler}) - Q_a(\text{audit})}{Q_a(\text{audit})} \right]$$
. (13)

- 4. Record percent difference. If the difference is less than or equal to  $\pm 10$  percent, the sampler calibration is acceptable. Differences exceeding  $\pm 10$  percent require sampler recalibration. Differences exceeding  $\pm 10$  percent may result in invalidation of all data subsequent to the last calibration or valid flow check. Before invalidating any data, double-check the sampler's calibration, the audit orifice transfer standard's certification, and all calculations.
- 5. Before leaving the site, a comparison between the flows determined should be made (i.e., fine + coarse = total). If the sum of the individual flows (fine and coarse) does not equal the total flow (within  $\pm 2$  percent), the audit data should be checked. If necessary, the audit should be repeated.

#### 7.1.5 Performance Audit Frequency

The frequency of audits of the flow rate depends on the use of the data (e.g., for PSD air monitoring or for SLAMS). For PSD monitoring, the flow rate of each sampler must be audited at least once each sampling quarter. For SLAMS, audit the flow rate of at least 25 percent of the samplers per monitoring network each quarter. Each sampler, therefore, is audited at least once per year. If there are fewer than four  $PM_{10}$  samplers within a reporting organization, reaudit one or more randomly selected samplers so that one sampler is audited each calendar quarter.

## 7.2 Systems Audit

A systems audit is an onsite inspection and review of the quality of the total measurement system (sample collection, sample analysis, data processing, etc.). This audit is normally conducted at the startup of a new monitoring system and as appropriate thereafter. Sections 7.2.1 and 7.2.2 present systems audit procedures for evaluating data processing and laboratory operations.

Part 1, Section 15, and Appendix 15 of this volume of the handbook provide detailed procedures and forms for systems audits and performance audits, respectively.

#### 7.2.1 Systems Audit of Data Processing

It is recommended that data processing be audited soon after the original calculations have been performed. This allows corrections to be made immediately and also allows for possible retrieval of additional explanatory data from field personnel when necessary. A minimum frequency of seven samples per 100 (minimum of four per lot) is recommended. The procedure is as follows:

- 1. Use the operational flow rates as reported on the sample data sheets.
- 2. Beginning with the raw data on the dichotomous sample data sheet and the filter net and tare weights, independently compute the concentration ( $\mu g/stdm^3$ ) and compare it with the corresponding concentration originally reported. If the mass concentration computed by the audit check ( $\mu g/stdm^3$ ) does not agree with the original value within round-off error, recheck all samples in the lot.
- 3. Record the audit values on a data sheet and report them, along with the original values, to the supervisor for review. The audit value is always given as the correct value based on the assumption that a discrepancy between the two values is always double-checked by the auditor.

#### 7.2.2 Analytical Process System Evaluation

A performance audit of the microbalances used to weigh dichotomous filters would require the use of ASTM Class 1 standard weights. Since microbalances are extremely delicate instruments and should not be operated by inexperienced personnel, it is recommended that the performance evaluation of the filter weighing process be done in the following manner:

- 1. Review the maintenance and calibration log for each balance. Routine balance maintenance and calibrations must be performed by the manufacturer's service representative at manufacturer-specified scheduled intervals. In no case should the interval between calibrations exceed 1 year.
- 2. Review QC data records for the filter-weighing process. Ensure that the following QC activities have been performed and documented:
  - Zero and calibration checks after every five filter weighings
  - Standard filter weighing every day of the balance operation
  - Duplicate filter weighing for every five to seven filters.

If QC checks were out of limits, note what action was taken.

- Select randomly and have the balance operator reweigh four equilibrated filters out of every group of 50 or less. For groups of 50 to 100, reweigh 7 from each group. It is of primary importance to be sure that the sample is representative of the various conditions that may influence data quality.
- Record the original values and the audit weights on the audit form. Calculate the weight difference for each filter as follows:

Difference = Original weight (mg) - Audit weight (mg).

For unexposed filters, the difference should be less than  $\pm 20 \ \mu g$  (0.020 mg). For exposed filters, the potential loss of volatile particles prohibits acceptance/rejection limits to be established. Forward the audit data to the laboratory supervisor for review.

# 8.0 Assessment of Monitoring Data for Precision and Accuracy

#### 8.1 Precision

One or more monitoring sites within the reporting organization are selected for duplicate collocated sampling as follows: for a network of 1 to 5 sites, 1 site is selected; for a network of 6 to 20 sites, 2 sites are selected; and for a network of more than 20 sites, 3 sites are selected. Where possible, additional collocated sampling is encouraged. Sites must be selected on the basis of having annual mean  $PM_{10}$  concentrations that are among the highest 25 percent of the annual mean  $PM_{10}$  concentrations for all the sites in the network. If such sites are impractical, however, alternate sites approved by the Regional Administrator may be selected.

Collocated  $PM_{10}$  samplers being used for assessment of precision should generally be of the same type. That is, they should have similar flow rates (e.g., high, medium, or low), similar inlet types (e.g., impaction or cyclonic), and similar flow controller types (e.g., mass flow controllers [MFCs] or rotameters). Where a  $PM_{10}$  network contains more than one type of sampler, each type should be represented by at least one collocated sampler pair, if possible.

The two collocated samplers must be within 4 m of each other, but at least 2 m apart to preclude air flow interference. Calibration, sampling, and analysis must be the same for both collocated samplers and all other samplers in the network. One of each pair of collocated samplers is designated as the primary sampler from which samples will be used to report air quality for the site; the other is designated as the duplicate sampler. Each duplicate sampler must be operated concurrently with its associated primary sampler at least once a week. The operation schedule should be selected so that the sampling days are distributed evenly over the year and over the 7 days of the week. The every-6th-day schedule used by many monitoring agencies is required. The measurements from both samplers at each collocated sampling site are reported. The percentage differences in measured concentrations ( $\mu$ g/m<sup>3</sup>) between the two collocated samplers are used to calculate precision as described in 40 CFR Part 58, Appendix A.

# 8.2 Accuracy

Each calendar quarter, audit the flow rate of at least 25 percent of the  $PM_{10}$  samplers such that each  $PM_{10}$  sampler is audited at least once per year. If there are fewer than four  $PM_{10}$  samplers within a reporting organization, randomly reaudit one or more samplers so that one sampler is audited each calendar quarter.

The accuracy of the dichotomous sampler method in the measurement of  $PM_{10}$  is assessed by auditing the performance of the sampler (at its specified flow rate) as described in Section 7.0. Both the audit flow rate and the corresponding sampler flow rate are reported. The percentage differences between these flow rates are used to calculate accuracy as described in 40 CFR Part 58, Appendix A.

# 9.0 Recommended Standards for Establishing Traceability

Two factors are essential for attainment of data of the desired quality: (1) the measurement process must be under statistical control at the time of the measurement, and (2) the combination of systematic errors and random variation (measurement errors) must yield a suitably small uncertainty. Evidence of good quality data requires the performance of QC checks, independent audits of the measurement process, careful documentation of data, and the use of equipment and instrumentation that can be traced to an appropriate primary standard.

The following standards are recommended for establishing traceability:

- 1. ASTM Class 1, 1.1, or 2 weights are recommended for the laboratory microbalance calibration. See Section 4.5 for details on balance calibration checks.
- 2. A positive-displacement primary standard or laminar flow element is recommended for calibrating the flow rate transfer standard that is used to calibrate the dichotomous sampler. See Section 2 for details on dichotomous sampler calibration.
- 3. A positive-displacement primary standard is recommended for calibrating the transfer standard used to audit the dichotomous flow rate calibration. See Subsection 7.1 for details on the flow rate performance audits.
- 4. The elapsed time meter should be checked semiannually against an accurate timepiece, and it must be accurate within 15 min/day.
- 5. Accuracy checks of associated monitoring equipment (e.g., thermometers, barometers, stopwatches) should be conducted at routine intervals and against standards of known accuracy and traceable to NIST.