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FINAL TIME CRITICAL REMOVAL ACTION AIR MONITORING PLAN FORMER VIEQUES  
NAVAL TRAINING RANGE VIEQUES ISLAND PUERTO RICO  
09/01/2005  
CH2M HILL

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*Final*

# **Time Critical Removal Action Air Monitoring Plan**

## **Former Vieques Naval Training Range Vieques, Puerto Rico**

**Contract Task Order 0047  
Contract No. N62470-02-D-3052**

Prepared for

**Department of the Navy  
Atlantic Division  
Naval Facilities Engineering Command**

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- A Table 1 of Ambient Monitoring Guidelines for Prevention of Significant Deterioration (EPA-450/4-87-007, May 1987)
- B E-BAM Instrument Manual
- C Sampling Standard Operating Procedures
- D Analytical Methods
- E Site Visit Log
- F Audit Data Sheet
- G Sample Data Report

# Acronyms

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°C	Degrees Celsius
°F	Degrees Fahrenheit
μCi	Microcuries
μg/ m <sup>3</sup>	Micrograms per Cubic Meter
<sup>14</sup> C	Carbon 14
ATSDR	Agency for Toxic Substances and Disease Registry
CFR	<i>Code of Federal Regulations</i>
COC	Chain-of-Custody
DOE	Department of Energy
DOI	Department of the Interior
DQO	Data Quality Objective
ECD	Electron Capture Detector
EPA	United States Environmental Protection Agency
GC	Gas Chromatograph
IM	Interim Measures
L/min	Liters per Minute
LCS	Laboratory Control Samples
LIA	Live Impact Area
m/s	Meters per Second
MDL	Method Detection Limit
MEC	Munitions and Explosives of Concern
mg/m <sup>3</sup>	Milligrams per Cubic Meter
mm Hg	Millimeters Mercury
MRA	Munitions Response Area
MRL	Minimal Risk Level
MRS	Munition Response Site
MS	Matrix Spike

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MSD	Matrix Spike Duplicate
NAAQS	National Ambient Air Quality Standards
NIST	National Institute of Standards and Technology
OT	Outside Temperature
PM <sub>10</sub>	Particulate Matter Less than 10 Microns in Aerodynamic Diameter
PSD	Prevention of Significant Deterioration
QA	Quality Assurance
QC	Quality Control
RBC	Risk-Based Concentration
RL	Reporting Limit
RPD	Relative Percent Difference
SIA	Surface Impact Area
SOP	Standard Operating Procedure
SRM	Standard Reference Materials
TCRA	Time Critical Removal Action
TEEL	Temporary Emergency Exposure Level
VNTR	Vieques Naval Training Range
XRF	X-ray Fluorescence

# 1.0 Background and Monitoring Objectives

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The object of the air monitoring program at the former Vieques Naval Training Range (VNTR) is to address the air quality impact of the time critical removal action (TCRA) and document that offsite concentrations of constituents emitted from open detonations of munitions associated with a TCRA do not exceed National Ambient Air Quality Standards (NAAQS), or pose a threat to human health or the environment. This document provides the details of how such monitoring will be conducted.

The TCRA/Interim Measures (IM) Work Plan, for the removal of surface munitions and explosives of concern (MEC) at the VNTR, was prepared as part of the ongoing Munitions Response Program to reduce risk to human health and the environment in an area where trespassing frequently occurs.

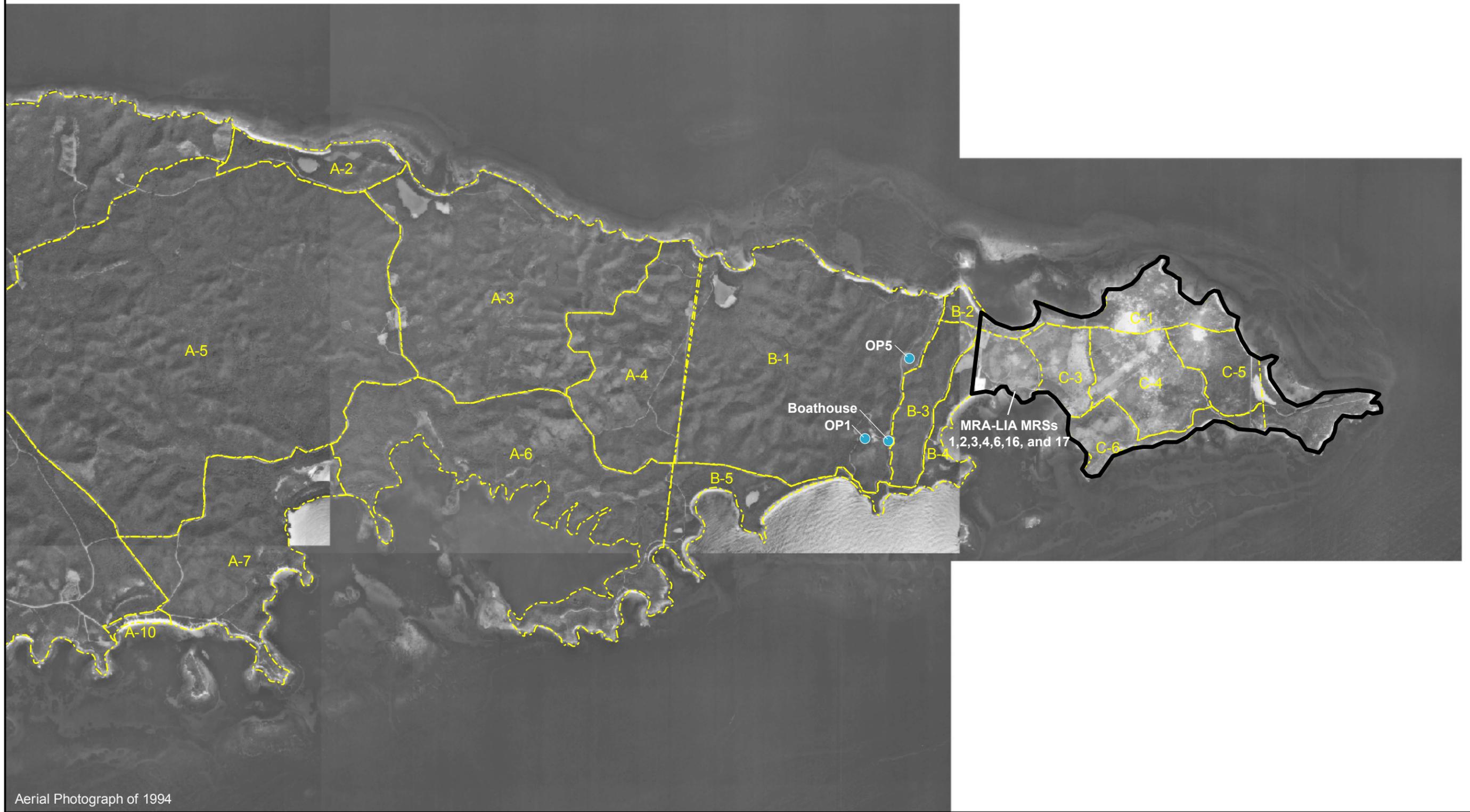
The air monitoring program will be performed adjacent to the Munitions Response Area (MRA) - Live Impact Area (LIA); an area of roughly 400 acres. The objective of the TCRA/IM is to reduce risk at munition response sites (MRSs) identified as posing an explosive hazard due to MEC present on the ground surface. The primary risk from explosive hazards is posed to unauthorized personnel who access the MRA-LIA. Access to the areas identified for MEC removal is restricted, but trespassing occurs in those areas. Restrictions to those areas are not anticipated to be lifted. TCRA/IM project objectives will be met by removing all MEC present on or exposed at the ground surface.

Air monitoring will include determination of continuous 24-hour concentrations of particulate matter less than 10 microns in aerodynamic equivalent diameter (PM<sub>10</sub>) using a network of Met One E-BAM portable beta attenuation mass monitors. Air samples will be collected for 8 hours prior and 16 hours after an open detonation. After E-BAM near real-time determination of PM<sub>10</sub> concentration, the filter tape will be analyzed for particulate metals and subsequently for explosive residues. The main data quality objective (DQO) of this project is to document the impact of air emissions from the TCRA on human health and the environment.

This document is written to explain the details of the Monitoring Plan and provide guidance for the Monitoring Plan Quality Assurance and Quality Control (QA/QC) procedures. As an additional reference for air quality monitoring criteria, Table 1 of Ambient Monitoring Guidelines for Prevention of Significant Deterioration (EPA-450/4-87-007, May 1987) has been included (see Appendix A).

This document also includes standard operating procedures (SOPs) to serve as guides for the operators responsible for the monitors and meteorological instruments. Specific procedures are presented to guide operators through routine activities, such as site visits, maintenance, calibrations, and audits. The object of this document is to ensure the collection of data of known and acceptable quality. This can be achieved through two measures: (1) control of procedures for data collection and (2) standardized assessment of the quality of captured data. This plan addresses the control of procedures by presenting SOPs and

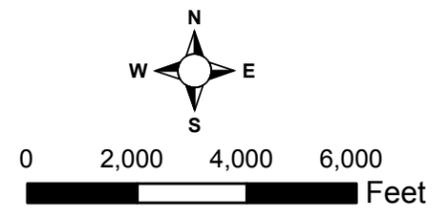
activity checklists. It also addresses the DQOs by specifying audit, calibration, laboratory, and data validation procedures.



Aerial Photograph of 1994

LEGEND

-  Interior Island MRS
-  A-5 Parcel Boundary and Designation
-  Air Monitoring Station



**FIGURE 1-1**  
AIR MONITORING STATION LOCATIONS  
FORMER VNTR, VIEQUES ISLAND, PUERTO RICO  
SEPTEMBER 2005

## 2.0 Source Environment Description

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### 2.1 Topography

Vieques is located in the Caribbean Sea approximately 7 miles southwest of the eastern tip of Puerto Rico, and 20 miles southwest of St. Thomas, U.S. Virgin Islands. Vieques is approximately 20 miles long and 4.5 miles wide, and has an area of approximately 33,088 acres. The topography is characterized by gentle to steep rolling hills and valleys throughout the island, with the eastern side exhibiting a more rugged terrain. The former VNTR is situated in the eastern half of the island. The TCRA munitions removal actions encompass approximately 440 acres of the MRA-LIA. The LIA consists of relatively flat, heavily vegetated terrain with various types of unexploded ordnance throughout the landscape.

### 2.2 Land Use

The LIA was used for air-to-ground ordnance, artillery, and individually served weapons training from the early 1960s to 2003. The United States and allied nations, particularly Great Britain, used the VNTR for military training. Since 2003, the VNTR has been designated as a wildlife refuge. The entire VNTR is required by Section 1049 of the National Defense Authorization Act for Fiscal Year 2002 to be managed by the Department of the Interior (DOI) as part of the National Wildlife Refuge Systems. Parts of the VNTR, including the LIA, will be managed as wilderness areas with no public access allowed. The Department of Energy (DOE) is developing a comprehensive land use plan for eastern Vieques that is to be completed by 2006.

### 2.3 Climate

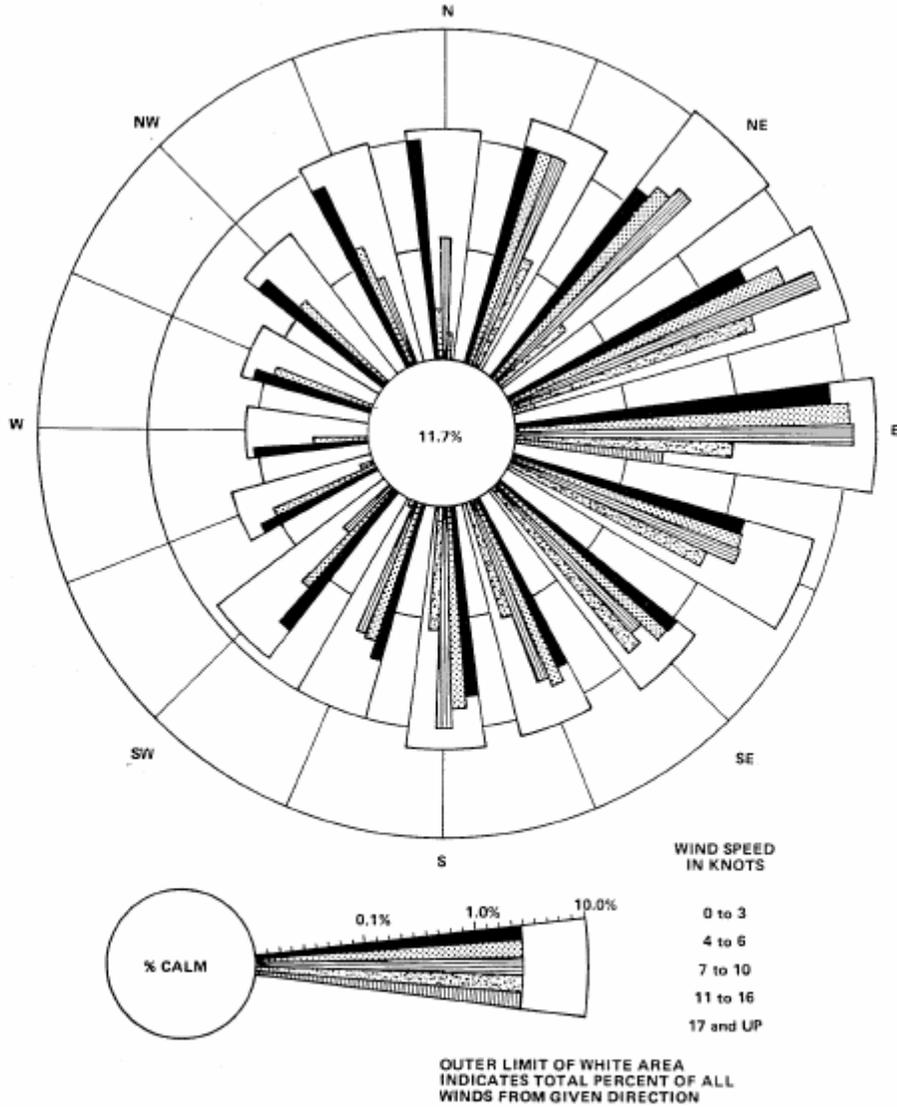
Vieques Island has a tropical-marine climate without frost. The temperature is nearly constant, with an annual average of approximately 79 degrees Fahrenheit (°F). The island is subject to tropical storms and hurricanes. As such, monitoring schedules must take into account the potential for unforeseen weather events and the relative fragility of E-BAM sampling units. Reasonable efforts will be made to remove or protect E-BAM units in the field before the onset of any tropical storms or hurricanes that would affect Vieques Island.

### 2.4 Meteorological Data

Figure 2-1 is a wind rose showing the prevailing wind speed and direction of Roosevelt Roads, located roughly 9 miles from Vieques. Continuous air monitoring from July through September of 1978 (Table 2-1) shows a close correlation between the wind directions observed at Roosevelt Roads and Vieques. The data shows that the predominant wind direction on Vieques from February through October is likely from the east. During November through January the prevailing wind direction is predominantly from the east-

northeast. During the course of this project, wind speed and direction data from the site will be obtained.

**FIGURE 2-1**  
Annual Wind Rose at Roosevelt Roads



**FIGURE 2-1**  
**ANNUAL WIND ROSE AT ROOSEVELT ROADS, PR**  
**(1/73 - 12/77)**

SOURCE: Seasonal and Annual Wind Distribution by Pasquill Stability Classes, STAR Program Station No. 11630 Roosevelt Roads, P. R. for Period 1/73-12/77 (80 obs/Day). National Climatic Center, Federal Building, Asheville, North Carolina, May 22, 1978

**TABLE 2-1**  
Wind Characteristics at Three Weather Stations within 20 Miles of Vieques and Local Oceanographic Wind Data

Month	Roosevelt Roads, P.R. <sup>a</sup> NWSED		Alexander Hamilton Field, St. Croix <sup>b</sup>		Vieques Island <sup>c</sup>	
	Mean Speed (Knots)	Prevailing Direction	Mean Speed (Knots)	Prevailing Direction	Mean Speed (Knots)	Prevailing Direction
January	8.3	ENE	11.0	ENE	12.3	ENE
February	8.1	E	10.6	ENE	11.6	E
March	8.5	E	10.9	ENE	11.2	E
April	8.2	E	10.7	ESE	10.7	E
May	7.7	E	10.4	ESE	11.2	E
June	7.8	E	11.3	ESE	13.4	E
July	8.6	E	11.3	ENE	14.5	E
August	8.0	E	10.5	ENE	12.3	E
September	6.2	E	10.0	ESE	10.1	E
October	5.3	E	9.6	ESE	10.1	E
November	5.9	E	9.6	ENE	8.9	ENE
December	7.2	ENE	9.7	ENE	12.3	ENE

**Source:**

<sup>a</sup>Summary of Meteorological Observations, Surface (SMOS), Roosevelt Roads, P.R. for Period 1948 – 1972, Job No. 72005, Naval Weather Service, Environmental Detachment, Federal Building, Asheville, North Carolina, March 1973. (Period of Record, 1948 – 1950, 1957 – 1972).

<sup>b</sup>Calvesbert, R.J., "The Climate of Puerto Rico and the U.S. Virgin Islands, June 1970," In Climates of the United States, Volume I, U.S. National Oceanic and Atmospheric Administration. U.S. Department of Commerce, 1974. (Period of Record – 6 Years).

<sup>c</sup>Turner, D.B., Workbook of Atmospheric Dispersion Estimates, Environmental Protection Agency Research, Triangular Park, North Carolina, 1970, Page 41. (Compiled and Extrapolated from Various Sources such as Ship Logs, Ship Weather Reporting Forms, Published Ship Observations, Automatic Observing Buoys, Teletype Reports and Punch Cards, Irregularly Over a 60 Year Period 1911 – 1970).

## 3.0 Health-Based Guidance Levels

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Health-based guidance levels in air for the explosive constituents are presented in Table 3-1. These represent concentrations based on annual averages, 8- and 24-hour average periods, and are intended to be protective of human health. The annual average concentrations were derived from the United States Environmental Protection Agency (EPA) Region 3 Risk-Based Concentrations (RBCs), with the exception of tin, which is based on the Agency for Toxic Substances and Disease Registry (ATSDR) Minimal Risk Levels (MRLs), and lead, which is based on the NAAQS. The short-term values are derived from the DOE Temporary Emergency Exposure Levels (TEEL) based on no observed effects associated with 1-hour exposure (TEEL-0). The 1-hour TEEL-0 values were adjusted to 8- and 24-hour averaging periods with adjustment factors used for air dispersion modeling, as presented in EPA, 1992, Appendix D. In addition, the values derived from the TEEL-0 were further adjusted using a ten-fold uncertainty factor to develop values that were protective of the general public (the TEEL values are intended for protection of workers). All assumptions and sources for these values are shown in Table 3-1. It should be noted that these values do not denote a “bright line” between safe and harmful levels of exposure. Measured concentrations over their respective averaging times that routinely exceed these values do not indicate a potentially significant risk to human health, but rather indicate that further evaluation of potential exposures associated with the removal action may be warranted.

**TABLE 3-1**  
Health-Based Guidance Levels in Air

Compound	Abbrev.	Health-Based Guidance Levels ( $\mu\text{g}/\text{m}^3$ in air)								TEEL-0 Values
		Annual Average (chronic exposure)		24-hour average		8-hour average		1-hour average (acute exposure)		( $\text{mg}/\text{m}^3$ )
		Value	Source	Value	Source	Value	Source	Value	Source	Value
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	80	EPA, 2005	900	see note	1575	see note	2250	see note	NA
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	10	EPA, 2005	50	see note	85	see note	125	see note	NA
Nitrobenzene	NB	2	EPA, 2005	200	TEEL-0	350	TEEL-0	500	TEEL-0	5
1,3-Dinitrobenzene	1,3-DNB	0.4	EPA, 2005	40	TEEL-0	70	TEEL-0	100	TEEL-0	1
1,3,5-Trinitrobenzene	1,3,5-TNB	110	EPA, 2005	400	TEEL-0	700	TEEL-0	1000	TEEL-0	10
2,4-Dinitrotoluene	2,4-DNT	11	EPA, 2005	8	TEEL-0	14	TEEL-0	20	TEEL-0	0.2
2,6-Dinitrotoluene	2,6-DNT	11	EPA, 2005	8	TEEL-0	14	TEEL-0	20	TEEL-0	0.2
2,4,6-Trinitrotoluene	2,4,6-TNT	2	EPA, 2005	60	TEEL-0	105	TEEL-0	150	TEEL-0	1.5
Cadmium	Cd	0.04	TEEL-0	0.2	TEEL-0	0.35	TEEL-0	0.5	TEEL-0	0.005
Copper	Cu	8	TEEL-0	40	TEEL-0	70	TEEL-0	100	TEEL-0	1
Chromium	Cr	0.4	EPA, 2005	4	TEEL-0	7	TEEL-0	10	TEEL-0	0.1
Lead	Pb	1.5	NAAQS	2	TEEL-0	3.5	TEEL-0	5	TEEL-0	0.05
Phosphorus	P	0.1	EPA, 2005	20	TEEL-0	7	TEEL-0	10	TEEL-0	0.1
Tin	Sn	110	ATSDR, 2005	120	TEEL-0	210	TEEL-0	300	TEEL-0	3
Iron	Fe	16	TEEL-0	80	TEEL-0	140	TEEL-0	200	TEEL-0	2
Nickel	Ni	8	TEEL-0	40	TEEL-0	70	TEEL-0	100	TEEL-0	1
Arsenic	As	0.08	TEEL-0	0.4	TEEL-0	0.7	TEEL-0	1	TEEL-0	0.01
Mercury	Hg	1.1	EPA, 2005	1.4	TEEL-0	2.45	TEEL-0	3.5	TEEL-0	0.035

**Notes:**Annual average values:

EPA, 2005. <http://www.epa.gov/iris/>. Values are inhalation RfCs, or extrapolated from oral RfDs, in cases where appropriate inhalation values are not available. Concentrations extrapolated from oral RfDs were calculated using standard default exposure factors

Lead - annual average value is based on the National Ambient Air Quality Criteria.

Phosphorus - annual average value is based on white phosphorus.

Tin - ATSDR, 2005 - ATSDR intermediate Minimal Risk Level (MRL), adjusted using a 10-fold uncertainty factor to obtain a chronic value. <http://www.atsdr.cdc.gov/mrls.html>

NAAQS - <http://epa.gov/air/criteria.html>

TEEL-0 - Temporary Emergency Exposure Limits based on no observable effect following one hour exposure <http://www.atlantl.com/DOE/teels/teel/teels.XLS>. For purposes of this plan, an additional 10-fold uncertainty factor was added to adjust values for protection of the general public (TEEL values are considered workplace exposure limits)

1-hour, 8-hour and 24-hour average values:

TEEL-0 - Temporary Emergency Exposure Limits based on no observable effect following one hour exposure <http://www.atlantl.com/DOE/teels/teel/teels.XLS>. For purposes of this plan, an additional 10-fold uncertainty factor was added to adjust values for protection of the general public (TEEL values are considered workplace exposure limits)

TEEL value not available for HMX. Value was extrapolated from annual average based on prevention of noncancer effects using air modeling adjustment factors (see below).

TEEL value not available for RDX. Appropriate noncancer criteria is not available for RDX, for use in calculating guidance level for short term exposure. Value is extrapolated from annual average based on noncancer effects level, using air modeling adjustment factors.

## Adjustment factors for averaging times (from one hour concentrations)

Averaging time (hour)	Adjustment factor
3	0.9
8	0.7
24	0.4
annual	0.08

Source: EPA, 1992. Workbook of Screening Techniques for Assessing Impacts of Toxic Air Pollutants (Revised). EPA-454/R-92-024

## 4.0 Monitoring Program Description

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The main DQO is to document the impact of air emissions from the TCRA on public human health and the environment. PM<sub>10</sub> monitoring will be conducted using a network of Met One Instruments E-BAM portable particulate monitors. The E-BAM contains a small <sup>14</sup>C (Carbon 14) source. The activity of the source is 60 μCi (microcuries), which is below the federal limit for these sources as defined in 10 *Code of Federal Regulations (CFR)*, Section 30.70–Schedule A.

Meteorological data will be limited to wind speed and direction measured at each of the three E-BAM monitoring sites. Wind speed and direction data will be uploaded by satellite to a secure Web site. In addition to PM<sub>10</sub> monitoring, particulate samples from the E-BAM monitors will be collected and analyzed for metals and explosives residue.

Actual wind speed and direction data will be used for modeling purposes if there is an exceedance of the health-based risk levels. The modeling results will be used to determine if there has been a potential impact on public human health.

### 4.1 Air Monitoring Program Schedule of Operations

This monitoring plan provides for continuous PM<sub>10</sub> particulate sampling on a specialized Teflon tape that is set to advance every 8 hours. A sampling time of 8 hours was chosen to provide adequate sensitivity and to correspond to the health-based guidance levels. Air samples will be collected 8 hours prior and 16 hours following an open detonation. The Teflon tape will be analyzed weekly for metals and explosive residues for the first month of the sampling program, and subsequent monthly analysis while TCRA operations are being conducted. Additional samples for metals and explosives can be analyzed at any time at the discretion of the project manager.

### 4.2 Data Measurement Time Period

Continuous PM<sub>10</sub> data will be collected and available in near real time for 8 hours prior and 16 hours following a disposal event. The continuous PM<sub>10</sub> instrument records an hourly average of PM<sub>10</sub> concentration as milligrams per cubic meter (mg/m<sup>3</sup>). These concentrations will be converted to micrograms per cubic meter (μg/m<sup>3</sup>). Metals and explosives residue concentrations will be expressed as μg/m<sup>3</sup>.

Metals and explosive residue analysis will be performed on the E-BAM Teflon tape for the time period when disposal operations are actually occurring as well as select background samples acquired during periods when no disposal operations are occurring. After the first month of monitoring and the analyses are complete, the sample analysis frequency will be re-evaluated.

The E-BAM will be configured to record concentrations at standard conditions (i.e., corrected for barometric pressure and temperature). This plan defines standard conditions as

760 millimeters mercury (mm Hg) and 25 degrees Celsius (°C). A 24-hour average will be calculated by averaging individual 15-minute averages. The 24-hour averaging period will last from midnight to midnight, Greenwich Mean Time.

Section 9, Data Management, describes the criteria for calculating the 24-hour average. Hourly averages of each meteorological parameter are stored in the E-BAM datalogger in units of degrees for wind direction and meters per second (m/s) for wind speed.

The E-BAM filter will be set to advance every 8 hours, providing three 8-hour composite samples available daily for analysis of metals and explosives.

### 4.3 Rationale for Location of Monitors

PM<sub>10</sub> monitors are sited according to EPA monitoring guidance documents. Site selection was based on several factors including DQOs, monitoring equipment configuration, site accessibility, proximity to the source area, security of the site location, and location with respect to the easterly wind direction. The entire LIA is considered as the detonation site. The VNTR's peninsular geography allows potential human health impacts only on its western boundary. Because of this and the predominant easterly wind direction, air monitors are located along a low ridge near the western side of the LIA and within the VNTR Surface Impact Area (SIA). The monitoring station locations are along a small ridge line over which plumes would have to pass to reach populated areas. This location allows for safe monitoring while being as near to the source of emissions as possible. The selected locations, which are within 1 mile of the detonations, provide a conservative assessment of the air quality impact of the TCRA on the public, which is over 4.5 miles west of the monitoring locations. Within the SIA, the choice of locations was heavily influenced by the lack of infrastructure (roads and an electric grid) in the area and the desire to perform monitoring with a minimum of environmental impact (clearing and leveling of land and access roads). The monitors were sited according to the prevention of significant deterioration (PSD) siting guidelines found in 40 *CFR*. The siting guidelines include placing the instrument a significant distance from trees, paved surfaces, large bodies of water, or other obstacles that could influence airflow and measurements at the station. A senior CH2M HILL technologist familiar with EPA siting criteria reviewed the monitor locations and confirmed that the objectives were met.

This protocol specifies locating three portable E-BAM air monitors at fixed sites to sample PM<sub>10</sub>, metals, and explosive residues. A fourth is to be used as a spare and to perform intermittent co-located sampling for determination of method precision.

## 5.0 Description of Monitoring Site

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The three portable E-BAM samplers are placed within the SIA near the western boundary of the LIA. The sites follow a low north-to-south ridge and are spaced between 1,000 and 3,500 feet apart.

### 5.1 Height of Measurements Aboveground

EPA siting guidelines call for the sample inlet to be 2 to 7 meters aboveground. Two of the E-BAM monitors are located on existing small structures with the inlet located 2 to 7 meters above the ground surface, which meets the EPA siting guidelines. One E-BAM is mounted on top of the OP1 structure to provide capture of any plumes that may rise above the other two monitors.

### 5.2 Nearby Obstructions

The monitoring sites for PM<sub>10</sub> were selected to meet the DQOs and according to the EPA summary of probe siting criteria, from the PSD monitoring guidance. Samplers are placed as close to the breathing zone as feasible while minimizing the effects of surrounding obstacles. The following criteria from the guidance was implemented:

- The sampler should be greater than 20 meters from the dripline and must be 10 meters from the dripline when trees act as an obstruction
- There must be an unrestricted airflow arc of 270 degrees around the sampler
- No furnace or incineration flues that emit particulate matter should be nearby
- The sampler must be 5 to 15 (or more) meters from roads

# 6.0 Description of Monitoring Instrumentation

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The monitoring instrumentation is manufactured by Met One Instruments of Grants Pass, Oregon. The Met One E-BAM automatically measures and records particulate concentration with built-in datalogging. It uses the principle of beta ray attenuation to provide a simple determination of mass concentration. A small  $^{14}\text{C}$  ( $< 60 \mu\text{Ci}$ ) element emits a constant source of high-energy electrons, also known as beta particles. The beta particles are efficiently detected by an ultrasensitive scintillation counter placed nearby. An external pump pulls a measured amount of air through a filter tape. Filter tape impregnated with dust is placed between the source and the detector, thereby causing attenuation of the measured beta particle signal. The degree of attenuation of the beta particle signal is used to determine the mass concentration of particulate matter on the filter tape. The mass is divided by the known volume of air passed through the filter tape to obtain the volumetric concentration of particulate matter in air.

Excerpts from the *E-BAM Particulate Monitor Operation Manual* are provided in the following sections. Appendix B contains the complete manual.

All meteorological sensors were selected to meet EPA performance and accuracy requirements as listed in the *Meteorological Monitoring Guidance and Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV*.

## 6.1 EPA Designation

The E-BAM method parallels the EPA Method Designation No.: EQPM-0798-122, August 3, 1998 for  $\text{PM}_{10}$ .

For this project the E-BAM will be operated with the BX-802  $\text{PM}_{10}$  sampling inlet for 24-hour averages (compiled from 15-minute averages), with a filter change frequency of 8 hours, with Teflon filter tape. The Teflon tape is being used to facilitate the analysis of metals and explosive residue. The instrument's "standard" flow rate of 16.7 liters per minute (L/min) will be used. The following options from Met One Instruments were chosen:

- Input Power: 12 Volt DC @ 35 watts
- Inlet Adaptor for Flow Calibration (Delta Cal)
- $\text{PM}_{10}$  Sampling Inlet (BX-802)
- Wind Speed/Direction Sensor for E-BAM Sampler (EX-034)
- Consumables and Manufacturer Recommended Spares

## 6.2 Principle of Operation

This section is divided into two sections. Section 6.2.1 gives the basic theory of operation of the E-BAM. Section 6.2.2 addresses the practical aspects of translating the basic theory of operation into an actual measurement.

## 6.2.1 Basic Theory of Operation

When the high-energy electrons emanating from the  $^{14}\text{C}$  interact with nearby matter, they lose their energy and, in some cases, are absorbed by the matter. The high-energy electrons emitted are known as beta rays, and the process is known as beta ray attenuation. When matter is placed between the  $^{14}\text{C}$  source and a device designed to detect beta particles, the beta rays are absorbed or their energy is diminished. Beta absorption results in a reduction in the number of particles detected. The magnitude of reduction in detected beta particles is a function of the mass of the absorbing matter between the  $^{14}\text{C}$  beta source and the detector.

The number of beta particles passing through absorbing matter, such as dust deposited on a filter tape, decrease nearly exponentially with the mass through which they must pass. Equation 1 shows this relationship.

### Equation 1

$$I = I_0 e^{-\mu x}$$

In Equation 1,  $I$  is the measured beta ray intensity (counts per unit time) of the attenuated signal (dust-laden filter tape),  $I_0$  is the measured beta ray intensity of the unattenuated signal (clean filter tape),  $\mu$  is the absorption cross-section of the material absorbing the particle ( $\text{cm}^2/\text{g}$ ), and  $x$  is the mass density of the absorbing matter ( $\text{g}/\text{cm}^2$ ).

Equation 1 very closely resembles the Lambert-Beers law used in spectrometric analysis. Just as the Lambert-Beers law is an idealization of what is observed, Equation 1 also is a simplification of the true processes occurring meant to simplify the corresponding mathematics. However, experimental measurement shows that in properly designed monitors, such as the E-BAM, use of this equation introduces no substantial error.

Equation 1 may be rearranged to solve for  $x$ , the mass density of the absorbing matter. This is shown in Equation 2

### Equation 2

$$-\frac{1}{\mu} \ln \left[ \frac{I}{I_0} \right] = \frac{1}{\mu} \ln \left[ \frac{I_0}{I} \right] = x$$

In practice, the absorption cross-section is experimentally determined during the calibration process. Once  $I$  and  $I_0$  are experimentally measured, it is a simple matter to calculate  $x$ , the predicted mass density.

In practice, air is sampled at a constant flow rate ( $Q$ ) for a specified time ( $\Delta t$ ). The sampled air is passed through a filter of surface area  $A$ . Once  $x$ , the mass density of collected particles, has been determined, it is possible to calculate the concentration of particulate matter ( $\mu\text{g}/\text{m}^3$ ) with Equation 3.

**Equation 3**

$$c \left( \frac{\mu\text{g}}{\text{m}^3} \right) = \frac{10^6 A(\text{cm}^2)}{Q \left( \frac{\text{liter}}{\text{min}} \right) \Delta t(\text{min}) \mu \left( \frac{\text{cm}^2}{\text{g}} \right)}$$

In Equation 3,  $c$  is the particulate concentration ( $\mu\text{g}/\text{m}^3$ ),  $A$  is the cross-sectional area on the tape over which dust is deposited ( $\text{cm}^2$ ),  $Q$  is the rate at which particulate matter is being collected on the filter tape ( $\text{L}/\text{min}$ ), and  $\Delta t$  is the sampling time (minutes). Combining these equations yields the final expression for the particulate concentration in terms of measured quantities (Equation 4).

**Equation 4**

$$c \left( \frac{\mu\text{g}}{\text{m}^3} \right) = \frac{10^6 A(\text{cm}^2)}{Q \left( \frac{\text{liter}}{\text{min}} \right) \Delta t(\text{min}) \mu \left( \frac{\text{cm}^2}{\text{g}} \right)} \ln \left( \frac{I_0}{I} \right)$$

The key to the success of the beta attenuation monitor is due in part to the fact that  $\mu$ , the absorption cross-section, is almost insensitive to the nature of the matter being measured. This makes the E-BAM very insensitive to the chemical composition of the material being collected.

It is instructive to perform a conventional propagation of errors analysis on Equation 4. Doing so, one can develop an equation for the relative measurement error ( $\sigma_c/c$ ) as a function of the uncertainty in each of the parameters comprising Equation 4. This leads to Equation 5.

**Equation 5**

$$\frac{\sigma_c}{c} = \sqrt{\frac{\sigma_A^2}{A^2} + \frac{\sigma_Q^2}{Q^2} + \frac{\sigma_t^2}{t^2} + \frac{\sigma_\mu^2}{\mu^2} + \frac{\sigma_I^2}{I^2 \ln \left[ \frac{I}{I_0} \right]^2} - \frac{\sigma_{I_0}^2}{I_0^2 \ln \left[ \frac{I}{I_0} \right]^2}}$$

Inspection of Equation 5 reveals several things. The relative uncertainty of the measurement ( $\sigma_c/c$ ) is improved by increasing the cross-sectional area of the filter tape ( $A$ ), the flow rate ( $Q$ ), the sampling time ( $t$ ), the absorption cross section ( $\mu$ ), and  $I$  and  $I_0$ . In practice, the uncertainty associated with the filter area ( $\sigma_A/A$ ) may be minimized by ensuring that the tape is in exactly the same position during the  $I_0$  measurement as in the  $I$  measurement. Proper design of the shuttle mechanism inside of the E-BAM will lead to minimal error here.

The uncertainty in flow rate ( $\sigma_Q/Q$ ), may be minimized by properly controlling the flow of the instrument. For the standard E-BAM, this value is on the order of  $\pm 3$  percent. For the E-BAM equipped with the optional mass flow controller device, ( $\sigma_Q/Q$ ) decreases to  $\pm 1$  percent.

The relative error due to the uncertainty in the absorption cross-section ( $\sigma_{\mu}/\mu$ ), is the result of its slight variation as a function of the chemical composition of the matter being monitored. Generally, this relative error is on the order of  $\pm 2$  to 3 percent, with judicious selection of the calibrated value of  $\mu$ .

The uncertainty associated with the measurement of  $I$  and  $I_0$  pertains to the physical nature of the process leading to the emission of beta particles from the decay of  $^{14}\text{C}$ . The process follows Poisson statistics. Poisson statistics show that the uncertainty in the measurement of  $I$  ( $\sigma_I/I$ ) and  $I_0$  ( $\sigma_{I_0}/I_0$ ) are minimized by increasing the sampling time. Mathematical analysis shows that doubling the sampling time and hence the measured intensity of  $I$  or  $I_0$  will reduce the uncertainty of the measurement by a factor of 1.41 (square root of 2).

### 6.2.2 Practical Considerations

The E-BAM uses a sampling algorithm that optimizes the total time required to complete a cycle. The basic cycle always includes an automatic calibration performed during the sampling period, but at a different point on the filter tape, as the data is being sampled.

The initial count of clean filter tape ( $I_0$ ) is performed at the beginning of the cycle for a period of 4 minutes. The filter tape is advanced four windows, and the sampling (vacuum pumping) begins on the spot in which  $I_0$  was just measured. Air is drawn through this spot on the filter tape for a predetermined time period (50 to 470 minutes).

At the same time, the second count ( $I_1$ ) occurs (at a point on the tape four windows back) for a period of 4 minutes. The purpose of this measurement is to perform compensation for instrument drift caused by varying external parameters such as temperature and relative humidity. A third count ( $I_2$ ) occurs with the reference membrane extended over the same place on the tape. The sample time should be greater than or equal to 5 minutes, so as to allow for the overlapping auto calibration time. The purpose of this measurement is to verify that the instrument is operational.

The tape is moved back four windows to measure the beta ray absorption through the section that has collected dust ( $I_3$ ). Finally the concentration calculation is performed to complete the cycle. A new measurement cycle then begins.

## 6.3 Calibration Procedures and Frequency

The E-BAM consists of three basic components: the detector/logger, the pump, and a sampling inlet. Each component is self-contained and may be easily disconnected for service or replacement.

Met One Instruments maintains a complete test facility for calibration of all particulate instrumentation. Each instrument is tested to traceable standards and then operated in a chamber with dust-laden atmosphere. The test results provide data points used for data regression and final calibration. The built-in membrane calibrator is tested to ensure conformity during the test period.

- Based on field experience, the user will need to service the pump every 2 years. Built-in firmware performs hourly diagnostic tests on the unit and tests for correct operation of all components. The flow system is verified every 2 weeks and calibrated when the

monitor reaches the warning limit of  $\pm 2$  percent of 16.67 L/min. A warning limit has been devised so drift will be minimized and identified before an out-of-range limit of  $\pm 4$  percent is reached.

- For the E-BAM to operate in actual conditions and report data in standard conditions, the “standard” flow rate option must be selected. This option enables the E-BAM to pull 16.7 *actual* L/min through the PM<sub>10</sub> inlet head. The E-BAM datalogger then converts actual flow to standard using the internal pressure and outside temperature (OT) data. The flow and PM<sub>10</sub> concentration data are then corrected to standard temperature and pressure and stored in the datalogger.

Primary calibration of the meteorological sensors is performed by the manufacturer. Recalibration is not usually required in the field; however, if visual inspection of the instruments indicates a problem exists, the manufacturer will be consulted to troubleshoot and rectify the malfunction. Meteorological instruments that can be calibrated in the field will be calibrated upon installation and annually thereafter.

Calibration procedures for the meteorological sensors and the particulate samplers are included in Appendix C.

## 6.4 Description of Calibration System to Be Used

### 6.4.1 Automatic Calibration Method

The E-BAM has a built-in mass membrane calibrator. The membrane is automatically moved into the beta particle pathway to determine the “mass” of the membrane each hour or when the filter tape advances. Each membrane has a factory-verified mass, and that value is stored in the E-BAM. When the hourly membrane calibration is made, the computed value is compared to the stored factory value to determine proper operation. The membrane must be withdrawn for normal measurements. Should the instrument fail to perform to specification, an error flag is logged in memory, and data collected subsequently are flagged. The data coordinator then invalidates data that have been flagged during data review. Failure of this test will result in further troubleshooting, such as cleaning of the instrument or other procedures based on consultation with the manufacturer.

Zero testing of blank filter paper is performed at the beginning and end of each sample period (in this case, hourly) to ensure the stability of the measurement system. Zero testing is based on the ability of the E-BAM to hold a constant output when measuring blank filter paper. If the difference between the two values exceeds the factory preset limit of  $\pm 2$  percent, a data error message is logged in the error log and the digital data value is flagged.

### 6.4.2 Field Verification and Calibration of Flow System

The site operator conducts monthly flow verification of the E-BAM. The flow check is performed before any instrument adjustment is made. The resulting data from checks are used to measure precision. Flow in the E-BAM is compared to a primary flowmeter. If the results of the flow check do not fall within the project’s warning threshold of  $\pm 2$  percent, the instrument is adjusted and rechecked. If the instrument flow checks fall outside the

4 percent criterion, data from the date of the failed flow check back to the last successful flow check may be suspect. The data coordinator may choose to re-calculate using an integrated flow for this time period.

For the E-BAM to operate in actual conditions and report data in standard conditions, a “standard” flow rate option must be selected. This option enables the E-BAM to pull 16.7 *actual* L/min through the PM<sub>10</sub> inlet head. The E-BAM datalogger then converts the actual flow to standard using the internal pressure and temperature sensors. The flow and PM<sub>10</sub> concentration data are then stored in standard conditions in the datalogger.

The flow calibration also requires checking and adjusting (if necessary), the OT sensor and the internal pressure sensor of the instrument.

The E-BAM used for this monitoring program is equipped with a volumetric flowmeter. Volumetric flow measures the volume of flow in actual conditions. Volumetric flow check and calibration requires a reference volumetric flowmeter with a flow audit cap. A DeltaCal volumetric flow calibration kit will be used to perform flow checks and calibrations. This flowmeter is a primary standard and no calibration is necessary. However, as part of the QA requirements of this project, the flow standard is factory certified once every year. As required by EPA guidance, annual certification calibrations are performed on the temperature and barometric pressure transfer standards. All certification documentation is kept on file with the data manager. The procedure for conducting a field flow verification and calibration is provided in Appendix C, as well as procedures for checking and adjusting the temperature sensor and the internal instrument pressure.

# 7.0 Chemical Analysis of Particulate Samples

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The E-BAM instrument determines PM<sub>10</sub> concentrations by collecting particulate on a special Teflon fiber tape. One sample is collected onto a “sample window” on the tape during the sampling period. Following completion of the PM<sub>10</sub> analysis, the fiber tape is wound onto a spool. Samples of the fiber tape can be collected and sent to an outside laboratory for additional analysis.

For this study, samples of the Teflon fiber tape will be collected and analyzed for metals and explosive residue content to determine whether the TCRA events result in an increase of these analytes above existing levels. To determine any potential contribution, “sample windows” of the fiber tape will be collected and analyzed. Samples collected during periods of time when no TCRA events are conducted will not be collected and analyzed for metals/explosives except for the purposes of determining “background” concentrations.

## 7.1 Sample Collection

PM<sub>10</sub> data will be available in near real-time and the analytical data will be available on a fast-track basis. It will be immediately known if there is a short-term exceedance of the 24-hour NAAQS for PM<sub>10</sub>. PM<sub>10</sub> is to be used as the real-time screening criteria. Samples will be removed from the units within 48 hours after completion of each of the periodic detonation campaigns and sent for analysis. Every week when there are disposal operations, samples of the Teflon fiber tape will be collected from the E-BAM instruments. The fiber tapes will be indexed to allow individual “sample windows” to be correlated with specific dates and times. The field technician will obtain a log detailing each of the disposal events and the corresponding dates and times over the course of the previous week. The technician will then remove the historical fiber tape from the takeup spool on the E-BAM using powder-free latex or nitrile gloves (powdered gloves use an aluminum compound in their formulation, and must not be used for this study). The tape will be packaged in air-tight packaging and priority shipped to the analytical laboratory (Cooper Environmental) for processing. In addition to the collected samples, the field technician will obtain the PM<sub>10</sub> data, actual airflow passed through the fiber tape for each sample, and meteorological data for each sample period.

In the analytical laboratory, the “sample windows” corresponding to the period prior to a detonation event and following a disposal event will be located. Each sample window will be cut from the fiber tape using stainless steel scissors which have been pre-rinsed using a dilute nitric acid solution. Any handling of the fiber tape or cut out “sample windows” should be performed using stainless steel forceps which have also been rinsed in a dilute nitric acid solution. Both the forceps and the scissors will be rinsed three times with the dilute acid after each sample is cut to minimize the possibility of cross-contamination. Each fiber sample will be placed in a clean polyethylene or polystyrene sample container using acid-cleaned forceps. The container will be labeled with the sample location, and the date and time the sample was collected.

## 7.2 Laboratory Analysis

Particulate samples collected on the Teflon fiber will be analyzed at Cooper Environmental. Preparation and analysis of the samples will be in accordance with applicable EPA guidance. The analytical scope and a summary of the QA/QC protocol are provided in this document. Method-specific QC requirements are provided in the referenced analytical methods (see Appendix D).

Metals analysis will be performed by a modified version of “X-ray Fluorescence Analysis of PM<sub>2.5</sub> Deposits on Teflon Filters”. This method is based on EPA Method IO-3.3 and will be performed by Cooper Environmental. Explosive residue determination will be accomplished by EPA Method 8095.

### 7.2.1 Metals by X-Ray Fluorescence

The Teflon fiber samples are analyzed for metals by X-ray Fluorescence (XRF) based on Method IO-3.3 (Inorganic Compendium). Each sample will be removed from its container with properly cleaned tweezers, visually inspected, and placed in the XRF instrument. Specific targeted x-ray radiation will excite the sample. The resulting spectra will undergo deconvolution by a least squares algorithm and correction for any spectral interferences. Metal results will be reported as  $\mu\text{g}/\text{m}^3$ . Target metals and their respective detection limits are listed in Table 7-1.

X-ray fluorescence is a nondestructive analytical technique and must be performed before any other laboratory analysis.

TABLE 7-1  
Target Analyte List and Reporting Limits

Compound	Abbreviation	Target Reporting Limit
Chromium	Cr	0.03 $\mu\text{g}/\text{m}^3$ (8hr sample)
Cadmium	Cd	0.05 $\mu\text{g}/\text{m}^3$ (8hr sample)
Iron	Fe	0.005 $\mu\text{g}/\text{m}^3$ (8hr sample)
Phosphorous	P	0.1 $\mu\text{g}/\text{m}^3$ (8hr sample)
Nickel	Ni	0.005 $\mu\text{g}/\text{m}^3$ (8hr sample)
Copper	Cu	0.004 $\mu\text{g}/\text{m}^3$ (8hr sample)
Tin	Sn	0.05 $\mu\text{g}/\text{m}^3$ (8hr sample)
Arsenic	As	0.003 $\mu\text{g}/\text{m}^3$ (8hr sample)
Mercury	Hg	0.006 $\mu\text{g}/\text{m}^3$ (8hr sample)
Lead	Pb	0.005 $\mu\text{g}/\text{m}^3$ (8hr sample)

### 7.2.2 Explosives by Method 8095

Explosive residues will be determined by EPA Method 8095. The sample is extracted with acetonitrile and the extract injected into a gas chromatograph (GC) equipped with an

electron capture detector (ECD). The compounds are resolved on a fused-silica capillary column, identified by the ECD, and quantitated against a calibration curve. Results will be expressed as  $\mu\text{g}/\text{m}^3$ . Target analytes are listed in Table 7-2.

**TABLE 7-2**  
Target Analyte List and Reporting Limits

Compound	Abbreviation	Target Reporting Limit
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	250 ng/filter
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	50 ng/filter
Nitrobenzene	NB	250 ng/filter
1,3-Dinitrobenzene	1,3-DNB	50 ng/filter
1,3,5-Trinitrobenzene	1,3,5-TNB	100 ng/filter
2,4-Dinitrotoluene	2,4-DNT	50 ng/filter
2,6-Dinitrotoluene	2,6-DNT	50 ng/filter
2,4,6-Trinitrotoluene	2,4,6-TNT	50 ng/filter

### 7.3 Determination of Analytical Reporting Limits

The clean, unused Teflon fiber tape used by the E-BAM instrument, the sample containers, and all handling steps in collecting the particulate samples may contribute to the elevation of reporting limits for all methods: XRF and GC/ECD.

### 7.4 Laboratory Data Reporting

Analytical results will be reported by the laboratory within 4 weeks of sample collection. The laboratory shall provide a Level II equivalent data package on 5 percent of the data for the purpose of data validation. The remainder of the data shall be a Level 1 deliverable.

## 8.0 Quality Assurance Program

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The main DQO for this project is to document the impact of air emissions from the TCRA on public human health and the environment. Quality assurance consists of two distinct, yet equally important, functions in meeting the DQOs. The first function is to assess the quality of the monitoring data by determining its precision and accuracy. "Precision" is a measure of the repeatability of data results obtained during a given monitoring period. "Accuracy" is a measure of closeness of an observed measurement to the true value. The second function is to control and improve the quality of the data by implementing QC policies, procedures, and corrective actions. These two functions together produce quality data that are acceptable for further data analysis. The purpose of this section is to describe the precision and accuracy of each measurement and give an overview of the QC effort of the program.

This document was developed based on accepted procedures and guidance documents for conducting monitoring. These guidance documents were developed by the EPA. To provide a uniform assessment of data quality, the data assessment procedures used for guidance are specified explicitly in Sections 5, 6, and 7 of *Ambient Monitoring Guidelines for Prevention of Significant Deterioration* (EPA-45-0/4-80-012). These guidelines are minimum QA guidelines established for the control and assessment of the quality of monitoring data. It is necessary to conduct sufficient QA activities to ensure that the data measurements are acceptable, complete, precise, accurate, representative, and comparable. Such activities are conducted in accordance with EPA guidelines and equipment manufacturers' procedures, and include, but are not limited to, the following tasks:

- Document and revise, when necessary, methods used for monitoring and analysis
- Describe calibration procedures appropriate to each method
- Use working standards traceable to National Institute of Standards and Technology (NIST) standard reference materials (SRM)
- Maintain QC charts and perform corrective action when an out-of-control condition is detected
- Perform regular preventive maintenance according to manufacturers' specifications
- Adequately train staff who participate in the monitoring project
- Maintain a data completeness objective of 80 percent

## 8.1 Quality Assurance Program Objectives

Quality assurance procedures ensure that data generated by this monitoring program will be of sufficient and adequate quality for their intended use. The following performance criteria must be met to ensure the DQO:

- Conduct all field operations in a manner consistent with published EPA monitoring guidance documents where possible. This will ensure high-quality and defensible data.
- Ensure that all field log records are complete to minimize data loss that could result from record-keeping errors.
- Ensure that all calculated data are reported correctly.
- Follow proper sample handling, shipment, and chain-of-custody (COC) procedures.
- Adhere to all analytical QA/QC requirements.

This project-specific document defines the procedures that must be followed to meet the above objectives.

The following parameters are used to define the success in meeting the above objectives:

- Completeness
- Accuracy
- Precision
- Sensitivity

**Completeness** is the ratio of the number of actual valid data obtained through the measurement system to the number of data points expected or possible under normal conditions. Completeness will be calculated for the overall project as follows:

$$\text{Data completeness} = \frac{\text{Number of valid hours}}{\text{Number of possible hours}} \times 100$$

The data capture goals for this project are to monitor for 8 hours prior and 16 hours following a TCRA event. This project has set a completeness goal of 80 percent, which exceeds the goal based on EPA completeness criteria as defined in the previously referenced guidance documents. To meet this completeness goal, 80 percent of 8 hours prior and 16 hours following a TCRA event must be consolidated.

**Accuracy** is a measure of the agreement between an experimental determination and the true value of the parameter being measured. Accuracy is estimated through the use of known reference materials or matrix spikes. It is calculated from analytical data and is not measured directly. Spiking of reference materials into a sample matrix is the preferred technique because it provides an assessment of the matrix effects on analytical accuracy. Accuracy, defined as percent recovery (%R), is calculated as:

$$\%R = \left[ \frac{(SSR - SR)}{SA} \right] \times 100$$

where SSR is the spiked sample result, SR the sample result (native), and SA the spike concentration added to the spiked sample.

Field accuracy methods comprise actual versus recorded flow rate measurements determined during field audits.

**Precision** is a measure of the agreement or reproducibility of a set of replicate results obtained from duplicate analyses made under identical conditions. Precision is estimated from analytical data and cannot be measured directly. The precision of a duplicate determination can be expressed as the relative percent difference (RPD), as calculated as

$$RPD = \left\{ \frac{(|X_1 - X_2|)}{(X_1 + X_2)/2} \right\} \times 100 = \left\{ \frac{|X_1 - X_2|}{\frac{(X_1 + X_2)}{2}} \right\} \times 100$$

where  $X_1$  is the result from the native sample and  $X_2$  that from the duplicate sample.

Precision is determined for the PM<sub>10</sub> flow rate by conducting biweekly flow checks. It is defined as the comparison of the same measurement with two separate sampling systems under identical conditions. A co-located sampler will be temporarily installed at one of the sites to evaluate precision for the sampling and analysis program.

**Sensitivity** is a measure of how low of a concentration an analysis can reliably detect and report a particular analyte. Higher sensitivities allow the laboratory to establish method detection limits (MDLs) and reporting limits (RLs) at sufficiently low levels so that the project DQOs can be achieved.

## 8.2 Project Safety

The project will follow the guidance outlined in the *Explosives Safety Submission Expanded Range Assessment/Phase I Site Inspection, Former Vieques Naval Training Range and the Time Critical Removal Action/Interim Measures Work Plan, Surface Munitions and Explosives of Concern at Munitions Response Area-Live Impact Area, Munitions Response Sites 1 through 8, 13,15,20, and 29 through 31, the Time Critical Removal Action/Interim Measures (TCRA/IM) Work Plan*, as well as all established CH2M HILL health and safety practices.

## 8.3 Field Documentation Procedures

Operational tasks are logging day-to-day status of equipment, including noting the date, time, and operator's initials in the logbooks at the site upon each site visit; providing for continuous data collection; and troubleshooting and correcting any operational problems. The field operators accomplish these tasks by visiting the station a minimum of every week and completing the site visit checklist (see Appendix E). The operator routinely sends copies of these reports to the data manager.

Operators are responsible for maintaining the monitor in a neat and orderly manner and for performing some remedial maintenance on a regularly scheduled basis, as defined in the numerous standard operation procedures in this document. These procedures and other corrective action meet the project's QA and data completeness goals.

A bound field logbook is kept for the project. Every time an instrument receives maintenance or QA checks are performed, such as flow checks, the results are recorded. Calibrations, maintenance, adjustments, out-of-control, relocations, downtime, or any other events pertinent to each instruments' operation are recorded.

Other important documents such as calibration certificates, performance audit assessments, and other pertinent information will also be kept in the project notebook as well as in the central file with the data manager.

### 8.3.1 Field Records

Field operators are required to keep accurate written records of their activities in a bound logbook. All entries should be legible, written in waterproof ink, and contain accurate and inclusive documentation of an individual's field activities, including observations, any problems encountered, and actions taken to solve the problem. Data recorded in the field logbook include site visits, maintenance and calibration activities, field measurements, conditions, and other information pertinent to sample collection. Entry errors or changes should be crossed out with a single line, dated, and initialed by the person making the correction. Entries made by individuals other than the person to whom the logbook was assigned are dated and signed by the individual making the entry. Field logbooks are available to the QA manager for review.

### 8.3.2 Site Visits

The project QA program calls for two site visits during the first week of operation and visits every other week thereafter. The SOP for site visits (see Appendix C) describes the procedures for the site visit. The PM<sub>10</sub> monitor is visited a minimum of every other week. Any needed maintenance or troubleshooting for the monitors will take place at this time.

As part of the QA program, a site visit log was developed to guide technicians performing site visits (see Appendix E). This form can be used to log all activities at the site, even when an inspection is not conducted. Because the project does not have a backup datalogging system for recording raw data such as a strip chart, it is important to have site documentation that includes written parameter values. In this manner, the data manager is able to use the site documentation as an additional tool for data validation.

The originals of the completed log sheets are kept with the data manager. This way the data manager can trace data anomalies, if necessary, and the field operator has access to specific site information.

## 8.4 Quality Assurance for PM<sub>10</sub> Monitoring

### 8.4.1 Calibration Frequency

PM<sub>10</sub> instruments are calibrated as part of the QC program. The E-BAM is factory calibrated. The only user calibration is a flow verification calibration that may include verification of the temperature and internal barometric pressure sensor. Before any adjustments are made to the instrument, the flow is verified against a flow standard and then adjusted if the percent difference from the standard is greater than  $\pm 2$ . These calibrations are done onsite before monitoring commences and routinely throughout the monitoring program as outlined below:

- After any repair work that may alter the operation of the instrument
- When an instrument has been moved
- At least once a quarter or whenever the instrument has failed a performance audit or a QC flow check

Calibration results are recorded on the PM<sub>10</sub> calibration/flowcheck/audit data sheet. A copy of this is provided in Appendix F. The instrument flow calibration procedure is presented in Appendix C. This field procedure is also conducted by the site operator during each site visit. Site visits are conducted on a routine basis and occur approximately every other week.

### 8.4.2 Calibration of Transfer Standards

The transfer standards used for wind speed, wind direction and flow measurements will be calibrated annually against NIST standard reference materials. The DeltaCal flowmeter is a certified transfer standard, but will still be recalibrated and certified annually against NIST traceable standards by a laboratory or the manufacturer.

## 8.5 Quality Assurance for Meteorological Instrumentation

Meteorological instruments are calibrated, operated, maintained, and repaired according to CH2M HILL SOPs and the recommendations of the manufacturer's operations and maintenance manual. Calibration procedures follow requirements as outlined in *Quality Assurance Manual for Meteorological Monitoring Systems, Volume IV* (EPA-600/4-82-060).

### 8.5.1 Calibration Frequency

If the TCRA requires more than 1 year to complete, the instruments will be calibrated annually at the factory under controlled conditions using such equipment as wind tunnels and temperature chambers. Field calibrations are also performed in the field upon station installation, if significant repairs are required, and if field checks or performance audit results warrant.

## 8.6 Quality Assurance for Laboratory Samples

Laboratory quality control samples including method blanks, laboratory control samples (LCS), and duplicates are analyzed regularly by the laboratory to ensure that the analytical instruments are performing at optimum levels. A summary of the laboratory QA/QC procedures is provided in this document. Method-specific QC requirements are provided in the referenced analytical methods.

Analytical instruments will be calibrated using NIST traceable standards in accordance with manufacturers' directions, method requirements, and SOP specifications. Records of standard preparation and instrument calibration shall be maintained. The analysis run log, maintained for each analytical instrument shall include: date/time of calibration, analyst initials, standard used, and concentration.

### 8.6.1 Blanks

Method blanks are used to monitor a system for interferences and contamination from glassware, reagents, etc. The blank is taken through the entire sample preparation process and is included with each analytical batch, or every 20 samples, whichever is more frequent.

Field blanks are included with each sample set for each monitoring site. Field blanks monitor the integrity of the sampling process.

### 8.6.2 Laboratory Control Sample

Laboratory Control Samples are defined as reagent water spiked with a known amount of analytes from a source different than that used for the calibration standards. This parameter is used to evaluate the validity of the calibration standards and the standard preparation procedure.

### 8.6.3 Duplicate

Depending on the method, duplicate samples are analyzed with a frequency of 5 percent (1 per 20 samples). Duplicate samples that do not meet established criteria will be reanalyzed and evaluated by the laboratory personnel and project chemist.

### 8.6.4 Surrogate Compounds

Gas chromatography analyses include the addition, detection, and recovery of surrogate spiking compounds. Method-specific surrogates are added to every sample during the sample preparation step. This parameter evaluates matrix effects and sample preparation techniques.

### 8.6.5 Instrument Stability

For each XRF analysis batch or everyday samples that are analyzed, a QC standard is analyzed. Measured concentrations of this standard which contains Cd, Ni, Se, Si, Pb, and V are compared with initial calibration concentrations. Agreement must be within  $\pm 5$  percent or all samples must be reanalyzed.

## 8.6.6 Run-time Quality Control

XRF analyses utilize run-time QC procedures to indicate instrument performance at the time of data acquisition. The parameters that are measured and their significance are: peak areas (monitors change in sensitivity), background areas (monitors contamination or background changes), centroid (monitors gain and zero adjustment), and FWHM (monitors degradation of the detector resolution). These four parameters are monitored from sodium to lead and include atmospheric argon.

## 8.6.7 Chain of Custody

Proper sample handling, shipment, and maintenance of COC are key components of the quality system designed to obtain data that can be used to make project decisions. It is very important that all sample handling protocols and COC requirements be followed completely, accurately, and consistently.

A properly completed COC form will accompany samples to the laboratory. The unique sample IDs and descriptive identification information (site location, date, time, etc.) will be listed on the COC form. When transferring possession of samples, the individuals relinquishing and receiving them will sign, date, and note the time on the COC form.

The original record will accompany the shipment, with field copies being retained by the sampler. Upon receipt of field samples, the analytical laboratory representative will sign the COC to accept custody of the samples and will then properly store them to await analysis.

## 8.7 Performance Audits and Procedures

Auditing is an independent function that is to be performed under its own set of procedures. The purpose of the audit is to inspect the calibration and test the instruments with known inputs and accurately measure the system's responses to determine instrument performance and accuracy. Quality assurance audits and process evaluations must be performed to determine the accuracy of the monitoring system and hence the data it produces. The audits are performed by experienced CH2M HILL air monitoring personnel who are not responsible for the day-to-day operation of the station. This section describes the procedures for conducting the performance audits for the particulate monitor and meteorological sensors.

### 8.7.1 Project Audit Requirements

EPA quality assurance guidelines call for a performance audit to be conducted within 30 days of installation of a new station. The initial audit will be conducted as part of installation. The performance audit will be conducted in accordance with EPA guidelines as described in the documents previously referenced in the plan. The audit will be conducted by individuals experienced in air monitoring performance audits who are not involved in the routine operation of the station. Audits consist of challenging the monitor with a series of test equipment to determine operational characteristics.

Quality assurance documents also require performance audits for particulate monitoring to be conducted every 3 months (quarterly) and before station shutdown. The initial audit will also include a systems audit to ensure the station's SOPs are in accordance with applicable

monitoring requirements. EPA QA documents suggest that a systems audit should be conducted once per year.

Audit procedures for the E-BAM call for comparing the audit flow rate measured by the audit device to the indicated sampler flow rate. Flow rates measured in L/min are compared at actual conditions of temperature and pressure. Field measurements of temperature and pressure are recorded using certified equipment traceable to NIST standards. The difference between the audit flow and the E-BAM indicated flow must be within  $\pm 4$  percent to pass the audit.

The E-BAM is operated under normal conditions as part of the performance audit. A measurement of the indicated sampler flow rate is determined using a BGI, Inc. deltaCal or equivalent flow measurement device. This flow monitor shall be certified traceable to NIST.

### **8.7.2 Audit Procedures**

The audits will be performed by experienced individuals who are not involved in the routine operation of the station. After completion of each audit, each instrument will be calibrated, if necessary, according to the manufacturers' specifications. The auditor will review the preliminary findings with the station operator after completion of the field audit. A summary report will be completed after each audit.

To meet this project's DQOs, performance audits are conducted on a quarterly schedule. Audits are conducted in accordance with EPA-defined criteria. For particulate monitoring, EPA guidance suggests conducting audits within 30 days of station startup, every quarter during operation, after significant downtime or major repairs, if the station is relocated, and within 30 days of station shutdown.

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 explanations and information that will help the auditor to determine possible causes of discrepancies between audit-standard values and the sampling equipment values.

### **8.7.3 PM<sub>10</sub> Audit**

Observe and record the following parameters on the E-BAM Calibration/Flowcheck/Audit Data form:

- Sampler location, date, and time
- Sampler model, serial number, and calibration relationship
- Temperature and barometric pressure
- Unusual weather conditions
- Audit transfer standard serial number and calibration relationship

A copy of the form is provided in Appendix F. The pressure, temperature, wind speed and wind direction sensors are also included in the audit.

Calculate the percentage difference between the monitor's indicated flow rate and the corresponding audit flow rate determined from the audit transfer standard by using the equation on the audit data sheet. If the audit flow rate percentage difference is less than or

equal to  $\pm 4$  percent, then the audit is acceptable. If this criteria is not met, the flow meter must be recalibrated. This data will be documented in the Quarterly Data Reports to the client.

#### **8.7.4 Audit Reporting**

The CH2M HILL auditor leaves copies of the audit data sheets at the monitoring site at the conclusion of the audit. The results of the quarterly performance audits are tabulated and presented in a summary report. The report contains the results of the audit and points out discrepancies in station operational procedures with respect to EPA monitoring guidelines. Recommendations for corrective actions are also included in the report.

### **8.8 Preventive and Corrective Maintenance**

To minimize data loss due to instrument malfunction, certain maintenance steps are taken. Spare parts inventories and preventive maintenance schedules have been developed for each monitoring instrument. These schedules are contained in the instrument SOPs (see Appendix C). All maintenance steps taken, scheduled or unscheduled, must be documented in the station logbook and on forms that are kept in the central file.

Proper operation of the instrument is monitored in real time through a satellite link to each instrument. It is important to correct instrument malfunctions as soon as they are identified. The spare instrument and spare parts inventory should minimize data loss due to sensor malfunction. The station operator will be responsible for routine maintenance. The site operator will also provide initial troubleshooting, maintenance, and repair. CH2M HILL's air monitoring staff are also available to provide prompt troubleshooting and maintenance. Corrective actions will be recorded in the field logbook and submitted to the data manager for use in data validation.

The data manager and QA manager will be jointly responsible for approving and initiating corrective action after the data have been reviewed. Changes will be documented, dated, and signed. When it is necessary to make changes in the field, they will be made by the field operator after the field operator communicates with the data manager or QA manager.

### **8.9 Quality Assurance Reports to Management**

The hourly  $PM_{10}$  data will be averaged for each 24-hour sampling period. The meteorological data is also processed into hourly average values. All monitoring data will be processed and summaries will be reported quarterly. A description of QA activities conducted during the quarter will be included in the quarterly report. This will include audits, preventive maintenance, and repair activities.

### **8.10 Site Operator Training**

CH2M HILL has implemented recruitment, screening, and training protocols to ensure that station operators are qualified. Training is completed by all new station operators before they are allowed to operate field stations independently. Training will begin during site installation and continue on a routine basis throughout the project duration.

# 9.0 Data Management

---

The VNTR monitoring program consists of hourly PM<sub>10</sub> data collected continuously before and after an open detonation. This method of data collection depends on the field operator's attention to the project's QA/QC tasks and careful documentation of these tasks on the field data sheets and in the project logbook. The field operator is the first level of data screening, and the data manager or QA manager the last. This section describes the tasks that need to be completed to manage the project data in a reliable manner.

## 9.1 Data Management

PM<sub>10</sub> concentrations are calculated by the E-BAM datalogger from the volume of air passing over filter tape, the beta attenuation of the particulate mass filter tape, and the sample time. Valid data depends on the field operator carefully monitoring and recording the flow rate and other operational characteristics.

### 9.1.1 Field

Data management for the PM<sub>10</sub> monitor and the meteorological sensors begins in the field at the monitoring site. Criteria have been established to assist the site operator in determining whether a sample is valid. During a site visit, the operator checks and records the operational parameters of the E-BAM since the instrument's internal logger does not store this type of data. This will include logging the date, time, and previous hourly concentration value on the Site Visit Log.

The site technician will download the E-BAM error log to determine if any anomalies have occurred during data collection.

During each site visit, the site operator will conduct a flow check verification. The volumetric flow rate of the E-BAM must be within  $\pm 4$  percent. If the flow check results exceed this amount the operator will recalibrate and adjust the flow rate.

Meteorological parameters will be checked during each site visit to assess reasonability with current conditions.

### 9.1.2 Validation of Database Data

Once the data are downloaded, the data manager checks the data for flags set by the instrument's internal logger. The data manager will review the results from the field QA checks performed on the monitor, as well as the quarterly performance audit results for further determination of data validity.

Data entered into the database are randomly checked by hand by the QC manager against the raw data, including temperature, barometric pressure, observed hourly concentration data recorded by the site operator during each site visit, and the sampler's calibration information. Statistical outliers are back-checked. The data are then incorporated into the report.

## 9.2 Automated Data Collection

Data will be collected through a satellite data link that will provide near-real time data for PM<sub>10</sub>, wind speed, and wind direction. Data will be reported on a private or public Web site, at the discretion of the Navy.

CH2M HILL will maintain the raw data before validation. These raw data are not altered and are stored separate from the database. A copy of the raw data entered into the database forms the basis for data validation. Validation procedures will be documented and kept on file with CH2M HILL's data manager.

### 9.2.1 Logged Error Flags

Any flagged hours will be investigated and data invalidated. These errors are set by the E-BAM's internal logger and indicate out-of-range conditions or mechanical problems. The E-BAM Operators' Manual (see Appendix B) contains a complete list of these errors.

### 9.2.2 PM<sub>10</sub> Data

#### 9.2.2.1 Out-of-Control Flowchecks

If the routine flowcheck or performance audit results fall outside the control limit of  $\pm 4$  percent, the PM<sub>10</sub> data may be recalculated with an integrated flow over the suspect time period.

#### 9.2.2.2 Negative Data

Negative hourly values will be corrected to zero. Repeating values or an abundance of  $-5 \mu\text{g}/\text{m}^3$  values should be suspect and the field operator notified. If either case occurs, check for proper operation of monitor or for background drift using an appropriate zero air filter manufactured by Met One.

#### 9.2.2.3 Daily Data Patterns

E-BAM data should express somewhat routine characteristics. "Flat" or erratic hour-to-hour mass values may be the result of an operational malfunction. Changes in weather such as rain or seasons should influence "normal" daily values.

#### 9.2.2.4 Q<sub>tot</sub> Validation Criteria

The "Q<sub>tot</sub>" value recorded in the E-BAM datalogger is the average hourly volume measured during each sample. Fifty minutes of a 16.67 L/min flow equals 0.8335 m<sup>3</sup>:

$$\frac{(50 \text{ minutes}) (16.67 \text{ L}) (1 \text{ m}^3)}{(1 \text{ minute}) (1,000 \text{ L})} = 0.8335 \text{ m}^3$$

Therefore, the ideal hourly Q<sub>tot</sub> value will be rounded and recorded as 0.8335 m<sup>3</sup>. Q<sub>tot</sub> is an indication that the E-BAM flow was operating at the 16.67 L/min set point and stable throughout the sampling period. The Q<sub>tot</sub> value is an important data point for each hourly average mass value. The Q<sub>tot</sub> suggested criterion is 0.800–0.867 m<sup>3</sup>.

Checks will be made by using macros developed in Microsoft Excel to screen the data. Any data that fall outside defined constraints will be manually checked with the field logs and analyzed by an experienced air quality scientist familiar with validating onsite particulate monitoring data.

This screening process makes it possible to detect anomalous data that might not be detected during a cursory manual data review. Data that do not meet these criteria should be reviewed for reasonability. A decision must then be made whether to validate, flag, or invalidate the questionable data. Data will be determined to be invalid based on sound scientific judgment, the results of the screening process and review of the site field records of maintenance, calibrations, observations, and audit results. Revisions will be documented and approved by the task manager. The site operator will be notified of any systemic errors that can be alleviated or corrected in the field.

CH2M HILL may choose to modify screening criteria during the monitoring project. Changes will be based on the professional judgment of the scientist reviewing the data. Data screening changes will be documented in the quarterly data report and included in any revisions to the project QA Plan.

## **9.3 Data Reporting**

After the data have been reviewed, data reports will be prepared. These reports will summarize the data gathered and discuss the data review process and QA activities. The quarterly data reports will be submitted to the Navy.

### **9.3.1 Format of Data Submission**

Final data reports will include hourly averages for the meteorological parameters and PM<sub>10</sub> data, and 24-hour averages of PM<sub>10</sub> data for the quarter. The data completeness (i.e., the percent of valid data for each parameter) will be summarized monthly and quarterly. In addition, any QA activities, including the results of performance audits and the yearly calibration certifications will be presented.

### **9.3.2 Frequency of Data Reporting**

The quantitative data will be processed and reported quarterly within 60 days of the end of each quarter. PM<sub>10</sub> data is available on the Web site on a near-real-time basis. Qualitative analysis data will be available on a fast-track basis and will be provided within 1 week of availability from the laboratory.

## **9.4 Data Storage and Security**

All data collected for the VNTR monitoring project will be stored on the data manager's computer in the CH2M HILL office and backed up to the office server.

## 10.0 References

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40 CFR Part 50 Appendix J, "Reference Method for the Determination of Particulate Matter as PM<sub>10</sub> in the Atmosphere."

40 CFR Part 58 Appendix E, "Probe and Monitoring Path Siting Criteria for Ambient Air Quality Monitoring."

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Department of the Navy. December 1979. *Draft Environmental Impact Statement: Continued Use of the Atlantic Fleet Weapons Training Facility (Vieques)*.

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*Guideline on Air Quality Models* (EPA-450/2-78-027R).

*Meteorological Monitoring Guidance for Regulatory Modeling Applications*. February 2000. (EPA-454/R-99-005).

*Quality Assurance Handbook for Air Pollution Measurement Systems: Volume II, Ambient Air Specific Methods, EPA/600/R-94/038b, Volume IV, Meteorological Measurements*. March 1995. (EPA-600/R-94/038d).

*Standard Operating Procedures for Met-One Instruments Beta Attenuation Mass Monitor (BAM-1020)*. May 2003. AQSOP 400. Air Quality Surveillance Branch, California Air Resources Board.

APPENDIX A

**Table 1 of Ambient Monitoring Guidelines for  
Prevention of Significant Deterioration  
(EPA-450/4-87-007, May 1987)**

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**Table 1. Outline of Contents of a Monitoring Plan**

- I. Source Environment Description (within 2 km of source)
  - Topographical description
  - Land-use description
  - Topographical map of source and environs (including location of existing stationary sources, roadways, and monitoring sites)
  - Climatological description
  - Quarterly wind roses (from meteorological data collected at the source or other representative meteorological data)
- II. Sampling Program Description
  - Time period for which the pollutant(s) will be measured
  - Rationale for location of monitors (include modeling results and analysis of existing sources in the area)
  - Rationale for joint utilization of monitoring network by other PSD sources
- III. Monitor Site Description
  - Universal Transverse Mercator (UTM) coordinates
  - Height of sampler (air intake) above ground
  - Distance from obstruction and heights of obstructions
  - Distance from other sources (stationary and mobile)
  - Photographs of each site (five photos: one in each cardinal direction looking out from each existing sampler or where a future sampler will be located. Ground cover should be included in the close-up photograph.)
- IV. Monitor Description
  - Name of manufacturer
  - Description of calibration system to be used
  - Type of flow control and flow recorder
- V. Data Reporting
  - Format of data submission
  - Frequency of data reporting
- VI. Quality Assurance Program
  - Calibration frequency
  - Independent audit program
  - Internal quality control procedures
  - Data precision and accuracy calculation procedures

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Source: *Ambient Monitoring Guidelines for Prevention of Significant Deterioration, EPA-450/4-87-007, May 1987.*

APPENDIX B

# **E-BAM Instrument Manual**

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# **E-BAM OPERATION MANUAL**



**Met One Instruments, Inc**  
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Grants Pass, Oregon 97526  
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## **Technical Support**

Should you require support, please consult your printed documentation to resolve your problem. If you are still experiencing difficulty, you may contact a Technical Service representative during normal business hours—7:30 a.m. to 4:00 p.m. Pacific Standard Time, Monday through Friday.

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Met One Instruments, Inc.  
1600 Washington Boulevard  
Grants Pass, OR 97526

## **Safety Notice**

The contents of this manual have been checked against the hardware and software described herein. Since deviations cannot be prevented entirely, we cannot guarantee full agreement. However, the data in this manual is reviewed regularly and any necessary corrections included in subsequent editions.

Faultless and safe operation of the product presupposes proper transportation, storage, and installation as well as careful operation and maintenance. The seller of this equipment cannot foresee all possible modes of operation in which the user may attempt to utilize this instrumentation. The user assumes all liability associated with the use of this instrumentation. The seller further disclaims any responsibility for consequential damages.

The Met One Instruments E-BAM contains a small C<sup>14</sup> (Carbon 14) beta radiation emitting source. The activity of the source is 60 microcuries, which is below the “Exempt Quantity”, as defined in 10 CFR, Section 30.70 – Schedule B. The customer/owner of the model E-BAM may select to return the device to Met One Instruments for recycling of the C<sup>14</sup> source when the device has reached the end of its usable life, although is under no obligation to do so.

Neither the C<sup>14</sup> source, nor the detector, are serviceable in the field. Should these components require servicing and/or replacement, The E-BAM must be returned to the factory for servicing and/or re-calibration.

## **Safety Conformity**

The E-BAM is registered with the NRC under registration certificate number NR-1124-D-102-E.

## **Warranty**

Products manufactured by Met One Instruments, Inc. are warranted against defects in materials and workmanship for a period of (1) year from the date of shipment from the factory. Offered products not manufactured by Met One Instruments, Inc. will be warranted to the extent and in the manner warranted by the manufacturer of that product.

Any product found to be defective during the warranty period will, at the expense of Met One Instruments, Inc. be replaced or repaired and return freight prepaid. In no case shall the liability of Met One Instruments, Inc. exceed the purchase price of the product.

This warranty may not apply to products that have been subject to misuse, negligence, accident, acts of nature or that have or modified other than by Met One Instruments, Inc. Consumable items such as bearings are not covered under this warranty.

Other than the warranty set forth herein, there shall be no other warranties, weather expressed, implied or statutory, including warranties of fitness or merchantability.



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# 1 Safety

## 1.1 Safety Indicators

This manual uses a **CAUTION** and a **WARNING** indication. Familiarize yourself with the following definitions for the meanings of these indicators.

A **CAUTION** indicates a hazard and calls attention to a procedure that if not correctly followed could result in damage to the instrument. Do not proceed beyond a caution indicator without understanding the hazard.

A **WARNING** indicates a hazard to you and calls attention to a procedure that if not correctly followed could result in injury or even death. Do not proceed beyond a warning without understanding the hazard.

## 2 Introduction

### 2.1 Unpacking

When you unpack the E-BAM and accessories, inspect the carton for obvious damage. If the carton is damaged notify the carrier. Unpack everything and make a visual inspection. The E-BAM is shipped with the following accessories:

E-BAM Console	PM10 head
Ambient temperature probe	Cross Arm Support
Inlet adaptor tube	TUS Software
Tripod	Power Cable
Communication cable	Zero Calibration Plate
Filter tape, 1 roll	Span Calibration Plate
E-BAM operation manual	E-BAM calibration data sheet

If purchased, the following options may also be included:

- PM 2.5 sharp cut cyclone
- TSP sampling head
- BX-305 flow test inlet valve
- Battery 12VDC
- Battery charger
- AC power module

If any of the above components are missing contact the Met One service department at [service@metone.com](mailto:service@metone.com) or 541.471.7111. Keep the carton and packing material for reuse.

## **2.2 E-BAM Overview**

The E-BAM is a portable air monitor. It is based upon the principle of beta attenuation. Beta attenuation is a proven technology that has been used for particulate monitoring for over 40 years.

### **2.2.1 Beta Attenuation**

Beta attenuation is defined as the decrease in the number of beta particles due to absorption by the traversed medium. The E-BAM uses  $^{14}\text{C}$  Carbon a naturally occurring radioactive isotope as the source for beta particles.  $^{14}\text{C}$  Carbon beta particles are electrons emitted from the nucleus of an atom when a neutron decays to a proton and an electron. This electron is a subatomic particle having a mass of 0.00054858 amu and an average energy of 49 KeV. Due to the low mass and low energy, beta particles can only travel a foot or two through the air and can be completely attenuated with a few sheets of notebook paper. It is this property of attenuation of beta particles that allows the measurement of mass.

In the E-BAM a three-step process measures the mass of the suspended particulate. Step one is to make a first count across a piece of filter paper. Secondly, particle-laden air is passed through the paper and the particulate is deposited on the paper. Finally, step three is to make a second count across the filter paper with the deposited particulate. This second count will be less than the first count due to the absorption of beta particles by the deposited particulate. Some simple calculations are made and the mass of the deposited particulate is measured. The mass is divided by the amount of air sampled to give the concentration in mass per unit volume. For a complete discussion of beta attenuation in the E-BAM see section 8.1.

## **2.3 Getting Started**

### **2.3.1 Selecting the Sampling Site**

Assemble the E-BAM in the area where the aerosol is to be sampled. The most desirable height for TSP and PM10 monitoring is near the breathing zone. However, practical considerations such as prevention of vandalism, security, accessibility, availability of electricity, etc., generally require the sampler to be elevated. Therefore, a range of acceptable heights will need to be used. In addition, the type of source, i.e., elevated or ground level, predominantly influencing the area of impact must be considered when locating the monitor. For purposes of determining elevated source impact, the sample air intake must be located 2-15 meters above ground level. For ground level sources with steep vertical concentration gradients, the air intake must be as close to the breathing zone as practical.

### 2.3.1.1 Spacing from Obstructions

If the sample is located on a roof or other structure then there must be a minimum of 2 meters separation from walls, parapets, penthouses, etc. Trees provide surfaces for particulate deposition and also restrict airflow. Therefore the sampler should be placed at least 20 meters from the drip line of trees.

Obstacles such as buildings must be also avoided so that the distance between obstacles and the sampler is at least twice the height that the obstacle protrudes above the sampler. In addition, there must be unrestricted airflow in an arc of at least 270 degrees around the sampler, and the predominant direction for the season of greatest pollutant concentration potential must be included in the 270 degree arc.

### 2.3.1.2 Spacing from Roads

Ambient monitors for TSP and PM-10 should be located beyond the concentrated particulate plume generated by traffic, and not so close that the heavier re-entrained roadway particles totally dominate the measured ambient concentration. Roads with lower traffic (less than approximately 3,000 vehicles per day) are generally not considered to be a major source of vehicular related pollutants. In this case the monitor must be located greater than 5 meters from the edge of the nearest traffic lane and 2 to 15 meters above ground level. In the case of elevated roadways where the monitor must be placed below the level of the roadway, the monitor should be located no closer than approximately 25 meters from the edge of the nearest traffic lane. This separation distance applies for those situations where the road is elevated greater than 5 meters above the ground level, and applies to all traffic volumes.

**Note: Whenever the E-BAM is to be installed at a height greater than 3 meters it is recommended that it be securely bolted in place. If the E-BAM is accidentally dropped from a height greater than 3 meters, there is a chance that containment of the radioactive source may be compromised and it is recommended that the E-BAM be sent back to the factory for radiation leak testing.**

### 2.3.1.3 Other Considerations

Stations should not be located in an unpaved area unless there is vegetative ground cover year around so that the impact of re-entrained or fugitive dusts will be kept to a minimum.

### 2.3.1.4 Additional Information

More detailed information instrument siting can be found in EPA Document EPA-450/4-87-007 May 1987 "Ambient Monitoring Guidelines for Prevention

of Significant Deterioration (PSD)”. Information specific to PM10 monitoring can also be found in EPA Document 40 CFR-58 Appendix E.

### 2.3.2 Assembly

The E-BAM can be taken out of the box, assembled and collecting data in less than 30 minutes. There are six basic steps to assembly.

1. Lift the tripod and remove the three lock pins. Spread the tripod legs and re-insert the three lock pins. If the E-BAM is to be used under conditions that require additional stability, the tripod may be bolted to a firm surface using the ¼” holes in the tripod feet.
2. Lift up the E-BAM enclosure with the aerosol inlet oriented upwards. Slide the slot on the back of the cabinet down over the tab on top of the tripod. A ¼ inch nut and bolt is provided to attach the bottom of the cabinet to the tripod.
3. Remove the plastic end caps from the inlet adaptor tube and push it into the E-BAM inlet. It must go through two O-rings so push and twist it in all the way in until it stops. Hand tighten the large black lock screw located at the top of the E-BAM enclosure.
4. Pick up the PM10 sampling inlet and push it onto the inlet adaptor tube. It must go through two O-rings so push and twist it in all the way in until it stops. Note: the O-rings are factory lubricated but with frequent removal/replacement they will need to be re-lubricated with silicone O-ring grease.
5. Install the cross arm on the pipe at the top of the tripod and tighten the two Allen screws. Clip the temperature sensor onto one arm of the tripod and plug the signal cable into the 5-pin plug under the cabinet. Attach any accessory sensors to the other arm of the cross arm.
6. The standard E-BAM is supplied without a power source. Met One Instruments supplies various power sources as accessories. Any 12 Volt DC power source, such as a car battery, can be used to power the E-BAM. The power source should provide at least 4 Amps continuous power. Connect the 12 Volt power source to one end of the E-BAM power cable. Plug the other end of the power cable into the receptacle at the bottom of the enclosure.

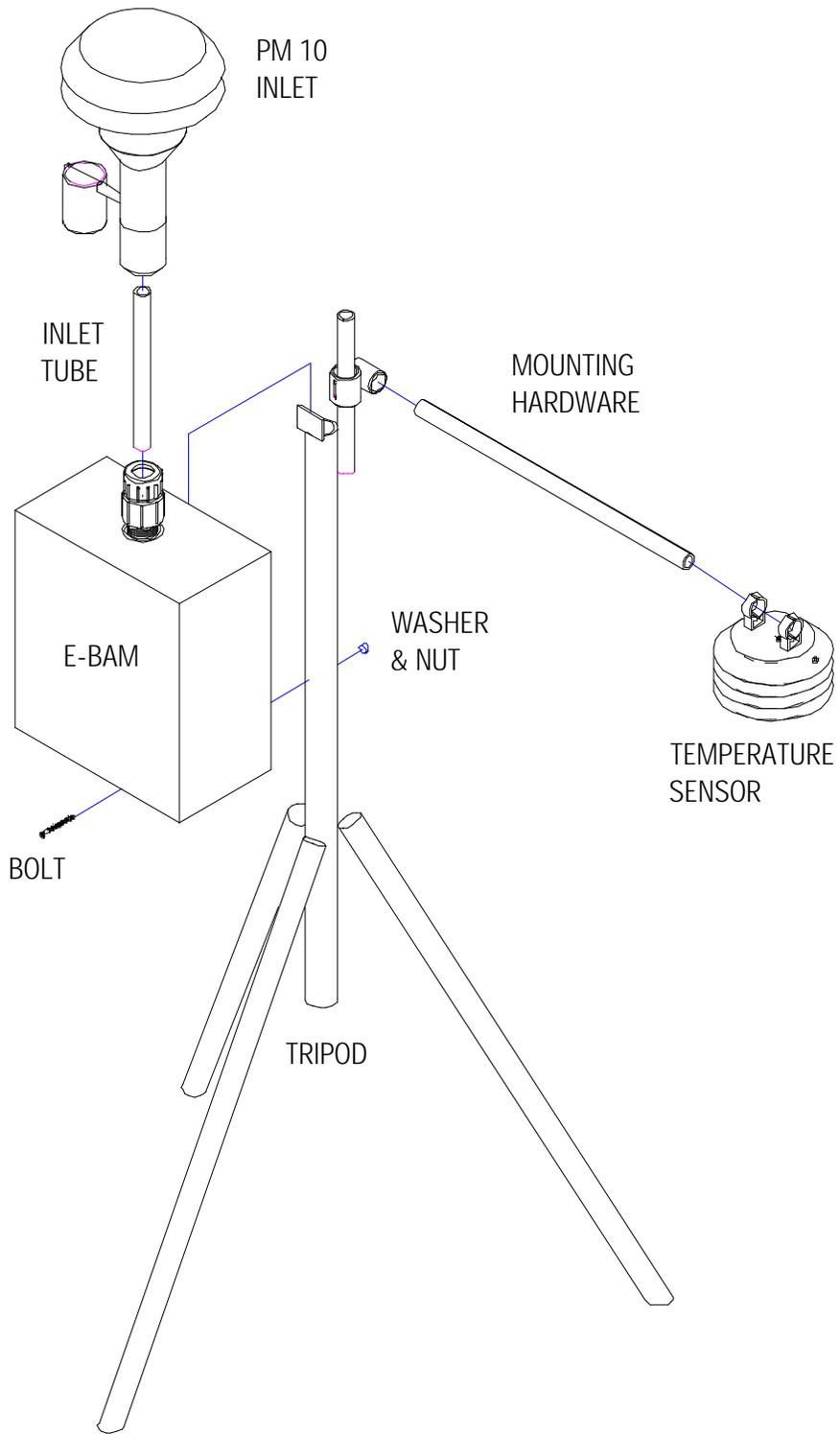


Figure 1 E-BAM setup assembly.

### 2.3.3 Power

The E-BAM is a battery-operated instrument. The power requirements are 11-16 VDC at 3 amps continuous. It can also be operated off an AC power source using an AC to DC converter. Met One can supply an AC power supply for the E-BAM in a weatherproof enclosure. For a complete listing of available options for the E-BAM see section 3.3.6. The AC to DC converter must be capable of producing 10 Amps at 12VDC due to the start up current required from the E-BAM internal pump. If the incoming voltage drops below the cutoff voltage (see section 4.5) the E-BAM will stop operation and log an Alarm see section 4.10. Once proper voltage is restored the E-BAM will continue to sample as previously programmed. The E-BAM is supplied with an External Power Cable see Figure 2.

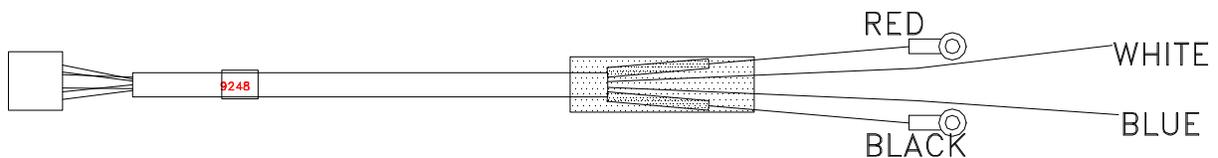


Figure 2 Power cable for the E-BAM.

The Power Cable has a six-wire connector on one end and four terminated wires at the other end. The red wire with a terminal lug is the positive power wire. The black wire with a terminal lug is the negative power wire. Apply 11-15 volts DC to these wires to power up the E-BAM. The other two wires (white and blue) are the contact closure for the E-BAM. The connector end plugs into the bottom of the E-BAM enclosure see Figure 3.

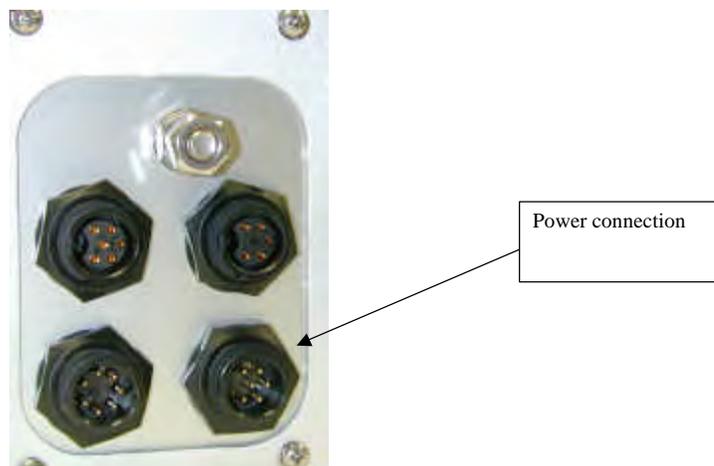


Figure 3 E-BAM Power Connection.

When power is applied to the E-BAM it will begin to operate after certain setup instructions are answered. If the setup instructions are not answered the E-BAM will use default answers and begin operation automatically after 30 minutes.

**2.4 Power Up Operation**

When the E-BAM is powered up it quickly runs through a minimum of eight screens. These screens verify that the time, date, and averaging period, are correct. They also check for filter tape and proper operation of vital subsystems during the E-BAM self test.

**2.4.1 Start Up Screens**

After it is assembled and the power is connected, the E-BAM is ready to be operated for the first time. Undo the latch and swing open the E-BAM cover. Note that the display is on and displaying ARE YOU READY TO START?

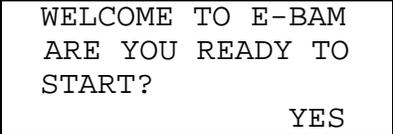


Figure 4 E-BAM Ready Start up screen.

Press the white “hot” key under YES to proceed. The pre-set time and date are displayed.

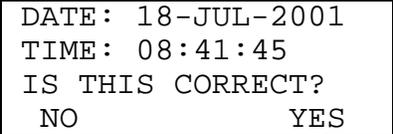


Figure 5 E-BAM Date Start up screen

If the time and date are correct, Push the right “soft” key on the keypad directly under where YES is displayed. Press NO to make a change. Use the arrow keys to make changes.

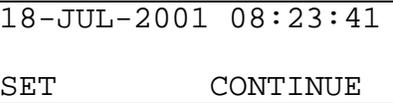


Figure 6 Modify Date/Time screen

When finished, press SET to save the changes. Press CONTINUE to exit without making changes.

LOCATION:	01
TAPE ADVANCE:	24 HRS
REALTIME AVG:	1 MIN
EDIT	OK

Figure 7 E-BAM Averaging and Location Start up screen.

To make changes press EDIT. Press OK to proceed to the next screen. Press EDIT to make changes.

Use the arrow keys to make changes.

LOCATION is a description for the E-BAM. Data that is downloaded will have a location ID number to enable tracking of measurement information. This may be a value from 0-99.

FILTER ADVANCE is how often the E-BAM will advance to a new spot of filter paper. The filter tape advances according to two criteria. First, according to the FILTER ADVANCE time set from this screen. This value can be any of the following times: 1, 2, 3, 4, 6, 8, 12, or 24 hours. Note: the filter advance does not change how often the concentration is calculated. The Real-Time concentration is always updated every minute and the Hourly concentration is always updated each hour.

Secondly, the filter will also automatically advance if the concentration is too high or the ambient temperature exceeds safe operation. When the filter tape is advanced due to high concentrations an event is written to the alarm log. When the filter tape is advanced due to ambient temperature an event is written to the alarm log (see section 4.10 for details of the event log).

REAL-TIME AVE is the averaging period for the Real-Time concentration value. The Real-Time concentration is calculated every minute from two four-minute counts (for more details on Real-Time concentration see section 3.2.1.2). The REAL-TIME AVE takes the mean value for the selected time period for the Real-Time concentration. The REAL-TIME AVE may be set to the following time periods: 1, 5, 10, 15, 30, or 60 minutes.

Press SAVE to save the changes. Press CONTINUE to proceed and not make changes.

If the nozzle packing material has not been removed, the nozzle will move upward and the display will ask you to remove nozzle-packing material.

PLEASE REMOVE NOZZLE PACKING MATERIAL. CONTINUE
--

Figure 8 E-BAM Nozzle Material Start up screen.

Remove the stainless steel nozzle packing material from under the nozzle and press CONTINUE. Note: this nozzle-packing slide is also the Zero adjustment membrane.

The next screen will be shown while the unit checks to see if the filter paper is loaded.

```
CHECKING FOR
LOADED TAPE.

PLEASE WAIT...
```

Figure 9 E-BAM Check Filter Tape Start up screen.

If the unit finds that the filter tape is not loaded, the following screen will ask you to load the filter tape.

```
PLEASE LOAD TAPE!
E-BAM WILL NOT
OPERATE WITHOUT TAPE.
CONTINUE
```

Figure 10 No Filter Tape loaded Screen.

Remove both of the clear plastic spool covers by unscrewing the captive knobs. An empty tape roll core tube must be installed on the hub of the take-up spool. Special care should be taken when handling unused filter tape because it tears easily. Place full roll of filter tape on supply (right) spool, with the tape feeding upward and counter-clockwise as shown below. Feed end of filter tape so that it enters the take-up spool in a counter-clockwise direction. Using any available adhesive tape, attach the leading end of the filter tape to the core tube to prevent slipping. Gently tension the tape. Re-install both spools. When finished answer CONTINUE.

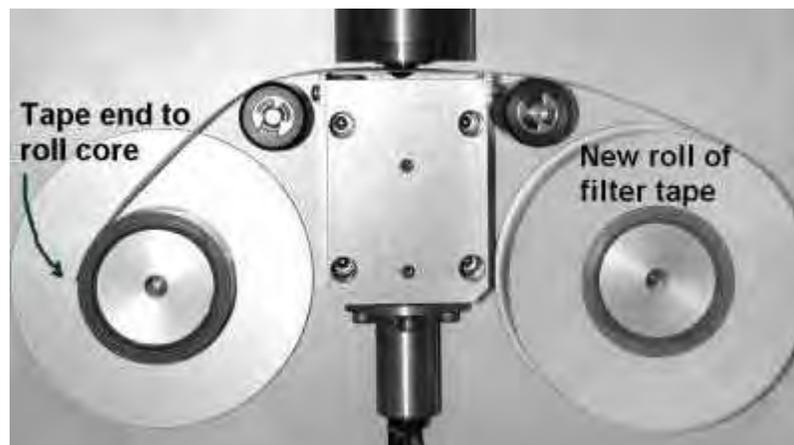


Figure 11 Filter Tape installation.

The filter tape will move and take up tension.

```
CHECKING FOR
LOADED TAPE.

PLEASE WAIT...
```

Figure 9 E-BAM Check Filter Tape Start up screen.

After the tape is checked, the BATTERY condition is displayed.

```
BATTERY: 13.0 VOLTS
ESTIMATED OPERATION
TIME FOR 100 AMP-HRS
IS 42 HRS. CONTINUE
```

Figure 12 E-BAM Power Start up screen

Press CONTINUE to proceed.

The E-BAM display will indicate SELF TEST RUNNING.

```
SELF TEST RUNNING...

*****999
```

Figure 13 E-BAM Self Test Start up screen

The SELF TEST will take several minutes and cannot be bypassed by the operator.

After SELF TEST is complete the following screen is displayed.

```
SELF TEST COMPLETE:
E-BAM FUNCTIONING
PROPERLY.
CONTINUE
```

Figure 14 E-BAM Self Test Complete Start up screen

Press CONTINUE and the E-BAM will begin sampling.

The Self Test can fail during any of the following sub-test:

- Test**
- Tape Broken
- Nozzle Motor Failed
- Beta Counts Failed
- Flow System Failed
- Pressure Sensor Failed

Figure 15 E-BAM Self Test failure screens.

If any fault is located during self-test, the name and type of fault will be shown. If there is a reasonable expectation that the operator could correct the fault, then an

instruction for correction will be shown on the E-BAM display with step-by-step instructions to test and verify the system integrity. The operator must acknowledge any errors within one minute or the unit will automatically begin operation. When the E- BAM is first powered on it will require a one-hour warm up period. Data acquired during the first hour should be discarded.

## 2.5 Using the Menu System

After the Start up screens the E-BAM defaults to the OPERATE screen. Concentration, Date/time and sampling condition are displayed. Pressing the DOWN arrow displays current internal and external sensor readings. Pressing the LEFT arrow key will scroll back through historical logged data. The UP/DOWN/LEFT/RIGHT keys are used to navigate a matrix of logged data. This matrix has 3 rows and up to 12000 columns of historical logged data. Press the RIGHT arrow and UP arrow to return to the current concentration display screen. Note: the RIGHT arrow key must be pressed the same number or more times as the LEFT arrow key was pressed. Also with the UP/DOWN keys. You may press ESC at anytime to immediately return to the current concentration screen from any historical data screen. The MENU key may be pressed at anytime in any screen to enter the main menu screen. This screen is the top of a tree style menu system. Use the cursor to highlight an entry and press MENU again to select that entry. Pressing ESC will back up along the tree to the top.

## 2.6 Basic Setup

The E-BAM is factory configured to measure PM10. Once power is applied to the unit it will automatically begin to sample until the power supply drops below 11 VDC. The following table lists the factory configuration for the E-BAM. After each entry is the manual section detailing how to modify the factory setting.

<u>Function</u>	<u>Setting</u>	<u>Manual Section</u>
Filter Advance	24 Hour	4.5
Real Time average	10 minutes	4.5
Time	Pacific Standard Time	4.5
Analog voltage	Hourly Concentration	4.5
Analog range	0-1 Volt	4.5
Heater RH setpoint	45%	3.2.2
Delta-T Setpoint	10 degrees C	3.2.2
Communication rate	9600 Baud	4.5
Location ID	01	4.5
Cut point	PM10	3.1.2.1

Figure 16 E-BAM factory default settings.

## **3 Operation of the E-BAM**

### **Introduction**

The Met One Instruments E-BAM is a portable and continuous reporting beta attenuation monitor (BAM). A measurement of the mass concentration of dust particles is made using the principle of beta attenuation. A small  $^{14}\text{C}$  (source) emits a constant 60 microcurie of low to medium energy electrons (156 KeV maximum with 49 KeV average energy) known as beta particles. These beta particles are efficiently detected by an ultra sensitive scintillation detector and counter positioned near the source. A vacuum pump pulls a measured amount of air through the filter tape. Filter tape that has been impregnated with dust particles is placed between the source and detector thereby causing attenuation of the measured beta signal. The degree of attenuation of the beta signal is used to determine the mass concentration of particulate matter on the filter tape. This measured mass is divided by the sampled volume of air to calculate the concentration of particulate matter in the sampled air.

### **3.1 Description of Operation**

#### **3.1.1 E-BAM Overview**

The E-BAM is an automated filter based TSP, PM10 and PM2.5 monitor. It has the same basic operation as manual filter based samplers such as the SSI (Size Selective Inlet High Volume Sampler) or the FRM (Federal Reference Method). These manual samplers consist of three sub-systems, which are the following: a Flow system, a Measuring system and a Data system.

Typically the flow system consists of an Inlet separator, filter holder, flow meter, flow controller and a pump (Figure 2).

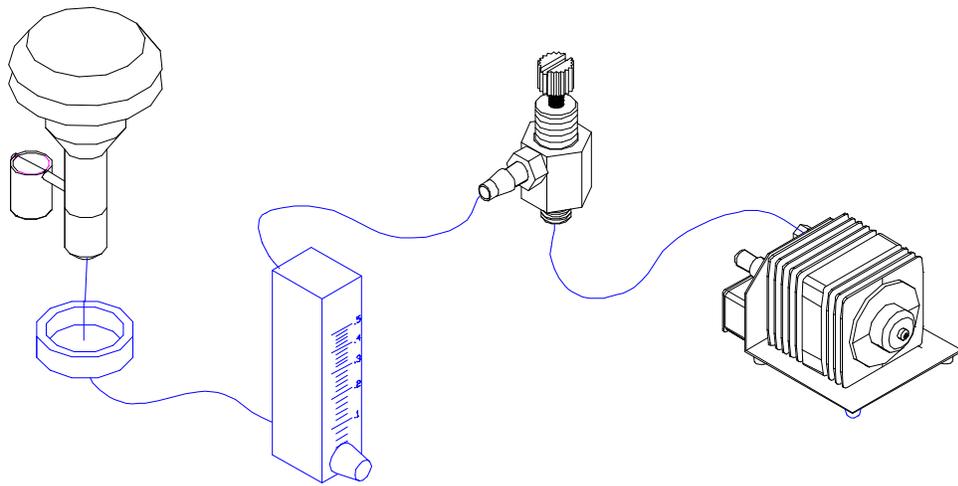


Figure 17 Typical flow system for a Manual Filter based Sampler

The measuring system requires a pre-weighed filter equilibrated at 20 degrees Celsius and 45% RH. This filter is installed in the sampler and a known volume of ambient air is drawn through the filter. The filter is then sent back to the lab to be equilibrated and weighed again (Figure 3). The concentration ( $\mu\text{g}/\text{m}^3$ ) is calculated by the change in mass of the filter divided by the volume of air drawn through the filter.

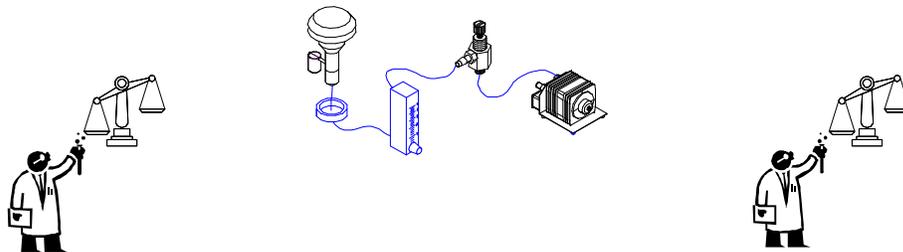


Figure 18 Pre-weighing, Sampling and post-weighing in manual samplers.

Finally, the data system tracks items such as the flow rate, filter and ambient temperature, barometric pressure, Wind speed and direction etc. These parameters are recorded with criteria to alert the user of invalid data.

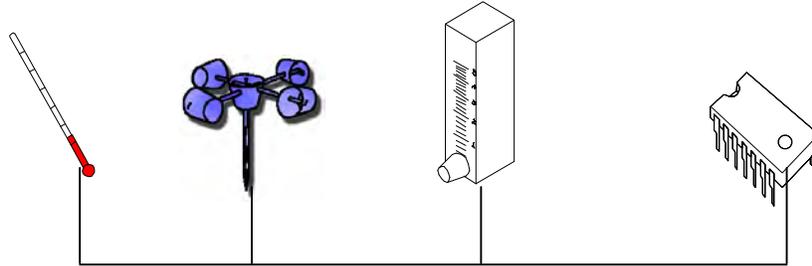


Figure 19 Data collection system

The E-BAM also has a flow system, measuring system and a data system. The advantage of the E-BAM is in the automation of the measuring system and the simplification of both the data and flow systems.

### 3.1.2 Flow System

The flow system of the E-BAM consists of the same components as the Manual sampler – Inlet, Filter holding mechanism, flow meter, flow controller and pump.

### 3.1.2.1 Inlets

Three different inlets are available with the E-BAM. Each E-BAM is equipped with the EPA designed PM10 inlet head (BX-802). The cut-point for the PM10 head is shown in Figure 20.

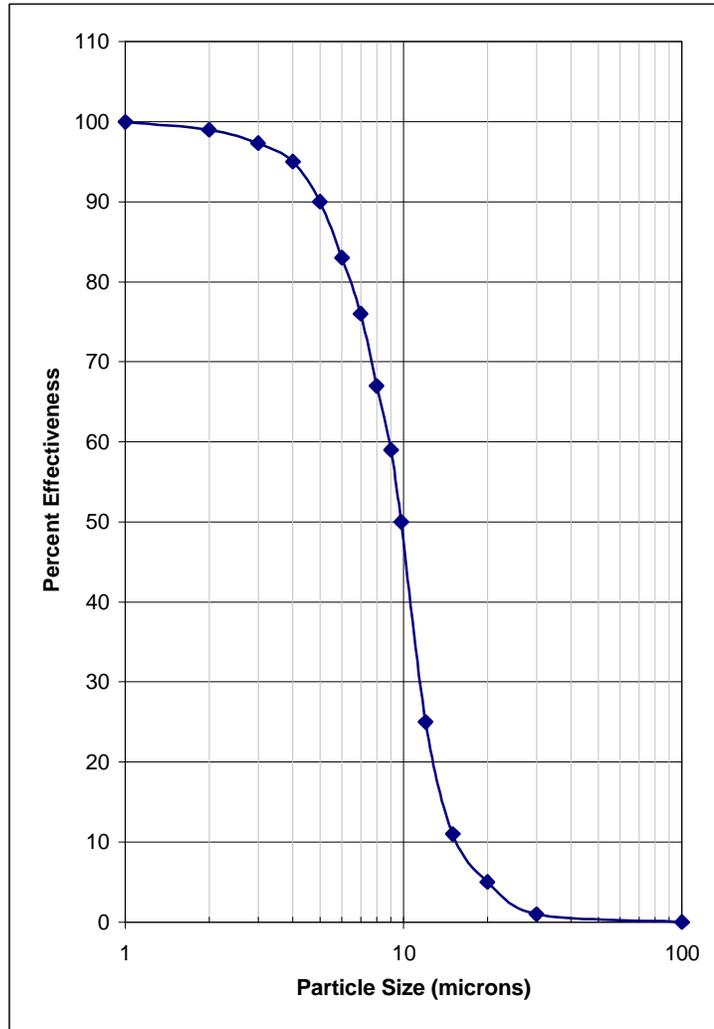


Figure 20 Cut point curve for the EPA Designed PM10 head.

The E-BAM can also be configured to monitor PM<sub>2.5</sub> with the addition of a BX-807, which is a Sharp Cut Cyclone (SCC). The SCC fits inline under the PM10 head and removes particles that are larger than 2.5 microns. The final inlet configuration for the E-BAM is for TSP (Total Suspended Particulate). The TSP head is used in place of the PM10 head and allows all suspended particulate to be monitored.

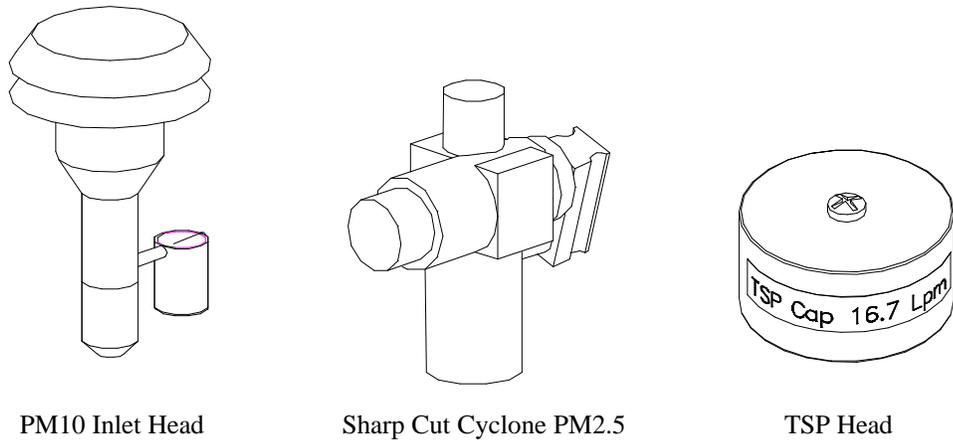


Figure 21 shows the three inlets for the E-BAM.

### 3.1.2.2 Filter Holder Mechanism

In the E-BAM a continuous roll of filter paper (22 meters in length and 30 mm in width) provides the collection medium for suspended particulate. This roll of filter tape is advanced when the concentration reaches a certain limit or when the filter spot has been sampled on for 24 hours. Figure 22 shows a sampled filter paper. The color and shade of the filter spot indicates concentration levels and chemical components. Note: on the E-BAM the spacing of the filter spots will vary depending on the amount of filter tape on the take up spool.



Figure 22 A sampled piece of filter paper.

The E-BAM nozzle assembly makes an airtight seal at the filter paper. The nozzle is lowered down on the filter paper and compresses a small area to form a seal. When the filter paper is advanced to the next spot the nozzle is lifted and lowered on the next spot. Figure 23 shows a detail of the nozzle assembly.

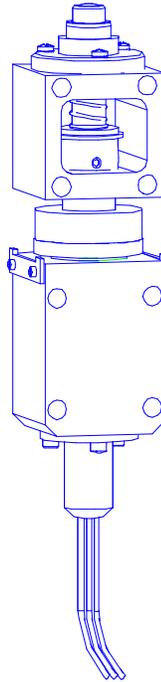


Figure 23 E-BAM Nozzle Assembly.

With routine maintenance the nozzle will provide automated sealing as the filter tape is advanced.

### 3.1.2.3 Flow Meter, Flow Controller and Pump

The flow rate in the E-BAM is important for one primary reason. The inlets are designed to make the necessary cut point at a designated flow rate. The inlets used by the SSI, FRM and E-BAM work on the principle of momentum. Momentum is the result of a particle's velocity multiplied by its mass. In the cut-point inlets particles follow a flow path that eliminates particles that have too much momentum. Particles that are smaller than the cut-point are able to follow the flow path and are measured as a component of concentration. The flow rate is important because it determines the velocity of the particle.

The E-BAM utilizes a closed loop controller that maintains the flow rate within 0.1 LPM of set point. Firstly, the flow rate is measured in the flow meter. The flow meter is a heated bridge delta Temperature mass flow meter. Two Temperature sensors (Upstream and Downstream) measure the temperature of the airflow. Between the temperature sensors is a heater. Air passes across the upstream temperature sensor then the heater and finally the downstream temperature. Due to conductive heating the air temperature increases as it passes across the heating element. The downstream temperature sensor measures this increase and mass flow is calculated. Next, the signal from the flow meter goes to the CPU along with ambient temperature and pressure.

Actual flow is calculated from the Ideal Gas Law (See Appendix C How to convert from Standard flow to Actual flow) and voltage to the DC pump is increased or decreased as required.

### 3.1.3 Measuring System

The greatest advantage of the E-BAM over traditional manual samplers is the automation of the measuring system. Instead of a manual laboratory analysis, the E-BAM uses a precise measuring technique called Beta Attenuation. For a detailed description of Beta Attenuation see Appendix 10.1 Theory of Operation.

In normal operation the E-BAM advances the filter tape to a clean spot. Once the nozzle has closed on the filter tape the E-BAM measures the mass (density) of the filter spot by taking a 4-minute count. This 4-minute count is simply how many beta particles that are emitted from the  $^{14}\text{C}$  source make it through the filter tape and are counted by the Photo Multiplier Tube (PMT). The more dense the filter material the fewer beta particles will be counted and visa versa. Figure 24 shows a detailed cut away of the Nozzle. Above the filter paper the Beta particles are 4 times as dense. Due to Beta attenuation across the filter paper the beta particle density (counts) decreases.

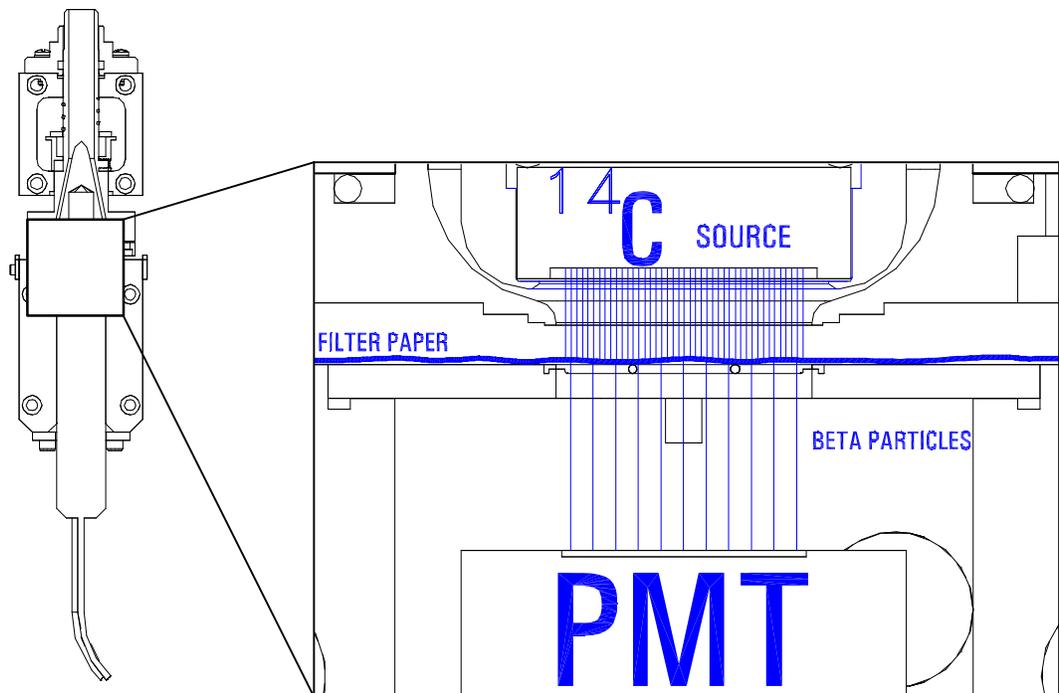


Figure 24 cut away nozzle detail of the Source, PMT and filter paper interface.

In the same manner as the manual filter based instruments this first 4-minute count is used as the clean filter paper mass. Air is sampled through the filter paper for the selected sample period. During this period particulate suspended in the air is deposited on the filter paper. Due to the unique design of the E-BAM where the source and detector are integral to the flow system the concentration can be calculated in real time.

Note: the first 10 minutes of a new sample spot will have a real time equilibration period. Real Time data subsequent to this will be stable. All hourly data is stable.

Beta attenuation is increased according to Lambert-Beers Law as the suspended particulate is deposited on the filter paper see Figure 25.

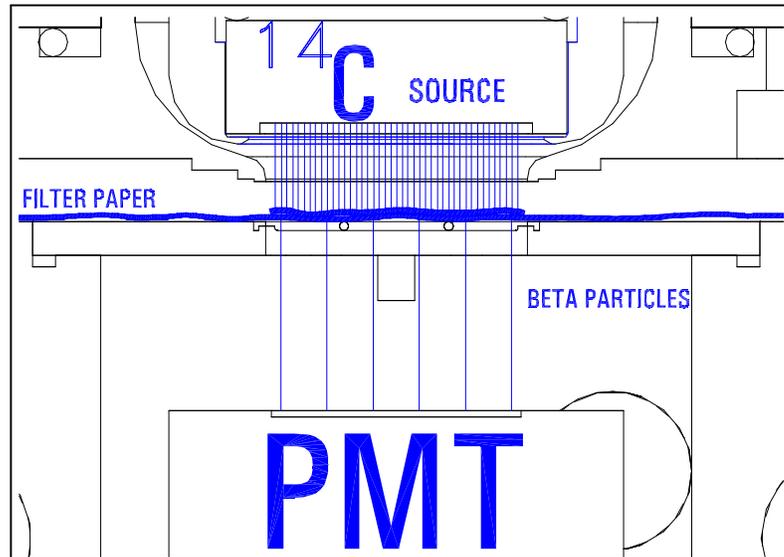


Figure 25 Attenuation due to deposits on the filter paper

Subsequent counts are made that correspond to a dirty filter paper condition. Comparing the clean original 4-minute count to the subsequent counts allows accurate calculation of concentration. The calculation is discussed below.

Lambert-Beers Law

$$I = I_0 e^{-m x}$$

Solve for  $x$

$$x = -\frac{1}{m} \ln\left(\frac{I}{I_0}\right)$$

$I$  – Beta counts across clean filter paper

$I_0$  – Beta counts across dirty filter paper

$m$  – Beta absorption coefficient

$x$  – mass density of deposition on filter

### 3.1.4 Data System

The final system of the E-BAM is the Data system. This system is what and how the E-BAM stores information and how to retrieve the stored information.

Standard to the E-BAM is a powerful 6-channel data logger. A complete met system can be connected to the E-BAM and used for data validation or air pollution studies. Every E-BAM comes standard with ambient temperature. This temperature value is used to calculate actual flow to maximize the accuracy of the cut point inlets. In addition to ambient temperature the E-BAM can be outfitted with the following meteorological sensors:

- Wind Speed
- Wind Direction
- Relative Humidity
- Barometric Pressure

Along with optional meteorological sensors the E-BAM stores all the important information relative to accurate ambient air concentration. Below is a list of the parameters and measurements recorded by the E-BAM:

- Hourly Concentration with data/time
- Real Time Concentration (1, 5, 15, 60 minute) with data/time
- Filter Temperature and RH
- Ambient Temperature
- Errors with data/time
- Flow Values
- Battery Voltage

Once the data are stored retrieval of the data can be done a number of ways. The E-BAM allows front screen viewing of the stored data.

```
08-JUL-2001 08:23:41
 9.999 MG/M3 01:00
 9.999 MG/M3 (HR)
SAMPLING...
```

```
FLOW: 99.9 LPM
WS: 999.9 M/S
WD: 999.9 DEG
AT: -99.9 C
RHx: 999 %
RHl: 999 %
FT: -99.9 C
```

Press the , , , and  keys to traverse the parameter list.

Pressing the Up/Down arrows moves the viewed window down or up the list. In the diagram above all that would be viewed is the two concentration values. Pressing the down arrow once moves the viewed window down and FLOW and WS would then be in the viewed window.

Pressing the Right/Left arrows moves the screen to historical data. Once the viewed window is in historical data the time in the top line of the viewed window will reflect the time of the historical record and SAMPLING will not appear in the window. Press ESC and the viewed window will go back to the current values.

The data can also be downloaded from the E-BAM by any number of methods. Each E-BAM is supplied with TUS software. This software automatically finds connection speeds, and hardware used to download data (i.e. RS232, GOES satellite, modems, and Cell phone/Radio modems). Met One can supply any configuration mentioned above.

The downloaded data are in a CSV file. This file type can be automatically opened with EXCEL or most spreadsheet programs. Met One offers a number of automatic programs that will download the data, graph, log error and signal alarms based on preset criteria. The software comes in two packages – MicroMet™ Plus or MicroMet™ AQ. Both simplify data collection, validation and viewing.

To download data install the software on a computer (TUS, MicroMet™ Plus or MicroMet™ AQ). Open the program and follow the instructions per the software package to configure and download the data. Make all the necessary connections and download. If the software is TUS open the CSV file with a spreadsheet or use the print function of TUS to view the data.

The E-BAM also has a voltage output for concentration. All E-BAMS are supplied with a communication cable. A portion of the cable is used for Serial downloads while another portion provides the concentration voltage output. The communication cable terminates into a 9-pin serial connector. Out of the back of

the connector are two wires – a black and a white wire. The black wire is common while the white wire is the concentration voltage. Connect these wires to an external data logger to record concentration data on a central logger. The concentration output full-scale voltage can be set to 1, 2.5, or 5 volts in the SETUP screen from on the main menu. The concentration range for any full-scale voltage range is 0 to 1 mg/m<sup>3</sup>. If the concentration exceeds 1 mg/m<sup>3</sup> the data is saved in the E-BAM internal data logger and may be downloaded supplement the excursion period. The E-BAM has a concentration range from 0-65 mg/m<sup>3</sup>.

### 3.2 Using the E-BAM

The E-BAM is designed to make environmental monitoring simple and effective. Applying power to the unit starts the E-BAM using the last programmed protocol. Before starting, the E-BAM initiates a Self Test (Section 2.4.1) to ensure accurate data. When all the internal self test are completed the unit will begin sampling unless a critical failure occurred and the E-BAM cannot function. The E-BAM will continue to sample until user intervention or power drops below the minimum voltage (see section 4.5).

#### 3.2.1 Normal Operation

Each E-BAM is factory configured to monitor PM10 concentrations. For a complete list of factory settings see section 2.6. Depending on the type of sampling these settings may need to be modified.

After initial installation or relocation of the E-BAM a Leak Check and Flow Audit procedure must be done. See section 3.3.1.1 and section 3.3.1.2 for Leak Check and Flow Audit procedures.

The default screen for the E-BAM is the Sampling screen see Figure 26.

```

08-JUL-2001 08:23:41
 9.999 MG/M3 01:00
 9.999 MG/M3 (HR)
SAMPLING...
FLOW: 99.9 LPM
WS: 999.9 M/S
WD: 999.9 DEG
AT: -99.9 C
FT: -99.9 C
RHi: 999 %
RHx: 999 %
BV: 99.9 V
FLOW: 99.9 SLPM

```

Figure 26 E-BAM default sampling screen.

The E-BAM Sampling screen is 13 lines long but only four lines are visible due to the display size. Using the down arrow will move the window so the other lines can be viewed. The lines consists of the following information:

Line	Description	Section
1	Date and Time	3.2.1.1
2	Real-Time Concentration	3.2.1.2
3	Hourly Concentration	3.2.1.3
4	E-BAM Status	3.2.1.4
5	Sample Flow Rate	3.2.1.5
6	Wind Speed	3.2.1.6
7	Wind Direction	3.2.1.6
8	Ambient Temperature	3.2.1.7
9	Filter Temperature	3.2.1.8
10	Relative Humidity Internal	3.2.1.8
11	Relative Humidity External	3.2.1.9
12	Battery Voltage	3.2.1.10
13	Secondary Flow	3.2.1.11

If further information is needed for a particular line see the section immediately following the description of that line.

### 3.2.1.1 Date and Time

The Date and Time are displayed as the DAY-MONTH-YEAR. This is the default format and may not be modified. There are two methods to change the date/time.

The first is in the Start-Up screens. Each time the E-BAM is powered up it goes thru a series of eight or more set up screens. The second screen shows the Date and Time. In this screen (see page 16) is the option to change the setting for both the Date and the Time.

The second is to press the MENU key on the E-BAM keypad. Pressing this key will bring up the main MENU (see page 4.3) for the E-BAM. Press the down arrow to highlight the SETUP selection and press MENU/SELECT. The first SETUP screen is the DATE/TIME screen with two identical Date/Times showing on the first two lines of the display. Using the RIGHT/LEFT arrow keys highlight the value(s) that need to be changed. Now pressing the UP/DOWN arrow keys will increment the selection to a higher or lower value. Once the Date/Time are correct press SET and then continue until the main menu is displayed.

### 3.2.1.2 Real-Time concentration

Beta Attenuation for particulate monitoring measures mass by comparing an initial or first count for some time period to a second count of the same time period (for a complete discussion of Beta Attenuation see Section 8.1). These counts are normally separated by some time period. In the E-BAM the first and second counts are always four-minutes long. During normal sampling the four-

minute first count and the four minute second count are measured subsequent to one another. This means that there is eight minutes from the start of the first count until the end of the second count. These counts are both advanced every 60 seconds. The datalogged concentration can be this 1-minute updated value or it can be a mean average of a user selectable time period. This selectable time period is called the REAL-TIME AVE and it may be any of the following values: 1, 5, 10, 15, 30, or 60 minute(s). The REAL-TIME AVE setting can be modified in the Start-Up Screens (see page 17) or in the SETUP menu. The SETUP menu is located in the main E-BAM menu. Press MENU/SELECT and then highlight SETUP with the cursor and press MENU/SELECT again. Press CONTINUE to bypass the DATE/TIME screen. The next screen is the Location Averaging Period screen. Modify the settings by highlighting the value(s) and incrementing the reading with the UP/DOWN keys. The only time that the calculation is different is at the beginning of a sample period after the filter tape has advanced. For seven minutes at the start of the sample period the calculation is slightly modified. See Figure 27 for details on the Real-Time concentration.

### **3.2.1.3 Hourly Concentration**

Hourly concentration is computed from an initial count compared to a second count (for a complete discussion of Beta Attenuation see Section 8.1). These two counts are separated by 60 minutes. This means that there is 60 minutes from the start of the initial count until the end of the final count. The Hourly concentration is a fixed calculation that is not modifiable. At the beginning of an hour the last hour's calculation is written to the datalogger. This value is held constant until the start of the next hour when it is updated with the new calculation of concentration. See Figure 27.

Real-Time Ave Period		10 Minutes						
Filter Advance		2 Hours						
Date/Time		20-JAN-2003 01:00:00						
Time Minute	Real-Time 1st Count Minutes	Real-Time 2nd Count Minutes	Real-Time Concentration mg/m3	Real-Time Logged mg/m3	Hourly 1st count Minutes	Hourly 2nd Count Minutes	Hourly Concentration mg/m3	Hourly Logged mg/m3
1								
2								
3								
4	1-4				1-4			
5	1-4	2-5	0.025		1-4			
6	1-4	3-6	0.030		1-4			
7	1-4	4-7	0.027		1-4			
8	1-4	5-8	0.040		1-4			
9	2-5	6-9	0.034		1-4			
10	3-6	7-10	0.050	0.034	1-4			
11	4-7	8-11	0.048		1-4			
12	5-8	9-12	0.043		1-4			
13	6-9	10-13	0.036		1-4			
14	7-10	11-14	0.038		1-4			
15	8-11	12-15	0.035		1-4			
16	9-12	13-16	0.030		1-4			
17	10-13	14-17	0.028		1-4			
18	11-14	15-18	0.020		1-4			
19	12-15	16-19	0.013		1-4			
20	13-16	17-20	0.010	0.030	1-4			
40	33-36	37-40	0.032	0.025	1-4			
60	53-56	57-60	0.020	0.018	1-4	57-60	0.028	0.028
80	73-76	77-80	0.034	0.028	61-64			0.028
100	93-96	97-100	0.030	0.032	61-64			0.028
120	113-116	117-120	0.031	0.036	61-64	117-120	0.031	0.031
Filter Advance at 2 hours.								
20	13-16	17-20	0.021	0.015	1-4			

Figure 27 Detail of the Real-Time and Hourly concentration calculations.

### 3.2.1.4 E-BAM Status

The status line is an indicator of the current E-BAM operation. This is a list of the E-BAM status messages

SAMPLING

UNIT OFF

If the E-BAM is displaying UNIT OFF pressing the hot key directly under the message will turn the E-BAM on.

### 3.2.1.5 Sample Flow Rate

This is a display of the instantaneous flow rate. The E-BAM flow rate may be set to a value from 10 LPM to 17.5 LPM. The flow can be controlled to Actual or Standard conditions see Figure 28.

Flow Type	Description
ACTUAL	Flow (ACTUAL) is reported in ambient volumetric conditions (Temperature and Pressure). The concentration is reported in mg/m <sup>3</sup> (ACTUAL conditions). Flow is controlled to ambient volumetric conditions. Flow is designated on the display as 16.7 LPM
STANDARD	Flow (STD) is reported in EPA conditions (25 C and 760 mmHg). The concentration is reported in mg/m <sup>3</sup> (EPA conditions). Flow is controlled to ambient volumetric conditions with the ambient Temperature/Pressure Sensors. Flow is designated on the display as 16.7 SLPM

Figure 28 Details for the flow settings.

The Flow settings can be modified in the Start-Up Screens (see page 16) or in the SETUP menu. The SETUP menu is located in the main E-BAM menu. Press MENU/SELECT and then highlight SETUP with the cursor and press MENU/SELECT again. Press CONTINUE to bypass the DATE/TIME screen, and the LOCATION screen. The next screen is the FLOW RATE screen. Modify the settings by highlighting the value(s) and incrementing the reading with the UP/DOWN keys.

### 3.2.1.6 Wind Speed and Direction

This is an optional accessory for the E-BAM. The Wind sensor is a combination sensor Model 034B. This sensor measures both parameters. The displayed units are M/S (meters per second) and degrees. The sensor can be mounted directly to the E-BAM tripod or remotely on an existing structure. It plugs directly into the E-BAM without the need for programming. For a complete list of accessories for the E-BAM see section 3.3.6.

### 3.2.1.7 Ambient Temperature

Every E-BAM comes equipped with an ambient temperature sensor. This sensor allows the E-BAM to control the flow to Actual conditions and report the concentration in actual conditions see section 3.2.1.5 for more details. The Ambient temperature sensor mounts directly to the E-BAM tripod see Figure 1 E-BAM setup assembly. This sensor has a temperature range of -50 to 50 degrees Celsius and an accuracy of 0.1 degrees Celsius.

### **3.2.1.8 Relative Humidity Internal and Filter Temperature**

These are standard measurements of the E-BAM. Both sensors are located downstream of the filter paper. The relative humidity is used to control an inlet heater. The inlet heater is used to prevent condensation on the filter paper. If there is condensation on the filter paper and water begins to collect along with the airborne particulate the mass measurements will be high. By adding heat to the air stream in a controlled manner condensation is avoided and proper mass measurements are calculated. To ensure the sample does not get over heated filter temperature is also measured. The filter temperature and the ambient temperature are used to calculate a Delta-Temperature. The Delta-Temperature is the Filter Temperature minus the Ambient Temperature. A maximum Delta-Temperature can be set to limit the heat applied to the sample air. The set points for the moisture-controlled heater can be modified in the Start-Up Screens (see page 17) or in the SETUP menu. The SETUP menu is located in the main E-BAM menu. Press MENU/SELECT and then highlight SETUP with the cursor and press MENU/SELECT again. Press CONTINUE to bypass the DATE/TIME screen, the LOCATION screen and the FLOW screen. The next screen is the HEATER screen. Modify the settings by highlighting the value(s) and incrementing the reading with the UP/DOWN keys.

### **3.2.1.9 Relative Humidity External**

This is an optional accessory for the E-BAM. The RH external sensor is a Model EX-593. It has a measurement range from 0 – 100% with an accuracy of 3%. The EX-593 can be mounted directly to the E-BAM tripod or remotely to an existing structure. For a complete list of accessories for the E-BAM see section 3.3.6.

### **3.2.1.10 Battery Voltage**

This is a measurement of the incoming power. If the unit is connected to a battery this will allow the logging of the power during the sample period. If the unit is connected to AC power through a DC power supply this should be a constant number.

### **3.2.1.11 Secondary Flow**

This channel is used only when the FLOW type is selected as STANDARD flow. Standard Flow will be displayed on line 5. This channel will display ACTUAL flow. If ACTUAL flow is selected this line will be blank. For a complete discussion of the flow types see section 3.2.1.5.

## **3.2.2 Inlet Heater**

All E-BAMs are equipped with a moisture controlled inlet heater. The heater is controlled by filter RH and filter temperature. Both sensors are located downstream of the filter paper. The relative humidity is the main controller of the inlet heater. The inlet heater is used to prevent condensation on the filter paper. If

there is condensation on the filter paper and water begins to collect along with the airborne particulate the mass measurements will be high. By adding heat to the air stream in a controlled manner condensation is avoided and proper mass measurements are calculated. To ensure the sample does not get over heated filter temperature is also measured. The filter temperature and the ambient temperature are used to calculate a Delta-Temperature. The Delta-Temperature is the Filter Temperature minus the Ambient Temperature. A maximum Delta-Temperature can be set to limit the heat applied to the sample air. The set points for the moisture-controlled heater can be modified in the SETUP menu. The SETUP menu is located in the main E-BAM menu. Press MENU/SELECT and then highlight SETUP with the cursor and press MENU/SELECT again. Press CONTINUE to bypass the DATE/TIME screen, the LOCATION screen and the FLOW screen. The next screen is the HEATER screen see Figure 29.

RH SETPOINT: 45 %
DELTA-T SETPT: 10 C
RH CONTROL: ON
SAVE                      CONTINUE

Figure 29 HEATER control screen.

Modify the settings by highlighting the value(s) and incrementing the reading with the UP/DOWN keys. Met One recommends using an RH Setpoint of 45% and a Delta-T setpoint of 8 degrees Celsius with RH control set to ON.

The Inlet Heater Operates according to the following parameters:

- When RH control is ON, the heater will be turned ON when the RH is above setpoint and will turn OFF 1 % below setpoint.
- When the Delta-T setpoint is exceeded by 1 degree C the heater is turned OFF. The Delta-T control over rides the RH setpoint control.
  - Delta-T is the result of the filter temperature minus the ambient air temperature.
- Anytime the pump is OFF, the heater is OFF.
- Delta-T violations are reported to the Alarm log and sets the alarm relay.
  - A violation occurs when RH control is ON and the Delta-T reading exceeds the Delta-T setpoint by 1 degree C.
  - Reset the alarm at power up and beginning of tape advance.

### **3.3 Maintaining the E-BAM**

#### **3.3.1 Flow System**

The flow system should be audited periodically to insure that accurate data is being collected. Two checks are required to verify the operation of the E-BAM. The checks are a leak check and a flow audit.

##### **3.3.1.1 Leak Check**

During normal operation the flow system is under a vacuum of 160 mmHg (6 inHg). The leak check procedure increases this vacuum to 400 mmHg (16 inHg). This 2.5 times increase in vacuum finds problems before they can affect the data. To perform a valid leak test follow the step-by-step procedure below.

1. E-BAM is in the PUMP TEST screen located in the MAIN MENU/FIELD CALIBRATION/PUMP TEST. This screen has two modes – LEAK CHECK and PUMP TEST. Select LEAK CHECK.
2. Remove the PM10 inlet and replace with a Leak test valve (BX-305).
3. Close the valve on the leak test valve.
4. Flow rate should drop to under 1.5 LPM. If the flow is under 1.5 LPM remove the Leak test valve and replace the PM10 head. If the flow is greater than 1.5 LPM proceed to 3.3.1.3.

##### **3.3.1.2 Flow Audit/Calibration**

After a successful leak check the next procedure to validate a flow system is a Flow Audit. In an E-BAM the flow audit process has been simplified to allow quick and accurate flow Audits. A Flow Audit is simply connecting a flow standard to the E-BAM inlet and comparing readings. If the flow standard uses tubing a BX-305 will allow easy connection to the E-BAM inlet. Be sure that the flow standard and the E-BAM are measuring the flow on the same units and type. The E-BAM has two flow types that are Actual flow and Standard flow see Figure 28 for an explanation of these flow types. To check the setting of the flow type in the E-BAM go the SETUP screens in the MAIN MENU. If the flow audit shows that the E-BAM flow rate has an error greater than 2% a Flow Calibration will need to be done.

To perform a valid Flow Calibration follow the step-by-step procedure below.

#### **TEMPERATURE**

1. Go to the MENU SCREEN and use the up and down arrow keys to select FIELD CALIBRATION then press the SELECT key.

OPERATE LOAD TAPE SETUP MEMORY SELF TEST FIELD CALIBRATION SHUTDOWN/SHIPPING VIEW ALARM LOG ABOUT
---

2. The FIELD CALIBRATION screen has seven selections:

TEMPERATURE PRESSURE FLOW FILTER RH <sub>i</sub> FILTER TEMP PUMP TEST ANALOG AUDIT MEMBRANE TEST
--

3. The first three selections TEMPERATURE, PRESSURE, and FLOW are used in a flow audit. Actual Flow is calculated from all three components. For a detailed description of how each is used to calculate flow see Appendix C How to convert from Standard flow to Actual flow
4. Select TEMPERATURE from the FIELD CALIBRATION screen. Note: before a flow audit temperature and pressure must first be audited.

POINT: LOW E-BAM: 21.8 C REF: -30.0 C CALIBRATE      DEFAULT
---

The calibration screens for TEMPERATURE, PRESSURE and FLOW are all similar. Each screen has a SETPOINT, E-BAM, REF, CALIBRATE and DEFAULT entry.

Set point; this is the intended value for the sensor. In the TEMPERATURE screen two values are selectable – HIGH and LOW. These correspond to ambient and ice bath values.

E-BAM: This is the measurement that the E-BAM is calculating for the selected sensor.

REF: This is the value that the Reference sensor is calculating. By entering the reference value into this entry and pressing.

CALIBRATE: Press this key to recalibrate the E-BAM sensor to the inputted REF value.

DEFAULT: Press this key to restore factory default values.

5. Allow an equilibration period of at least 15 minutes when the E-BAM and Reference temperatures are at the same location. If the test is an ambient test (above 20 degrees Celsius [68 degrees F]) select POINT: HIGH. If the test is an ice bath test select POINT: LOW. Note: it is recommended that the ambient point (HIGH) be done first due to the long equilibration period required for the temperature sensors to warm up.
6. Compare the reference temperature reading to the E-BAM reading on the LCD. If the readings are within 0.5 degree Celsius (1 degree F) no recalibration is necessary.
7. To recalibrate enter the reference temperature measurement into the REF: XX.X field and press calibrate.
8. Repeat this procedure for the second point.

## PRESSURE

The same protocol is used for PRESSURE with a single point calibration. Repeat the above steps using a reference pressure sensor. If the pressure values are within 2 mmHg no recalibration is necessary.

## FLOW

1. In the FIELD CALIBRATION screen select FLOW. Note: TEMPERATURE and PRESSURE must be audited prior to the FLOW.

TEMPERATURE
PRESSURE
>FLOW
MEMBRANE TEST

2. The FLOW calibration screen is similar to all E-BAM calibration screens.

FLOW SP: 16.7 LPM
E-BAM: 16.7 LPM
REF: 16.4 LPM
CALIBRATE      DEFAULT

FLOW SP: This is the flow rate value. In the FLOW screen three points are selectable – 14.0, 16.7, and 17.5 LPM.

E-BAM: This is the measurement that the E-BAM is calculating for the selected sensor.

REF: This is the value that the Reference sensor is calculating. By entering the reference value into this entry and pressing

CALIBRATE: Press this key to recalibrate the E-BAM sensor to the inputted REF value.

DEFAULT: Press this key to restore factory default values.

3. Once a setpoint is selected the E-BAM will automatically turn on the pump and regulate to the flow setpoint.
4. Remove the PM10 inlet and place the Reference flow audit device on the inlet tube. Wait for 5 minutes for the flow to equilibrate.
5. Compare the reference flow to the E-BAM flow. If the flows are within 2% no recalibration is necessary.
6. To recalibrate the flow enter the Reference Flow Meter reading into the REF: XX.X LPM entry and press CALIBRATE.
7. Repeat the above procedure for the remaining FLOW SP.

The FLOW system is checked and verified by these two tests – Leak and Flow Tests. When the E-BAM passes both tests the flow system will be working correctly.

### **3.3.1.3 Fixing a leak**

If you have performed the steps of a leak check and found that the E-BAM fails to have a flow rate less than 1.5 LPM there is a problem with the integrity of the flow system. Below is a list of solutions starting with the most common problems.

1. Nozzle/Vane build up – over time filter material can build up on the nozzle or on the vane under the filter paper. It is recommended that these areas be cleaned every 2 months or more frequently as needed. See the following steps A – D for the step-by-step procedure to clean the nozzle/van area.

#### **Nozzle Cleaning Procedure**

During normal operation the nozzle of the E-BAM can have a build up of filter material from on the sealing surface. Build up can also occur on the Vane. The vane is the crosshatch piece under the filter paper. Both of these must be cleaned periodically. Met One Suggests cleaning these areas every 2 months until the time interval is known.

- A. The nozzle needs to be lifted from the filter paper and the paper removed from the nozzle area. In the main menu enter the LOAD TAPE screen. The nozzle will automatically open. Lift the filter paper out of the nozzle area.

- B. Using a cotton applicator with a small amount of isopropyl alcohol gently clean the lip of the nozzle see Figure 30.

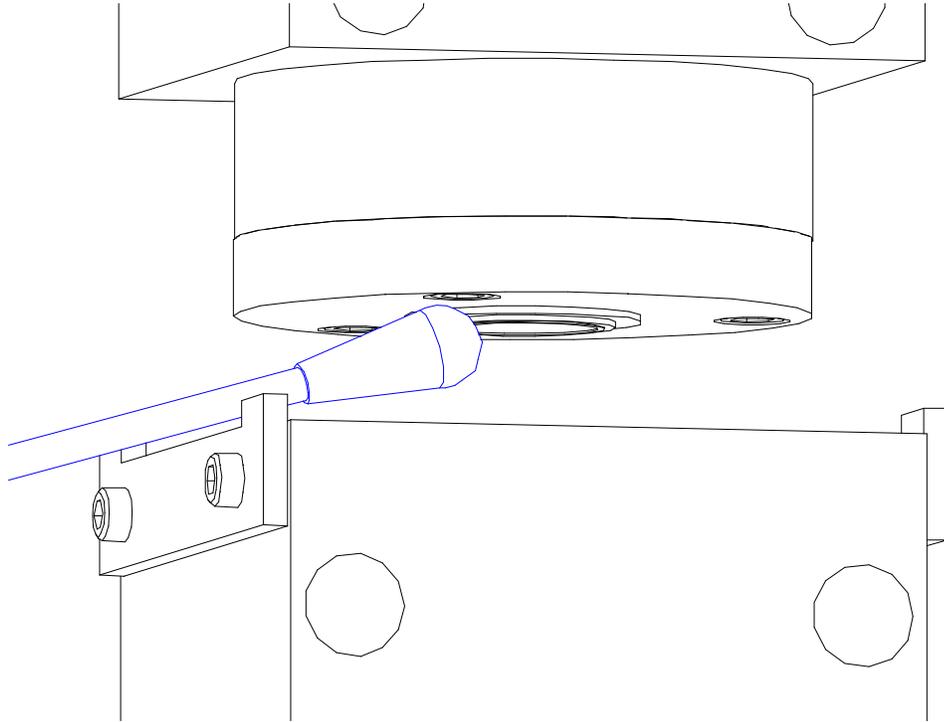


Figure 30 Nozzle cleaning diagram

The area to clean is the small lip that actually contacts the filter paper. Clean the entire diameter.

- C. After the nozzle has been cleaned the crosspiece vane needs to be cleaned. The vane sits directly under the nozzle.

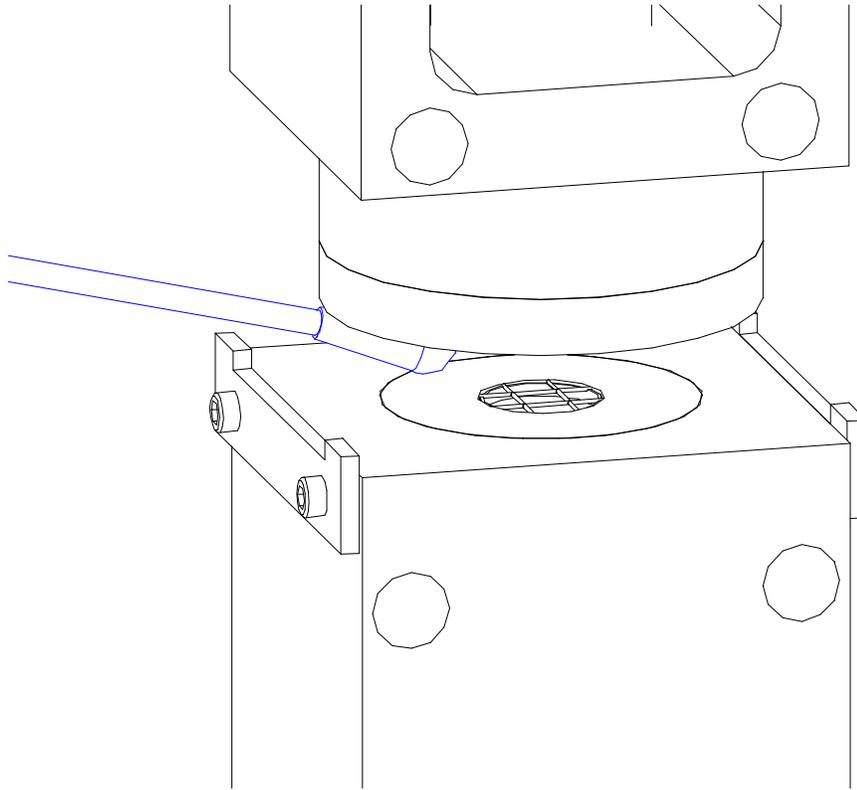


Figure 31 Vane Cleaning Procedure

Gently rub the cotton applicator across the four crosspiece sections of the vane and around the circumference of the vane.

D. Replace the filter paper and the E-BAM is ready for operation.

After a complete cleaning retest according to the Leak Check procedure. If the leak is above 1.5 LPM go to the next step.

2. Remove the Sharp Cut Cyclone (SCC) and retest. If the E-BAM passes the leak was in the SCC. Clean the SCC and replace O-rings as needed. If the E-BAM fails go to the next step.
3. Remove pump assembly – to remove the pump assembly see section 3.3.1.4. Do not remove the power from the PCB to the pump and don't remove the Tygon tubing from the pump ports. The pump will turn on if the E-BAM is in the FIELD CALIBRATION / PUMP TEST screen. Plug the top of the Nozzle assembly see Figure 32.

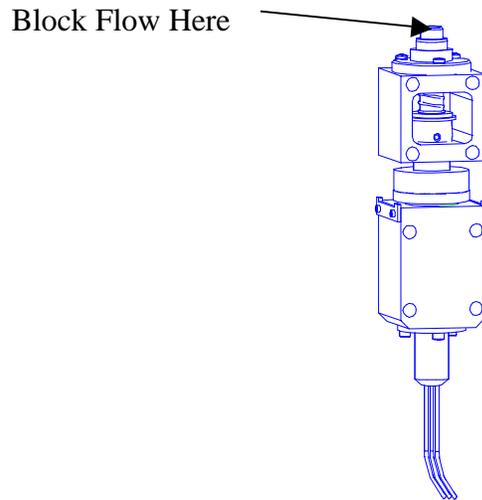


Figure 32 Location to plug during the Fixing a Leak procedure

If the flow stays below 1.5 LPM the leak is in the Inlet tube assembly. Check the O-rings for wear and replace as necessary. Remove the Inlet tube from the top of the E-BAM and check the O-rings inside the black plastic coupler for wear replace as necessary. Tighten all fittings and re-assemble. Recheck the flow and tighten and replace until the unit passes. If the flow rate always stayed above 1.5 LPM go to the next step.

4. Remove the chassis from enclosure and check fittings. To remove the chassis from the enclosure see section 3.3.2.2. Complete steps 1-4 only. This is a process of elimination. Each fitting must be checked for a leak. Start with the upstream connectors on the back of the chassis and work down to the flow meter. After each connector has been removed do a Leak check. When the Leak Check passes the last fitting removed must be replaced or serviced. If no leak is found down to the flow meter then the flow meter is faulty and must be replaced.

#### 3.3.1.4 Pump

The dual diaphragm pump in the E-BAM has an 8000 hour estimated lifetime. Under normal use it should be serviced once a year. The pump is not rebuildable. A replacement pump must be purchased and installed. For a complete list of spares/consumables and replacement parts see section 3.3.6.

#### Testing the Pump

A BX-305 will be needed to test the pump.

1. This test is found in the PUMP TEST screen. This screen is located in the MAIN MENU / FIELD CALIBRATION / PUMP TEST see Figure 33. There are two modes in this screen, which are LEAK TEST and PUMP TEST. Select PUMP TEST mode.

MODE: PUMP TEST
FLOW: 16.7 LPM
PRES: 999999 PA
EXIT

Figure 33 Pump Test screen.

2. Remove the inlet head(s).
3. Place the BX-305 over the inlet tube on the top of the E-BAM.
4. Turn the E-BAM on and allow the pump to equilibrate at 16.7 LPM. Slowly close the valve on the BX-305 until the flow is one of the values in Figure 34. Let the pump run at that flow rate for at least 1 minute. Re-adjust as necessary.

FLOW RATE LPM	Pump Condition Pressure Reading		
	Good	OK	Replace
14.0	52056	54138	57262
14.1	52212	54300	57433
14.2	52505	54605	57756
14.3	52662	54768	57928
14.4	52857	54971	58143
14.5	53131	55256	58444
14.6	53267	55398	58594
14.7	53482	55621	58830
14.8	53756	55906	59132
14.9	53932	56089	59325
15.0	54127	56292	59540

Figure 34 Vacuum test for pump condition.

5. Compare the pressure reading to the acceptable pressure readings in Figure 34. If the pressure is within tolerances the pump does not need to be replaced. If it is out of tolerance replace the pump.

**Remove the pump in the following order:**

1. Turn off the power to the E-BAM.
2. Open the cabinet door and remove the two 8-32 Phillips head screws holding the pump cover plate on.
3. Disconnect the pump power connector from the printed circuit board.
4. Take out the four 6-32 Phillips head screws holding the inlet tube to the pump.
5. Slide the upper sealing collar upward. There are no threads on the collar and it should move up with a slight twist and firm push.
6. Slide the lower sealing cover upward.
7. Take out the inlet tube.
8. Take out the three 6-32 Phillips screws holding the pump mount to the front panel.

9. Pull off the four 3/8 inch Tygon tubes from the pump ports. Be sure to mark the tubes so they go back on the same pump ports later.
10. The pump can now be taken out of the cabinet.
11. Re-install the pump in the reverse order.

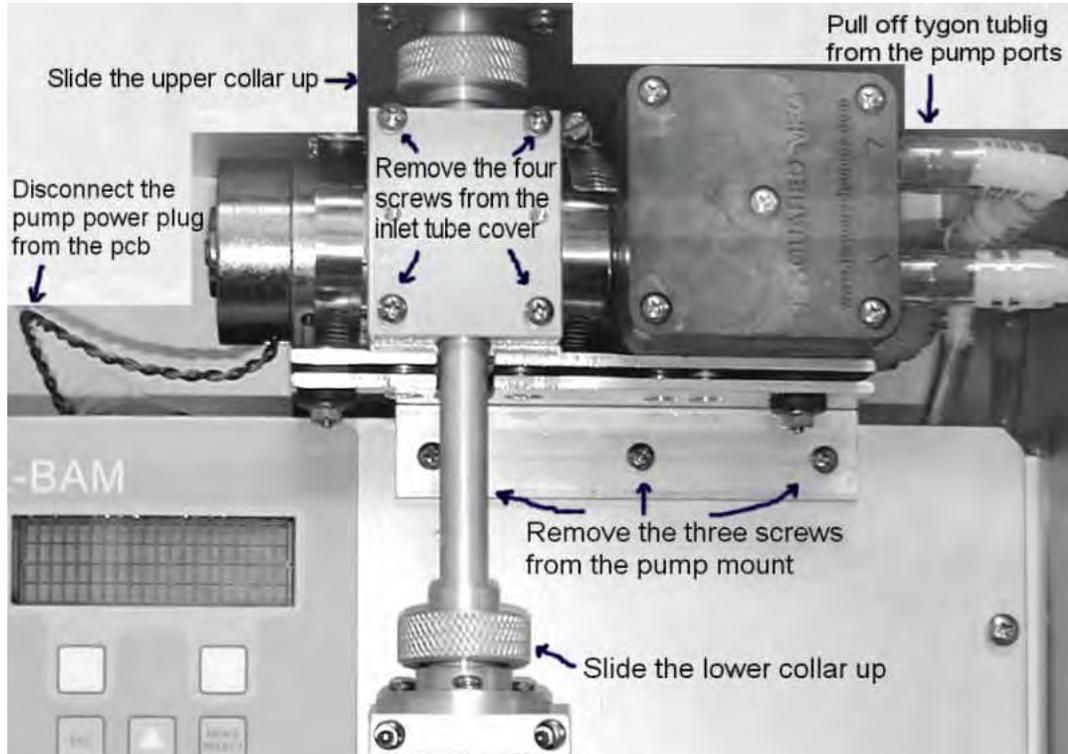


Figure 35 Removing the pump for replacement.

### 3.3.1.5 Inlet Heater Maintenance

The inlet heater needs periodic maintenance for the Filter RH and Filter Temperature sensors. Both sensors can be audited and calibrated from the E-BAM display.

#### Filter RH

The filter RH sensor is used to control the heater during periods when the sample air has an RH value that exceeds the setpoint see 3.2.2 for more details on the inlet heater. The Filter RH audit screen is located in the in the MAIN MENU / FIELD CALIBRATION / FILTER RH see Figure 36.

FILTER RH	
E-BAM:	57 %
REF:	xxx %
CALIBRATE	DEFAULT

Figure 36 Filter RH audit screen.

E-BAM: This is the measurement that the E-BAM is calculating for the selected sensor.

REF: This is the value that the Reference sensor is calculating. By entering the reference value into this entry and pressing CALIBRATE the E-BAM measurement will be calibrated to the entered value.

CALIBRATE: Press this key to recalibrate the E-BAM sensor to the inputted REF value.

DEFAULT: Press this key to restore factory default values.

### Filter Temperature

The filter temperature sensor is used to limit the heat added during periods when the sample air has an RH value that exceeds the setpoint see 3.2.2 for more details on the inlet heater. The filter temperature audit screen is located in the in the MAIN MENU / FIELD CALIBRATION / FILTER TEMPERATURE see Figure 37.

FILTER TEMPERATURE	
E-BAM:	xx.x C
REF:	-xx.x C
CALIBRATE	DEFAULT

Figure 37 Filter Temperature audit screen.

E-BAM: This is the measurement that the E-BAM is calculating for the selected sensor.

REF: This is the value that the Reference sensor is calculating. By entering the reference value into this entry and pressing CALIBRATE the E-BAM measurement will be calibrated to the entered value.

CALIBRATE: Press this key to recalibrate the E-BAM sensor to the inputted REF value.

DEFAULT: Press this key to restore factory default values.

### 3.3.2 Measuring System Checks – Zero and Span

The measuring system is the heart of the E-BAM. The major components of the E-BAM measuring system are the PMT, the source, and the CPU of the E-BAM. The source is very stable.  $^{14}\text{C}$  has a half-life of 5730 years. A beta gauge will operate correctly without source corrections for 10% of the half-life of the source. In theory the E-BAM will operate without need to recalibrate the source for over 500 years. The electronics, CPU and PMT, were selected for their dependability as well as accuracy.

To insure accurate measurements the E-BAM is supplied with a zero and span reference plate. This process does not recalibrate the instrument but does a check of the source, PMT and CPU to audit the measuring system. Met One Instruments recommends checking the measuring system with the Zero and Span plates once or twice a year.

### 3.3.2.1 Calibration Verification

Verification is accomplished using two calibration plates that represent a ZERO and SPAN factory set calibration points. The set of calibration plates are unique to each E-BAM. Always check that the serial number on the calibration plates match the serial number of the E-BAM to be calibrated. The SPAN calibration plate has a fragile membrane covering the hole. Never bump or touch the membrane. Always keep the membranes in their protective plastic case when not in use. When inserting the membranes into the E-BAM, be careful that you do not scrape or rub the filter tape with the metal plate or a calibration error may result. The membrane calibration will take about 16 minutes.

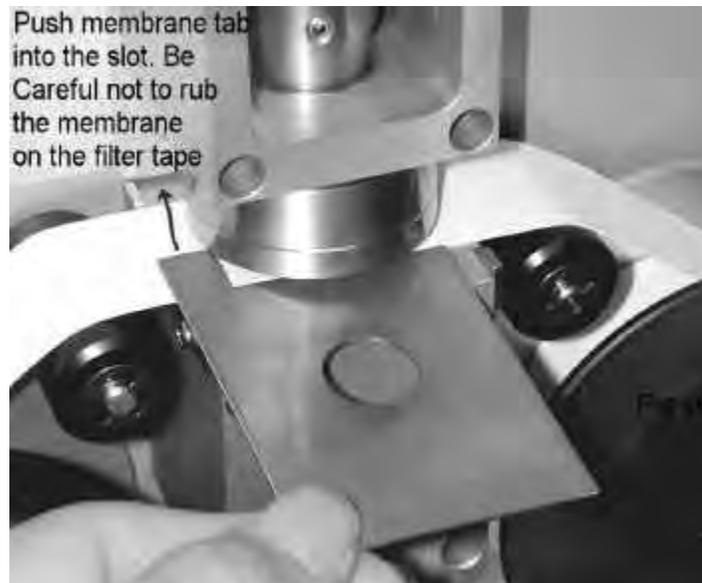


Figure 38 Inserting the Span/Zero plates

Undo the latch and swing open the E-BAM door. To turn on the display, press any key.

Go to the MENU SCREEN and use the up and down arrow keys to select FIELD CALIBRATION then press the SELECT key.

```
OPERATE
LOAD TAPE
SETUP
SELF TEST
>FIELD CALIBRATION
SHUTDOWN/SHIPPING
VIEW ALARM LOG
ABOUT
```

From the CALIBRATE MENU select MEMBRANE TEST and press the SELECT key.

```
TEMPERATURE
PRESSURE
FLOW
>MEMBRANE TEST
```

When you are ready to start the calibration press the START key.

```
MEMBRANE TEST
START ZERO TEST

START
```

The filter tape will advance, the nozzle will lower and the E-BAM will take a four-minute blank ZERO count. Press CANCEL to re-start the test.

```
BLANK ZERO COUNT

CANCEL
```

After the four-minute count the nozzle will move up and wait for you to insert the ZERO membrane.

```
INSERT ZERO MEMBRANE

CANCEL
```

Insert the ZERO Membrane and the nozzle will lower and the E-BAM will take a four-minute ZERO count see Figure 38.

CAL ZERO COUNT  
CANCEL

After the four-minute sample the nozzle will move up and ask you to remove the ZERO membrane.

REMOVE MEMBRANE  
CANCEL

When the ZERO membrane is removed the nozzle will lower and the E-BAM will take a four-minute blank SPAN count.

BLANK SPAN COUNT  
CANCEL

After the four-minute blank SPAN count, the nozzle will move up wait for you to insert the SPAN membrane.

INSERT SPAN MEMBRANE  
CANCEL

After you insert the SPAN membrane the nozzle will lower and the E-BAM will take a four-minute SPAN count see Figure 38.

```
CAL SPAN COUNT

CANCEL
```

Note the test results and take out the SPAN membrane.

```
MEMBRANE TEST RESULT
ZERO MEMBRANE: PASS
SPAN MEMBRANE: PASS
                        OK
```

If the ZERO or SPAN test failed, re-run the test. If the failure continues, clean the detector and re-run the test. If the failure persists, contact the factory service center. Press OK to return to the CALIBRATION MENU.

The measuring system's performance will be verified by completion of the Zero and Span tests.

### 3.3.2.2 Cleaning

Every year of operation, the accumulated dust on the detector-sensing region should be cleaned off. Refer to Figure 39 for visual instructions.

1. Disconnect all connections from under the E-BAM cabinet.
2. Remove the two 10-32 Phillips head screws from the connector plate under the cabinet.
3. Remove the four 10-32 Phillips head screws from the front panel of the E-BAM. Slide the upper sealing collar upward. There are no threads on the collar and it should move up with a slight twist and firm push.
4. The entire E-BAM assembly can now be lifted slightly and pulled out of the cabinet. Carefully lay the assembly on a clean flat surface with the front panel facing upward.
5. Take out the three 8-32 screws that hold the detector into the lower block and slide the detector out. Do not touch or bump the fragile sensing region on the end of the detector or permanent damage will result.
6. Use a can of office AeroDuster or similar low pressure clean air spray to blow off the dust on the sensitive area. Be sure to hold the nozzle at least two inches away when cleaning. If the dust will not blow off, use a Q-tip dipped in alcohol and rub lightly. When finished cleaning, re-assemble in reverse order.

Note: after each cleaning a Zero and Span must be done to ensure accurate data. See section 3.3.2.1 for instructions on Zero and Span verifications.

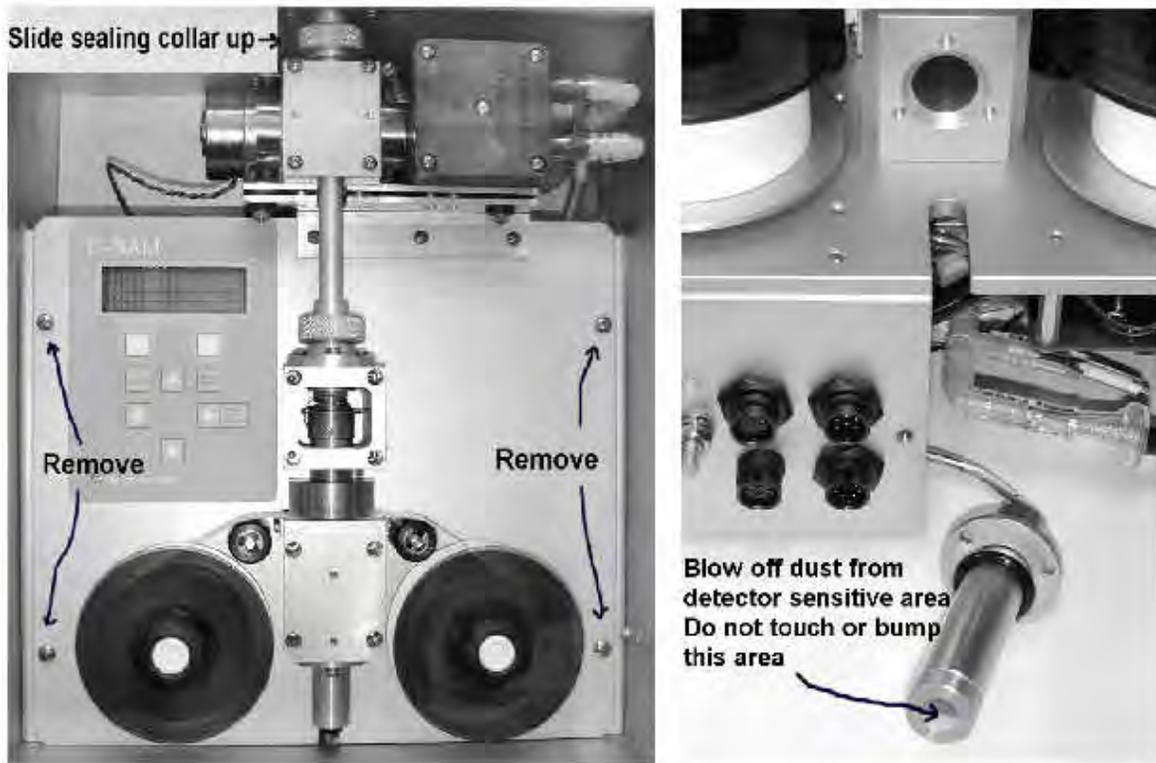


Figure 39 PMT removal for service.

### 3.3.3 Data System Checks

The data system is composed of two separate areas. The first area is the internal datalogger.

#### 3.3.3.1 Datalogger

The E-BAM has a 10 Channel datalogger. These channels record the following information:

- 1 – Real time Concentration
- 2 – Hourly Concentration
- 3 – Flow
- 4 – Wind Speed
- 5 – Wind Direction
- 6 – Ambient Temperature
- 7 – RH Ambient
- 8 – RH Filter
- 9 – Battery Voltage
- 10 – Filter

The datalogger can be checked out by downloading the internal data. Data may be downloaded through the following methods: using TUS software provided with each E-BAM, MicroMet Plus or AQ software purchased from Met One Instruments, a terminal program with ESC sequence commands or a terminal program with simple commands. For a complete list of ESC commands see section 10. For instructions on how to use the software packages reference the software manuals.

The following gives details on how to download data using a terminal program with a computer directly connected to an E-BAM.

2. Open the terminal software. If Windows<sup>™</sup> HyperTerminal is used go to **START / PROGRAMS / ACCESSORIES / COMMUNICATIONS / HyperTerminal**.
3. Open HyperTerminal, enter a name and press OK. A Connect To window will open like



Figure 40 HyperTerminal Connect to window.

4. Select the Com port that the E-BAM is connected to by using the drop down list from Figure 40. Press OK and that brings up

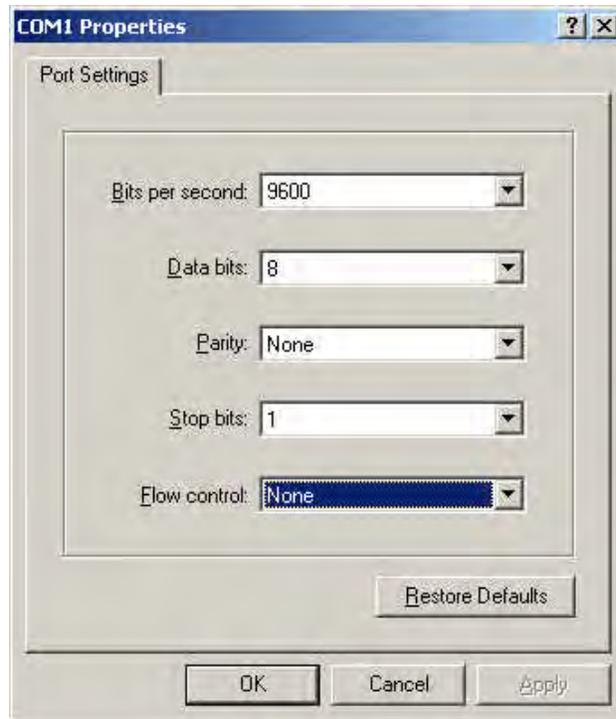


Figure 41 HyperTerminal communications protocol window.

In communications protocol window set the Bits per second window to match the setting on the E-BAM. The bits per second setting (baud rate) is found in SETUP screens. The SETUP screens are found in the MAIN MENU. The serial port baud rate can be set to 300, 600, 1200, 2400, 4800, 9600, 19200 and 38400. Use the arrow keys to make any changes. Note: the default setting is 9600. The other settings are **8** data bits, **NONE** parity, **1** stop bit, and **NONE** for flow control. Press OK and the setup is complete.

5. In the HyperTerminal window press ENTER 3 times.
6. Then press the number 2. This will download all the data from the E-BAM.

Once the data are downloaded check for missing time periods or aberrant data. This will verify that the averaging, recording and downloading of the data system is correct.

### 3.3.3.2 Analog Output

The second data system is the analog output of concentration. This data system requires a separate datalogger. The concentration will be calculated and converted to a corresponding voltage by user selectable parameters. Analog voltage can be either the hourly reading or the real-time reading. Full-scale voltage can be 1 volt, 2.5 volts, or 5 volts. To modify the analog voltage settings go to the SETUP screens. The SETUP screens are found in the MAIN menu.

To verify that the outputted voltage is correct use the ANALOG VOLTAGE screen. The ANALOG VOLTAGE screen is located in the MAIN MENU under the FIELD CALIBRATION heading. There are three modes for this screen – AUDIT, LOW, and HIGH.

The AUDIT mode allows the analog output to be set to any value from 0.000 volts up to the full-scale setting (full-scale is selected in the SETUP screens). After selecting AUDIT move the cursor using the arrow keys to highlight the SET PT value. Use the UP/DOWN keys to change the analog output setpoint. Using a voltmeter measure the output voltage on the communication cable (Figure 45) between the White (positive) and Black (ground) wires. If the voltmeter reading matches the SET PT value within 0.002 volts press exit to close this window. If the values don't match change the MODE to LOW.

```
MODE:  AUDIT
SETPT:  2.000 V
EXIT
```

Figure 42 Analog Voltage screen in AUDIT mode.

The LOW mode allows the E-BAM analog output to be adjusted to match an external datalogger or voltmeter. This adjustment is made at a low reading (.010 Volts). Connect the voltmeter, or datalogger, to the Communication Cable (Figure 45) between the White (positive) and Black (ground) wires. The voltmeter reading should be that same as the OUTPUT reading on the E-BAM. If the readings are different use the ADJUST command. Move the cursor using the RIGHT arrow key until it highlights the ADJUST value. If the voltmeter is reading higher than the OUTPUT reading of the E-BAM press the up arrow key to increase the ADJUST value. If the voltmeter is reading lower than the OUTPUT reading of the E-BAM press the DOWN arrow key to decrease the ADJUST value. Continue to change the ADJUST value until the voltmeter reading matches within 0.001 volts. Press SAVE and change the MODE to HIGH.

```
MODE:  LOW
OUTPUT: 0.010 V
ADJUST: -0.002
SAVE           DEFAULT
```

Figure 43 Analog Voltage screen in LOW mode.

The HIGH mode allows the E-BAM analog output to be adjusted to match an external datalogger or voltmeter. This adjustment is made at a HIGH reading close to the full-scale setting (full-scale is selected in the SETUP screens). Connect the voltmeter, or datalogger, to the Communication Cable (Figure 45) between the White (positive) and Black (ground) wires. The voltmeter reading should be that same as the OUTPUT reading on the E-BAM. If the readings are different use the ADJUST command. Move the cursor using the RIGHT

arrow key until it highlights the ADJUST value. If the voltmeter is reading higher than the OUTPUT reading of the E-BAM press the up arrow key to increase the ADJUST value. If the voltmeter is reading lower than the OUTPUT reading of the E-BAM press the DOWN arrow key to decrease the ADJUST value. Continue to change the ADJUST value until the voltmeter reading matches within 0.001 volts. Press SAVE and change the MODE to TEST.

MODE: HIGH
OUTPUT: 4.990 V
ADJUST: -0.001
SAVE                      DEFAULT

Figure 44 Analog Voltage screen in HIGH mode.

Use the voltmeter to verify that the correct voltage is outputted through the communication cable (Part number 9321). in the TEST mode at a low and high voltage reading. The communication cable schematic is shown in Figure 45.

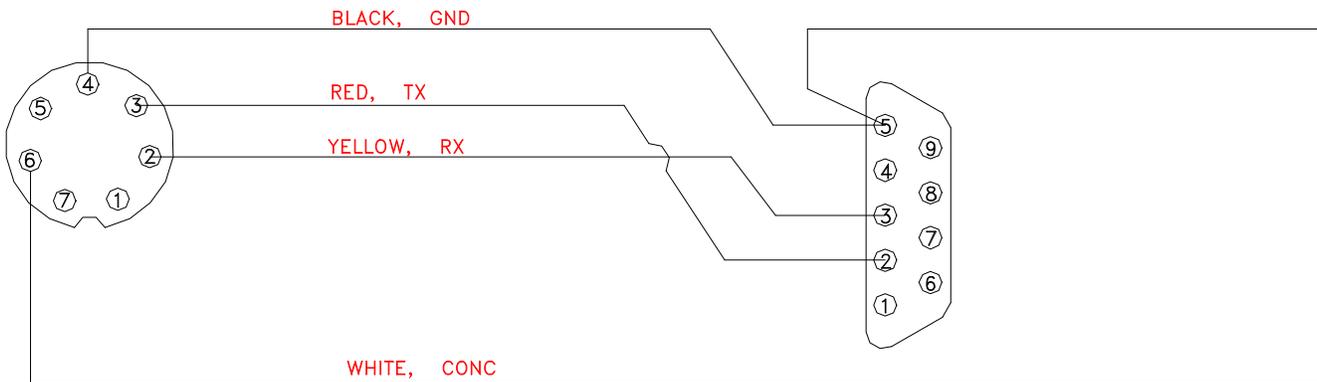


Figure 45 Communication cable for the E-BAM.

The black and white wires carry the analog voltage signal. White is positive and black is negative. If the analog voltage is incorrect the offset can be taken care of with an equal offset in the external datalogger or to correct the analog voltage offset go to the Figure 42 screen. This screen allows you to zero and span the analog voltage. Note: the E-BAM must be grounded to the same earth grounds as the datalogger. Use a 16-gauge wire connected to the ground lug see Figure 46. Connect the other end to the earth ground for the datalogger.

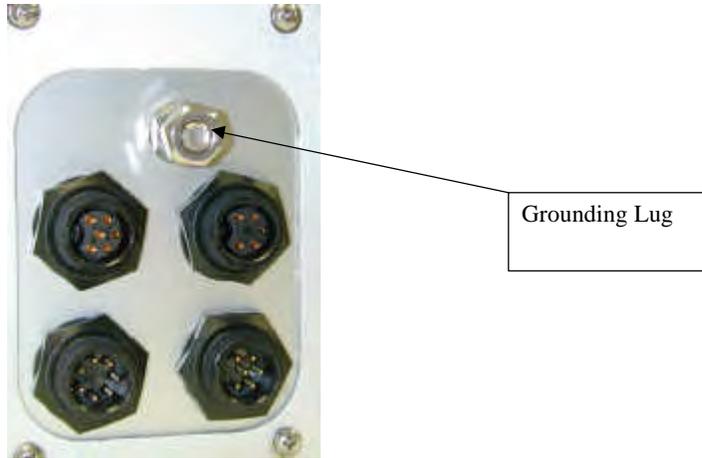


Figure 46 Grounding Lug for the E-BAM.

### 3.3.4 Miscellaneous Maintenance

1. Alarm Log – check this log to see if certain errors are occurring regularly. This log can be checked in the MAIN MENU under the heading of View Error Log. Note the errors and correct if necessary. For a troubleshooting guide see section 5.
2. Under continuous use, the PM10 inlet head should be removed and cleaned every six months. The BX-344 provides all the necessary items to clean the PM10 inlet. The PM10 inlet requires removal from the inlet tube, disassembly and cleaning. Disassemble the PM10 inlet and wipe clean with lint free cloth. Ensure that all O-ring surfaces are in excellent shape and are re-installed correctly. If O-rings are damaged see 3.3.6 for replacement parts.
3. If installed - Clean the PM2.5 SCC. The PM2.5 SCC inlet requires removal from the inlet tube, disassembly and cleaning. Disassemble the SCC and wipe clean with lint free cloth. Ensure that all O-ring surfaces are in excellent shape and are re-installed correctly. If O-rings are damaged see 3.3.6 for replacement parts.
4. Filter Tape – depending on Filter Tape Advance settings and concentration the filter tape will need to be replaced. A simple check each month will prevent the E-BAM from running out of filter tape.

### 3.3.5 Maintenance Schedule

Time Period	Item	Manual Section
Monthly	Leak Check	3.3.1.1
Monthly	Flow Audit	3.3.1.2
Monthly	Tape Check	3.3.4
Monthly	Alarm Check	3.3.4
Two Months	Pump Test	3.3.1.4
Six Months	Flow Calibration	3.3.1.2
Six Months	Analog Check for External Datalogger	3.3.3.2
Six Months	Inlet Cleaning	3.3.4
12 Months	Zero and Span Verification	3.3.2.1
12-24 Months	Pump replacement	3.3.1.4
12-24 Months	PMT Cleaning	3.3.2.2
5 Years	Internal Filter	

### 3.3.6 Accessories, Consumables and Replacement/Spares

#### Accessories

Description	Met One Part Number
WINS Impactor	BX-804
PM2.5 Sharp-Cut Cyclone	BX-807
TSP Inlet	BX-803
Solar Panel Array	Call
AC Power Supply	EX-120
Wind Speed/Direction	EX-034
Shielded Ambient RH	EX-593
Phone Line Modem	EX-961
Radio Modem	Call
Cell Phone Modem	Call
Zero Calibration Kit	BX-302
Flow Calibration Kit	BX-307
Flow Adapter	BX-305
Printer	BX-601
Inlet Extension Kit	BX-822

#### Consumables

Description	Met One Part Number
Filter Paper	460130
Internal Pisco Filter	580291
Internal Pisco Filter – Element only	580292

## Replacement/Spares

<u>Description</u>	<u>Met One Part Number</u>
Flow Meter 0-20 LPM	970608
Pump Assembly	8967
Pump (Only)	680865
Mounting Clamp $\frac{3}{4}$ x $\frac{3}{4}$	1552
Enclosure E-bam	9160
Tripod	EX-905
Inlet tube	9187
Black Plastic Inlet seal	480509
Power Cable	9248
Communication Cable	9321
Reference Membrane	9325
Zero Membrane	9166-s
PM10 Inlet	BX-802
Terminal Utility Software	TUS-1100
Pump Purge Tank	580255
Flow Meter Protection Filter	580299
Ambient Temperature Sensor	EX-592
Oring Kit (720043, 720066, 720069, 720110, 720150, 720200 3 each)	CALL
Internal RH Sensor	8624
Internal Temperature Sensor	8131
Gearmotor Assembly	8968
Spool Cover Assembly	9185
Pump Cover Sheet Metal	9233
Flow Meter	970608

### 3.4 Traceability

The E-BAM measurement provides gravimetric traceability for TSP, PM10 and PM2.5. Calibration of the E-BAM is done using the BAM1020 Standard. The BAM1020 is certified by the US-EPA as an Equivalent Method for PM10 (EQPM-0798-122).

## 4 Menu System

### 4.1 Keypad

The keypad has 6 regular keys and 2 soft keys. The regular keys never change function. The soft keys are dependent on the screen that is currently active.

<u>KEY</u>	<u>Description</u>
Soft Key Left	Command is displayed on the bottom left portion of the screen
Soft Key Right	Command is displayed on the bottom right portion of the screen
ESC	Cancels operation, Move Back one screen
MENU/SELECT	Selects highlighted selection, Bring up MAIN MENU.
	Move or Indexes up
	Move or Indexes left
	Move or Indexes down
	Move or Indexes right

<i>Soft Left</i>		<i>Soft Right</i>
<b>ESC</b>		<b>MENU/ SELECT</b>
		
		

Figure 47 Keypad description.

## 4.2 Operate Screen

Undo the latch and swing open the E-BAM door. To turn on the display, press any key. During normal operation, the OPERATE SCREEN will come on and display the following:

```
08-JUL-2001 08:23:41
 9.999 MG/M3  01:00
 9.999 MG/M3  (HR)
SAMPLING...
FLOW:  99.9 LPM
WS:  999.9 M/S
WD:  999.9 DEG
AT:  -99.9 C
FT:  -99.9 C
RHi:  999 %
RHx:  999 %
BV:  99.9 V
FLOW:  99.9 LPM
```

Press the ▲ and ▼ keys to traverse the parameter list.

## 4.3 Main Menu Screen

All functions of the E-BAM are accessed through the MENU SCREEN. Press the MENU/SELECT key on the keypad to display the MENU SCREEN.

The MENU SCREEN

```
OPERATE
LOAD TAPE
SETUP
MEMORY
SELF TEST
FIELD CALIBRATION
SHUTDOWN/SHIPPING
VIEW ALARM LOG
ABOUT
```

Press the ▲ and ▼ keys to traverse the MENU list.

Press the SELECT key to select the menu item.

Press the ESC key to return to the Operate screen.

#### 4.4 Loading Tape

Undo the latch and swing open the E-BAM cover. To turn on the display, press any key.

Press the MENU key on the keypad. Use the up and down arrow keys to select LOAD TAPE

OPERATE
<b>LOAD TAPE</b>
SETUP
MEMORY
SELF TEST
FIELD CALIBRATION
SHUTDOWN/SHIPPING
VIEW ALARM LOG
ABOUT

Press the SELECT key.

The nozzle will move upward and stop. The display will indicate LOAD TAPE.

PLEASE LOAD TAPE!
CONTINUE

Remove both of the clear plastic spool covers by unscrewing the captive knobs. An empty tape roll core tube must be installed on the hub of the take-up spool. Special care should be taken when handling unused filter tape because it tears easily. Place full roll of filter tape on supply (right) spool, with the tape feeding upward and counter-clockwise as shown below. Feed end of filter tape so that it enters the take-up spool in a counter-clockwise direction. Using any available adhesive tape, attach the leading end of the filter tape to the core tube to prevent slipping. Gently tension the tape. Re-install both spools. When finished answer CONTINUE see Figure 48.

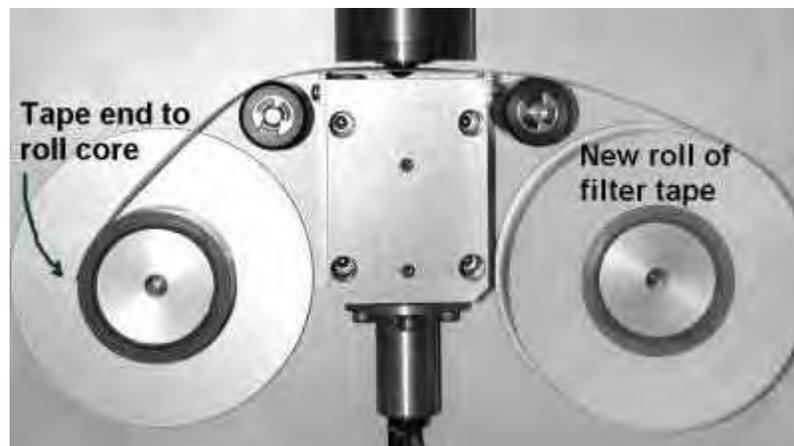


Figure 48 details filter tape installation.

The filter tape will move and take up tension.

```
CHECKING FOR
LOADED TAPE.

PLEASE WAIT...
```

After the tape has been verified the main menu will be displayed.

#### 4.5 Setup

All user-defined parameters are entered through the **SETUP** selection from the **MENU** screen.

Go to the **MENU SCREEN** and use the up and down arrow keys to select **SETUP**.

```
OPERATE
LOAD TAPE
SETUP
MEMORY
-----
SELF TEST
FIELD CALIBRATION
SHUTDOWN/SHIPPING
VIEW ALARM LOG
ABOUT
```

Press the **SELECT** key.

The set date and time screen will appear. Use the arrow keys to make any changes.

```
18-JUL-2001 08:23:41

SET          CONTINUE
```

Press **SET** to save the date and time. Press **CONTINUE** to not make changes and continue to the next screen.

```
LOCATION: 01
TAPE ADVANCE: 24 HRS
REALTIME AVG: 1 MIN
EDIT          OK
```

Press **SAVE** to save the changes or **CONTINUE** to proceed and not make changes.

The output full-scale voltage for the concentration can be set to 1, 2.5, or 5 Volts. Use the arrow keys to make any changes. Note: the concentration range is always 0-1 mg/m<sup>3</sup> regardless of full-scale voltage setting. If an excursion occurs where the concentration exceeds 1 mg/m<sup>3</sup> the data points are recorded in the internal E-BAM datalogger and can be downloaded to supplement the external datalogger information.

The MODE setting determines what type of concentration reading controls the concentration analog output. Select HOURLY for hourly concentration or REAL TIME for real time concentration.

REF DAC FS setting is what a setting that is determined by the production date. If the serial number of the E-BAM is the letter Y, A, or B the REF DAC FS setting should be 10.0 Volts. If the serial number is any other letter the setting is 8.0 Volts. This voltage determines the minimum voltage the E-BAM will operate with. For the 10.0 volts system the minimum voltage is 10.3 volts DC. For the 8.0 volts system the minimum voltage is 9.5 volts.

```
ANALOG FS:  5.0 V
          MODE: REAL TIME
REF DAC FS: 10.0 V
SAVE          CONTINUE
```

Press SAVE to save the changes or CONTINUE to proceed and not make changes.

The serial port baud rate can be set to 300, 600, 1200, 2400, 4800, 9600, 19200 and 38400. Use the arrow keys to make any changes.

```
SERIAL PORT
BAUD RATE: 1200
SAVE          CONTINUE
```

Press SAVE to save the changes or CONTINUE to proceed and not make changes.

The Heater setup screen is used to modify the inlet heater settings. The RH may be set to any value from 1-100%. The DELTA-T may be set to any value from 0-20 C. The RH CONTROL is set to ON or OFF. Setting the RH CONTROL to OFF disables the inlet heater. For a complete discussion of the inlet heater see section 3.2.2.

```
RH SETPOINT:100 %
DELTA-T SETPT: 10 C
RH CONTROL: OFF
SAVE          CONTINUE
```

Press SAVE to save the changes or CONTINUE to proceed and not make changes.

The Flow setup screen is used to change the flow rate and flow type. Flow type is either ACTUAL or STANDARD see Figure 28 for details. The flow rate may be set from 10 to 17.5 LPM.

```
FLOW
SETPOINT: 16.7 LPM
        TYPE: ACTUAL
SAVE          CONTINUE
```

Press SAVE to save the changes or CONTINUE to proceed and not make changes.

#### 4.6 Memory

The memory screen give detail on the amount of memory remaining and also allows the permanent deleting of memory.

```
OPERATE
LOAD TAPE
SETUP
MEMORY
SELF TEST
FIELD CALIBRATION
SHUTDOWN/SHIPPING
VIEW ALARM LOG
ABOUT
```

Highlight the MEMORY heading in the main menu and press SELECT. The following screen appears:

```
MEMORY
999 % REMAINING
999.9 DAYS REMAINING
CLEAR          EXIT
```

Two indications of memory are given which are “% REMAINING” and “DAYS REMAINING” is determined by the % Remaining and the averaging period selected see Figure 7. Pressing CLEAR opens the following screen:

```
1 CLEAR MEMORY
2 CLEAR: DATA LOGGER
3
4 CLEAR          EXIT
```

There are two memory locations to clear which are the DATA LOGGER and ALARM LOG. Selecting the memory location and pressing CLEAR opens the following screen:

```
>>>> CAUTION <<<<<
CLEAR DATA LOGGER
MEMORY?
NO YES
```

Pressing YES will permanently delete the memory.

#### 4.7 Self-Test

The E-BAM has a SELF TEST mode that will automatically test all parameters. The SELF TEST will take several minutes and cannot be bypassed by the operator. If any fault is located, the name and type of fault will be shown. If there is a reasonable expectation that the operator could correct the fault, then an instruction for correction is shown.

Go to the MENU SCREEN and use the up and down arrow keys to select SELF TEST. Press the SELECT key.

```
OPERATE
LOAD TAPE
SETUP
MEMORY
SELF TEST
FIELD CALIBRATION
SHUTDOWN/SHIPPING
VIEW ALARM LOG
ABOUT
```

The E-BAM display will indicate SELF TEST RUNNING.

```
SELF TEST RUNNING...

*****999
```

The SELF TEST will take several minutes and cannot be bypassed by the operator.

After SELF TEST is finished, the following screen is displayed.

```
SELF TEST COMPLETE:
E-BAM FUNCTIONING
PROPERLY.
CONTINUE
```

Answer CONTINUE and the E-BAM will begin sampling.

If SELF TEST failed, for example if the pump failed to turn on, the following screen would appear.

```

FLOW SYSTEM FAILED!

                                CONTINUE

```

#### 4.8 Field Calibration

This screen is used to verify the operation of the E-BAM. Each test is explained in the Maintenance section of the manual.

```

OPERATE
LOAD TAPE
SETUP
MEMORY
SELF TEST
FIELD CALIBRATION
SHUTDOWN/SHIPPING
VIEW ALARM LOG
ABOUT

```

Selecting FIELD CALIBRATION opens the following screen:

```

TEMPERATURE
PRESSURE
FLOW
FILTER RH
FILTER TEMP
PUMP TEST
ANALOG AUDIT
MEMBRANE TEST

```

Each of the FIELD CALIBRATION test are explained in the maintenance section. Refer the following list to find a complete explanation of the test.

<u>Test</u>	<u>Section</u>
TEMPERATURE	3.3.1.2
PRESSURE	3.3.1.2
FLOW	3.3.1.2
MEMBRANE TEST	3.3.2.1
FILTER RH	3.3.1.5
FILTER TEMPERATURE	3.3.1.5
PUMP TEST	3.3.1.4

## 4.9 Shutdown for Shipping

When the E-BAM must be moved or sent to the factory for service, the filter tape should be removed and the nozzle protector put in place.

Undo the latch and swing open the E-BAM cover. To turn on the display, press any key. Press the MENU key. Using the arrow keys, scroll to SHUTDOWN/SHIPPING and press ENTER.

OPERATE
LOAD TAPE
SETUP
MEMORY
SELF TEST
FIELD CALIBRATION
<b>SHUTDOWN/SHIPPING</b>
VIEW ALARM LOG
ABOUT

The SHUTDOWN/SHIPPING screen will appear and the nozzle will move up and stop. The display will blink INSERT NOZZLE PROTECTOR. Remove the filter tape and insert the nozzle protector. Press the DONE soft key. The nozzle will come down. Unplug the power cable and remove the E-BAM box from the tripod. The E-BAM is now ready for shipping.

## 4.10 View Error Log

The Error Log screen shows the current E-BAM error log. Press the MENU key.

OPERATE
LOAD TAPE
SETUP
MEMORY
SELF TEST
FIELD CALIBRATION
SHUTDOWN/SHIPPING
<b>VIEW ALARM LOG</b>
ABOUT

Use the down arrow to the VIEW ERROR LOG item and then press the SELECT key.

10-DEC-2001 09:10:29
POWER OUTAGE
OFF: 1.22:50:09
ON: 0.00:17:50

Press the left arrow key to go to an older error log event.

Press the right arrow key to go to a newer error log event.

Press the ESC key to return to the newest error log event.

#### **4.11 Error Screens**

The following is a list of events in the ALARM LOG:

1. POWER OUTAGE – Power was discontinued.
2. INTERNAL COMM DOWN – Internal communication error.
3. NOZZLE MOTOR FAILED UP – Nozzle failed to open.
4. NOZZLE MOTOR FAILED DOWN – Nozzle failed to close.
5. TAPE BROKE– filter tape is broken.
6. BETA COUNT FAILED – the PMT tube did not count a minimum of 40,000 counts for 1 minute.
7. PRESSURE TEST FAILED – one or both of the internal pressure sensors failed.
8. FLOW FAIL– flow was less than 5 LPM for more than 2 minutes or flow was out of regulation for more than 5 minutes.
9. SHIPPING DEVICE INSERTED – nozzle-packing material was not removed.
10. MEMBRANE FAILED – Membrane test failed.
11. BATTERY LOW – low battery voltage during normal operation.
12. HIGH TAPE DELTA-PRESS – high pressure drop across the filter paper.  
Note: filter tape is advanced subsequent to this event.
13. HIGH DELTA-T – delta temperature from Ambient to Filter exceed the setpoint.
14. PUMP OVER TEMP – pump was operating out of E-BAM specifications and the unit was turned off until pump returned to specified temperature conditions.
15. COP RESET – CPU failure and the system reset. Note: after firmware upgrades to the E-BAM a COP RESET will be recorded due to the resetting of the system for the new firmware.

#### **4.12 About Screen**

The ABOUT screen shows the current E-BAM installed version of firmware and the serial number. Press the MENU key.

OPERATE LOAD TAPE SETUP MEMORY
SELF TEST FIELD CALIBRATION SHUTDOWN/SHIPPING VIEW ALARM LOG <b>ABOUT</b>

Use the down arrow to go to **ABOUT** and then press **SELECT** key.

E-BAM V1.00 SN: A1234 Met One Instruments <a href="http://www.metone.com">www.metone.com</a>
---

Press the **MENU** key to return to the menu screen.

## 5 Troubleshooting

A basic troubleshooting guide is shown below:

<b>SYMPTON</b>	<b>PROBABLE CAUSE</b>	<b>REMEDY</b>
Low or no 12 VDC power	Bad battery, discharged battery, bad connection	Test battery, charge or replace if necessary, clean battery connections, replace power cable
Pump will not start	Bad pump	Check pump and replace if bad, Lift nozzle and check for obstruction in flow path.
Flow rate is too low	Air leak, bad pump, obstruction in air path	Check for an air leak, Check pump and replace if bad
No filter tape movement	Bad motor/drive	Replace the motor/drive unit
Filter tape slips	End of tape is slipping on take up spool	Tape end of filter tape to the take up spool
Filter tape is being cut by the nozzle	Debris under nozzle	Lift nozzle and clean off debris
Nozzle does not move	Bad motor or limit switch	Replace the motor or limit switch.

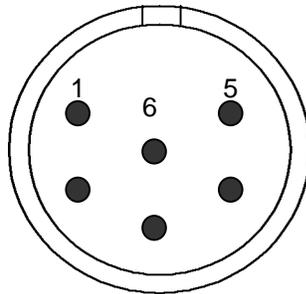
## 6 Specifications

Parameter	Specification
Measurement Method	Beta Attenuation
Beta Source	C <sup>14</sup> , less than 75 microcurie, Half life of 5730 years
Reference Method	Gravimetric method
Range	0 –65 mg per cubic meter
Accuracy	+/- 10% of indication (one hour mode)
Measurement Cycle	24 Hours
Detector:	Scintillation probe
Analog Output	0-1V, 0-5V, 0-10V selectable, 12 bit accuracy
Filter Tape	Continuous glass fiber filter
Inlet	PM10 Virtual Impactor type
Flow Rate:	16.7 liters per minute, adjustable
Flow accuracy	+/- 3% of reading
Flow Controller	Mass flow meter with DC motor controller
Sample Pump	Dual diaphragm type, internally mounted
Alarm Signals	Filter, flow, power and operation failure
Input Power	12 Volts DC @ 35 Watts
Display	4 line X 20 vacuum character florescent display (VFD)
Keypad	Membrane type, 3 X 4 matrix
Alarm Contact Closure	2 Amp @ 240 VAC
Communications	Standard RS232
Operating Temperature	-25 Deg C to 40 Deg C
Storage Temperature	-30 Deg C to 40 Deg C
Enclosure	Aluminum weatherproof, 14" W, 16" H, 8" D

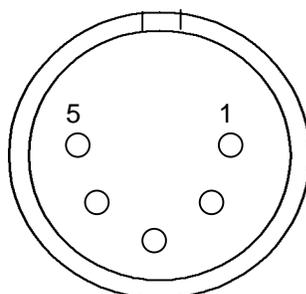
## 7 Appendix A

### 7.1 Input /Output Connectors

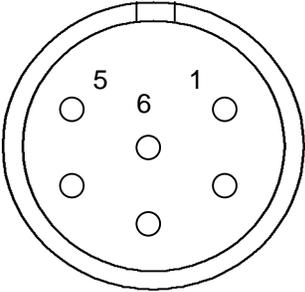
<b>POWER CONNECTOR (PLUG) (VIEW FROM BOTTOM OF E-BAM)</b>	
1	RELAY PIN (NORM CLOSED)
2	RELAY PIN
3	+12 V
4	+12 V
5	COMMON
6	COMMON



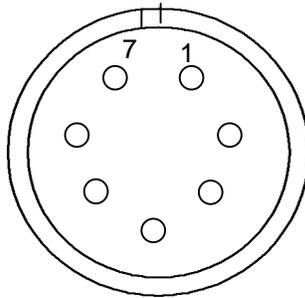
<b>WD / WS CONNECTOR (SOCKET) (VIEW FROM BOTTOM OF E-BAM)</b>	
1	WD REF
2	COMMON
3	WD SIGNAL
4	WS SIGNAL
5	SHIELD



<b>RH/TEMP/BARO CONNECTOR (SOCKET)</b> <b>(VIEW FROM BOTTOM OF E-BAM)</b>	
1	RH SIG
2	COMMON
3	+12 V
4	TEMPERATURE SIG
5	BAROMETER SIG
6	SHIELD



<b>COMM / CONC (SOCKET)</b> <b>(VIEW FROM BOTTOM OF E-BAM)</b>	
1	RST
2	RX
3	TX
4	COMMON
5	STX
6	CONC OUT
7	TCK



## 8 Appendix B

### 8.1 Theory of Operation

When the high-energy electrons emanating from the radioactive decay of  $^{14}\text{C}$  (carbon-14) interact with nearby matter, they lose their energy and, in some cases, are absorbed by the matter. These high-energy electrons emitted through radioactive decay are known as beta rays and the process is known as beta-ray attenuation. When matter is placed between the radioactive  $^{14}\text{C}$  source and a device designed to detect beta rays, the beta rays are absorbed and/or their energy diminished. This results in a reduction in the number of beta particles detected. The magnitude of the reduction in detected beta particles is a function of the mass of the absorbing matter between the  $^{14}\text{C}$  beta source and the detector.

The number of beta particles passing through absorbing matter, such as dust deposited on a filter tape, decrease nearly exponentially with the mass through which they must pass. Equation 1 shows this relationship.

$$I = I_0 e^{-\mu x}$$

Equation 1

In Equation 1,  $I$  is the measured beta ray intensity (counts per unit time), of the attenuated beta ray (dust laden filter tape),  $I_0$  is the measured beta ray intensity of the un-attenuated beta ray (clean filter tape),  $\mu$  is the absorption cross section of the material absorbing the beta rays ( $\text{cm}^2/\text{g}$ ), and  $x$  is the mass density of the absorbing matter ( $\text{g}/\text{cm}^2$ ).

Equation 1 very closely resembles the Lambert-Beers Law, which is used in spectrometric analysis. Just as the Lambert-Beers Law is an idealization of what is actually observed, Equation 1 is also an idealized simplification of the true processes occurring meant to simplify the corresponding mathematics. However, experimental measurement shows that in properly designed monitors, such as the BAM-1020, the use of this equation introduces no substantial error.

Equation 1 may be rearranged to solve for  $x$ , the mass density of the absorbing matter. This is shown in Equation 2.

$$-\frac{1}{x} \ln \left[ \frac{I}{I_0} \right] = \frac{1}{x} \ln \left[ \frac{I_0}{I} \right] = x$$

Equation 2

In practice, the absorption cross section is experimentally determined during the calibration process. Once  $I$  and  $I_0$  are experimentally measured, it is a simple matter to calculate  $x$ , the predicted mass density.

In practice, ambient air is sampled at a constant flow rate ( $Q$ ) for a specified time  $\Delta t$ . This sampled air is passed through a filter of surface area  $A$ . Once  $x$ , the mass density of collected particles, has been determined, it is possible to calculate the ambient concentration of particulate matter ( $\mu\text{g}/\text{m}^3$ ) with Equation 3.

$$c \left( \frac{\mu\text{g}}{\text{m}^3} \right) = \frac{10^6 A(\text{cm}^2)}{Q \left( \frac{\text{liter}}{\text{min}} \right) \Delta t(\text{min}) \left( \frac{\text{cm}^2}{\text{g}} \right)} \ln \left( \frac{I_0}{I} \right)$$

Equation 3

In Equation 3,  $c$  is the ambient particulate concentration ( $\mu\text{g}/\text{m}^3$ ),  $A$  is the cross sectional area on the tape over which dust is being deposited ( $\text{cm}^2$ ),  $Q$  is the rate at which particulate matter is being collected on the filter tape (liters/minute), and  $\Delta t$  is the sampling time (minutes). Combining these equations yields to the final expression for the ambient particulate concentration in terms of measured quantities. This is shown in Equation 4.

$$c \left( \frac{\mu\text{g}}{\text{m}^3} \right) = \frac{10^6 A(\text{cm}^2)}{Q \left( \frac{\text{liter}}{\text{min}} \right) \Delta t(\text{min}) \left( \frac{\text{cm}^2}{\text{g}} \right)} \ln \left( \frac{I_0}{I} \right)$$

Equation 4

The key to the success of the beta attenuation monitor is due in part to the fact that  $\mu$ , the absorption cross-section is almost insensitive to the nature of the matter being measured. This makes the BAM-1020 very insensitive to the chemical composition of the material being collected.

It is instructive to perform a conventional propagation of errors analysis on Equation 4. Doing so, one can develop an equation for the relative measurement error ( $\sigma_c/c$ ) as a function of the uncertainty in each of the parameters comprising Equation 4. This leads to Equation 5.

$$\frac{\sigma_c}{c} = \sqrt{\frac{\sigma_A^2}{A^2} + \frac{\sigma_Q^2}{Q^2} + \frac{\sigma_t^2}{t^2} + \frac{\sigma_{\mu}^2}{\mu^2} + \frac{\sigma_I^2}{I^2 \ln^2 \left[ \frac{I}{I_0} \right]^2} - \frac{\sigma_{I_0}^2}{I_0^2 \ln^2 \left[ \frac{I}{I_0} \right]^2}}$$

Equation 5

Inspection of Equation 5 reveals several things. The relative uncertainty of the measurement ( $\sigma_c/c$ ) is decreased (improved) by increasing the cross sectional area of the filter tape (A), the flow rate (Q), the sampling time (t), the absorption cross section ( $\mu$ ), I and  $I_0$ .

## 9 How to convert from Standard flow to Actual flow

Any volumetric flow can be found from a standard flow using the Ideal Gas law. The Ideal Gas Law in this situation is really a ratio of the temperature and pressure at referenced conditions (760 mmHg, 25°C EPA STD Conditions) versus Actual conditions of temperature and pressure.

### DEFINITIONS

- $Q_x$  = Volumetric flow at Actual conditions of pressure and temperature
- $Q_s$  = Volumetric flow at standard conditions of 760 mmHg (1 atm) and 25°C (298.13 K)
- $T_x$  = Temperature at Actual conditions in Kelvin (K)
- $T_s$  = Temperature at standard conditions in Kelvin (K)
- $P_x$  = Pressure at Actual conditions in Kelvin (K)
- $P_s$  = Pressure at standard conditions in Kelvin (K)
- $n$  = Number of moles of gas
- $R$  = Gas constant .0821 (liters atm/mole K) or 82.1 (cm atm/mole K)
- $m$  = Mass in grams (g)
- $m_{\text{dot}}$  = Mass Flow (g/min)

The conservation of mass tells us that if mass flow is held constant over temperature and pressure, then the following is true:

$$m_{\text{dots}} = m_{\text{dot}x}$$

Where:

$m_{\text{dots}}$  - mass flow, at standard conditions

$m_{\text{dot}x}$  - mass flow at actual conditions of temperature and pressure

Using this relation and the Ideal Gas Law Yields the following equation:

$$\frac{mP_x}{nRT_x} \cdot Q_x = \frac{mP_s}{nRT_s} \cdot Q_s$$

Solving for  $Q_x$  gives the following equation:

$$Q_x = Q_s \cdot \frac{P_s}{P_x} \cdot \frac{T_x}{T_s}$$

Example:

$$\begin{aligned} Q_s &= 16.7 \text{ LPM} \\ P_s &= 760 \text{ mmHg} \end{aligned}$$

Ts = 298.13 K  
Px = 710 mmHg  
Tx = 303 K

$$Q_x = 16.7 \text{ LPM} \cdot \frac{760 \text{ mmhg}}{710 \text{ mmhg}} \cdot \frac{303 \text{ K}}{298.13 \text{ K}}$$

Answer:  $Q_x = 18.16 \text{ LPM}$

## 10 ESC Commands

The E-BAM can be controlled remotely by use the following command set.

Each command and response string begins with an Esc character (27, 0x1B) and is terminated with a carriage return (13, 0x0D) and a line feed character (10, 0x0A).

An ASCII check sum follows each response (X9999).

Hardware protocol is RS-232, 8, N, 1.

### Commands

The E-BAM supports 5 data files—the EEPROM file (E), the Channel Descriptor file (1), the AutoMet data log file (2), the Error log file (3), and the One-Minute data log file (4).

The lowercase x of a command specifies the file to operate on. Valid values for x are shown in (x) above.

File modes can be linear (L) or circular (C). All files are record based. Record sizes and formats are described above.

Function	Command	Response
Read Model and Version	RV	RV E-BAM V1.23
Read File Info (FCB)	RFI	RFI Then print the FCB.
Read record index.	RFx R	RFx R n L RFx R n C
Read data file starting with absolute record index or the last (-n) records from the current record index.	RFx D n RFx D -n	RFx D n Then XMODEM file transfer
Print File Report starting with absolute record index or the last (-n) records from the current record index.	PFx n PFx -n	PFx n Then print the report.
Stop printing report.	PFS	PFS
Clear File Data (x: 2, 3, 4)	WFx C	WFx C
Read Date (mm-dd-yy)	RD	RD 05-10-01
Write Date (mm-dd-yy)	WD 05-10-01	WD 05-10-01
Read Time (HH:mm:ss)	RT	RT 09:08:02
Write Time (HH:mm:ss)	WT 09:08:02	WT 09:08:02
Read State Machine	RS	RS M, C, S
Write State Machine	WS M, C, S	WS M, C, S
Print factory calibration report starting with absolute record index or the last (-n) records from the current record index.	PFC n PFC -n	PFC n Then print the report.

APPENDIX C

# **Sampling Standard Operating Procedures**

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# C.0 Sampling Standard Operating Procedures

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## C.1 Site Operation

The station will be operated by local CH2M HILL personnel who have been trained by CH2M HILL staff experienced with the Environmental Protection Agency (EPA) guidance documents for operation of ambient air and meteorological monitoring stations. The station datalogger is linked by satellite that provides near real-time data for the system.

CH2M HILL personnel review the instantaneous data each day for faults, errors, and data reasonability. This is the first line of defense in assuring maximum data recovery. This daily data review and remote access, coupled with the local site operator visits enables us to monitor data recovery and minimize downtime. CH2M HILL has implemented this system to reduce field labor efforts, promptly identify instrument malfunctions, and maximize data completeness. Duties include verifying proper operation of the instruments and performing routine quality control checks. This process also serves as a tool for identifying and correcting instrument malfunctions in a timely manner, thereby minimizing loss of data.

Quality Assurance/Quality Control (QA/QC) and maintenance activities will be noted on field data forms and copies kept in a field logbook for easy referral by field personnel. Copies of all field checks and maintenance are sent to the Data Coordinator.

Duties of the site operator include verifying proper operation of the instruments, downloading data to a storage device when necessary, and performing routine quality control checks. The operator is responsible for performing maintenance on a regularly scheduled basis. These procedures and other corrective actions meet the QA and data completeness goals of the project.

Important documents such as calibration certificates, performance audit assessments, and other pertinent information will also be kept in a central file in CH2M HILL offices.

## C.2 Weekly Site Visits

At least one weekly inspection or visit will be made to each of the monitoring stations. For every site visit, the field technician is required to disable the datalogging system so that the test conditions to which the instruments are subjected are not included in the data set. Then the technician is required to follow safety procedures outlined in the Standard Operating Procedures (SOP), taking care not to damage the instruments.

### C.2.1. Field Records

Field operators are required to keep accurate written records of their activities in a bound logbook. All entries should be legible, written in waterproof ink, and contain accurate and inclusive documentation of an individual's field activities, including observations, any problems encountered, and actions taken to solve the problem. The type of data recorded in the field logbook includes site visits, maintenance and calibration activities, field

measurements, ambient conditions, and any other information pertinent to sample collection. Entry errors or changes should be crossed out with a single line, dated, and initialed by the person making the correction. Entries made by individuals other than the person to whom the logbook was assigned will be dated and signed by the individual making the entry. Field logbooks will be available to the data and QA managers for review.

A site visit log (Appendix G) serves as the official site documentation. This form officially documents all site activities. The field operator will use this form during each site visit to compare the instantaneous datalogger with the actual E-BAM and meteorological instrument conditions. All QA/QC and maintenance activities will be noted in a bound field notebook. All events pertinent to station operation will be recorded in the logbook.

It is important to have site documentation that includes written parameter values. This enables the data coordinator to use the site documentation as an additional tool for data validation.

The originals of the completed log sheets are given to the data coordinator and the copies are filed in the site notebook located at the field office. This makes it possible for the data coordinator to trace data anomalies, if necessary, and gives the field technician access to specific site information without having to verbally communicate with others while in the field.

At a minimum of once every week, a field technician visits the site and conducts an inspection, following (but not limited to) the items listed on the Site Visit Inspection Log. (The frequency of site visits may be decreased to every other week after the first month at the discretion of the project manager.) The field technician completes a log sheet during each inspection. All QA/QC and maintenance activities are noted on the log and additional pages are completed, if necessary. Any extraordinary activities or circumstances are noted in the COMMENTS area on the back of the form and continued on additional pages if necessary.

## C.3 Met One E-BAM Sampler

### C.3.1. Meteorological Instrument Inspection

During each site visit the wind speed sensor and wind direction vane are visually inspected. The field technician will verify that the cups are spinning and the vane is moving and note datalogger values that are recorded for wind speed and direction on the site visit log.

### C.3.2. Self Test

Performing the E-BAM 'SELF TEST' checks both the current operational status and resets any error codes that may exist. The E-BAM can generate an error code whenever an expected set point can not be maintained, as when a low flow condition occurs or when the filter tape runs out. **It is highly recommended to perform a 'SELF TEST' following the E-BAM installation, after the routine filter tape change, when troubleshooting and after correcting any problems.**

The E-BAM self test is performed by the following steps:

1. In the E-BAM menu screen, use the up and down arrows to select “SELF TEST”
2. Next, press the “SELECT” key.
3. The E-BAM display will indicate “SELF TEST RUNNING”. The SELF TEST will take several minutes and cannot be bypassed by the operator.
4. If all E-BAM checks pass, the display will read “SELF TEST COMPLETE, E-BAM FUNCTIONING PROPERLY”. If the display reads ‘FAILED’, remedy the problem and rerun ‘SELF TEST’. Most likely, the condition that caused the ‘SELF TEST’ failure will be indicated on the display.
5. At the completion of the E-BAM ‘SELF TEST’, press “CONTINUE” AND the E-BAM will begin sampling.

### **C.3.3. E-BAM Startup Calibration Information**

#### **C.3.3.1. Calibration Overview**

The E-BAM requires the calibration of the outside temperature sensor (OT), the internal pressure sensor and the volumetric flow controller. The ‘standard’ flow configuration is required for the E-BAM as configured with the external temperature sensor. By configuring the instrument in the standard mode, the E-BAM will control and calculate the operating flow using actual (local temperature and pressure) conditions and then will convert the actual flows to standard flows when storing the values in the data logger.

Met One E-BAM is calibrated using the following steps:

1. Leak Check
2. E-BAM flow audit/calibration
3. Outside temperature (OT) calibration/verification
4. Internal pressure calibration/verification
5. Actual flow calibration/verification

(All calibration information and data are recorded.)

#### **Apparatus for E-BAM Calibration**

1. NIST-traceable mass flow transfer standard
2. NIST-traceable temperature meter
3. NIST-traceable pressure meter
4. Tubing
5. Calibration forms

#### **C.3.3.2. Leak Check**

The LEAK CHECK procedure increases the vacuum 2.5 times normal operation to find problems before they can affect data.

Steps to perform a leak check:

1. Go to "LOAD TAPE" in the main menu and press "SELECT".
2. Insert a piece of thin rubber under the nozzle. Press "CONTINUE".
3. Go to the E-BAM PUMP TEST screen located in the MAIN MENU/FIELD CALIBRATION/PUMP TEST. Select "LEAK CHECK".
4. Flow rate should drop to under 1.5 LPM. If the flow is greater than 1.5 LPM perform necessary action to correct the leak.

#### **C.3.3.3. Flow Audit/Calibration**

A flow audit involves connecting a calibrated flow meter to the E-BAM inlet and comparing readings. If the error is greater than 2%, a flow calibration will have to be performed. (Note: temperature and pressure must be audited/calibrated before the flow.) Follow the steps below to perform a flow calibration:

#### **C.3.3.4. Outside Temperature Audit/Calibration**

Outside Temperature is a two point calibration, an ice bath is used for the low point and the ambient air temperature is used for the high point.

1. From the MENU screen use the up/down arrow keys to select FIELD CALIBRATION, press "SELECT".
2. The field calibration screen has seven selections. The first three (TEMPERATURE, PRESSURE, FLOW) are used in a flow audit/calibration. Actual Flow is calculated from all three components.
3. Select TEMPERATURE from the field calibration screen.
4. If the test is ambient select POINT:HIGH. If the test is an ice bath, select POINT:LOW. Allow an equilibrium period of at least 15 minutes.
5. Compare the reference temperature reading to the E-BAM reading on the LCD. If the readings are between 0.5 degrees Celsius, no recalibration is necessary.
6. To recalibrate, enter the reference temperature measurement into the REF:XX.X field and press calibrate.

#### **C.3.3.5. Internal Pressure Audit/Calibration**

1. The same protocol is used for PRESSURE with a single point calibration. Repeat the steps above using a reference pressure sensor. IF the pressure values are within 2 mmHg no recalibration is necessary.
2. To recalibrate, enter the reference pressure measurement into the REF:XX.X field and press calibrate.

#### **C.3.3.6. Actual Flow Audit/Calibration**

1. In the FIELD CALIBRATION screen select FLOW. (Note: temperature and pressure must be audited before flow)

2. For FLOW SP, three points are selectable: 14.0, 16.7, and 17.5 LPM. Select the desired flow, remove the PM10 inlet and place the reference flow audit device on the inlet tube. Allow an equilibrium period of at least 5 minutes.
3. Compare the reference flow to the E-BAM flow. If the flows are within 2% no recalibration is necessary.
4. To recalibrate, enter the reference flow measurement into the REF:XX.X field and press calibrate.

#### C.3.3.7. Clock/Timer Verification

Compare the instrument's clock with a known time source. Note: the E-BAM clock is set to GMT, this is +4 hours from EDT. The instrument's clock synchronizes each time a download is performed. Clock drift indicates that a problem may exist with the contact closure relay configuration. Diagnose and correct.

#### C.3.4. Sampling Startup

1. After the E-BAM is assembled and the power connected, it is ready to start operation. Open the hatch and note the display: ARE YOU READY TO START?
2. Press "YES". Verify that the correct date and time are displayed. If not, make the appropriate corrections. [To make changes, press EDIT, press OK to proceed to the next screen.]
3. Press "CONTINUE"  
**LOCATION** is a numeric descriptor location ID. (1-99)  
**FILTER ADVANCE** is how often the E-BAM will advance to a new spot of filter paper. (1,2,3,4,6,8,12, or 24 hours) For this project the filter advance is always set to 8 hours.  
**REAL-TIME AVE** is the averaging period for the real-time concentration value. (1,5,10,15,30, or 60 minutes) For this project the real-time average is set to 15 minutes.
4. Press "SAVE" to save changes and "CONTINUE" to proceed and not make changes.
5. If the nozzle packing material has not been removed, the display will ask you to remove it. Remove the stainless steel nozzle packing material from under the nozzle and press "CONTINUE".
6. The next screen checks for filter tape, if filter tape has not been installed, the unit will ask you to load it. After loading tape, press "CONTINUE".
7. Next the battery condition is verified. Press "CONTINUE" to proceed.
8. The E-BAM will next perform a self test. The self test takes several minutes and cannot be bypassed by the operator. If all E-BAM checks pass, the display will read "SELF TEST COMPLETE, E-BAM FUNCTIONING PROPERLY". If the display reads 'FAILED', remedy the problem and rerun 'SELF TEST'. Most likely, the condition that caused the 'SELF TEST' failure will be indicated on the display
9. The E-BAM will begin sampling.

10. The unit requires a 1-hour warm up period. Any data acquired in the first hour should be discarded.

### C.3.5. Sampling for metals and explosive residue

Samples for metals and explosive residue are collected weekly for the first month of the project and 48 hours after detonations occur for the duration of the project. For these samples, the filter tape must be carefully removed from the E-BAM and sent to an outside laboratory for analysis. Follow the steps below to collect samples for metals and explosive residue.

1. Prepare sample collection equipment. (Note: It is important that the samples are not exposed to metals or particulate matter.) Rinse the sample collection scissors with dilute nitric acid. Bring a clean sample bag, tape, chain-of-custody, sharpie and cleaned scissors to the site.
2. After completing the Site Visit Log, open the E-BAM hatch and manually advance the filter tape at least one full rotation of the collection spool. The unexposed tape will function as a sample blank.
3. In the main menu, enter "LOAD TAPE". This will open the nozzle. Lift the filter tape from the nozzle area. Cut the tape.
4. Remove the collection spool (left side) and wrap several lengths of filter tape around the spool to protect the sample and tape down the end.
5. Wrap the spool with bubble wrap and place into a sample collection bag.
6. Fill out chain-of-custody documentation and ship FedEx priority overnight to:  
  
Cooper Environmental Services  
10180 SW Nimbus Ave., Suite J6  
Portland, OR 97223  
  
(503) 624-5750
7. Wipe the inside of the E-BAM with isopropyl alcohol (IPA).
8. Install a new collection spool and secure the filter tape with adhesive tape to the new spool.
9. Annotate the filter tape with the sharpie and note date/time in the field log book.
10. Restart the E-BAM.

## C.4 Routine Maintenance and System Verification

### C.4.1. General Information

Perform the following checks on the E-BAM at the intervals specified in the service schedule. The checks may be performed more frequently but should be performed at least within the prescribed intervals. Document all service information and maintenance in a bound field notebook.

### **C.4.2. Daily Maintenance/System Verification**

Review station datalogger values for correct operation of the E-BAM.

### **C.4.3. Weekly Maintenance/System Verification**

1. Fill out Site Visit Log for each site.
2. Verify that the cups are spinning and the vane moving.
3. Check the E-BAM filter tape and replace when necessary. One roll of filter tape is 21 meters long and should last between 4 and 10 weeks.
4. Flow check: Perform an inlet flow verification to ensure a flow rate of 16.67 liters per minute ( $\pm 2$  percent). Remove only the PM10 inlet when measuring flow.
5. Download datalogger and submit to data manager.
6. Download error log and submit to data manager.

The weekly flow check can be performed while the E-BAM is in normal operating mode.

Remove only the PM10 inlet from the E-BAM inlet system. Affix either a mass flow measuring or volumetric flow measuring device onto the PM10 inlet and record the volumetric flow on the monthly quality control maintenance check sheet.

### **C.4.4. Monthly Maintenance /System Verification**

1. Record all maintenance in the bound field notebook.
2. Thoroughly clean the PM10 inlet.
3. Thoroughly clean the nozzle and vane.
4. Download and submit E-BAM data logger data.
5. Verify memory capacity.
6. Perform leak check.
7. Perform tape check.
8. Perform alarm check.

### **C.4.5. Semi-Annual Maintenance/System Verification**

1. Perform semi-annual E-BAM verification/calibration of the external ambient temperature, internal pressure, leak check and volumetric flow.
2. Check analog signal for external data logger.
3. Pump test.

### **C.4.6. Annual Maintenance/ System Verification**

1. Perform zero/span verification

## C.4.7. Bi-annual Maintenance/ System Verification

1. Replace pump.
2. PMT Cleaning.

## C.5 Maintenance Procedures

### C.5.1. General Information

Normal E-BAM maintenance requires keeping the E-BAM central unit dust free and inlet clean.

#### C.5.1.1. Sampler Maintenance

As with all monitoring equipment, the E-BAM should be kept clean and dust free.

#### C.5.1.2. PM10 Inlet Maintenance

The PM10 inlet requires removal from the inlet tube, disassembly and cleaning. The inlet is thoroughly cleaned every month. Disassemble the PM10 inlet and wipe clean with lint free cloth. Ensure that all 'O' ring surfaces are in excellent shape and are re-installed correctly. Replace 'O' rings when needed.

#### C.5.1.3. Nozzle Cleaning Procedure

In the main menu, enter "LOAD TAPE". This will open the nozzle. Lift the filter tape from the nozzle area. Use a Q-tip with isopropyl alcohol (IPA) and gently clean the nozzle and the 4 crosspiece sections of the vane. Replace the filter paper.

#### C.5.1.4. Pump Test

To perform a pump test, contact Met One (541) 471-7111 for specific instructions.

#### C.5.1.5. Zero/Span Check

The calibration plates are very fragile. Please handle with care. A set of unique calibration plates is supplied with each E-BAM unit. Verify the serial number of the plates before beginning the zero/span check.

1. Go to MAIN MENU, FIELD CALIBRATION, press "SELECT".
2. From the CALIBRATION menu, select "MEMBRANE TEST".
3. When you are ready to start press "START".
4. The E-BAM will guide you through the procedure. First the unit performs a zero count, next a zero on the zero plate, then a blank span count followed by a count on the span plate.
5. If the test fails, clean the detector and rerun the test. If the failure persists contact the project manager or Met One.

## C.5.2. Periodic Maintenance

### C.5.2.1. Wind Speed Sensor

Clean the cups as necessary and inspect for damage.

### C.5.2.2. Wind Direction Sensor

Clean the vane as necessary and inspect for damage.

### C.5.2.3. Inlet Heater Maintenance

The inlet heater maintenance includes both the filter relative humidity and temperature.

#### **Filter RH**

1. Go to MAIN MENU, FIELD CALIBRATION, FILTER RH.
2. Compare the reference RH reading to the E-BAM reading on the LCD. If the readings are  $\pm 2\%$ , no recalibration is necessary.
3. To recalibrate, enter the reference temperature measurement into the REF:XX.X field and press calibrate.

#### **Filter Temperature**

1. Go to MAIN MENU, FIELD CALIBRATION, FILTER TEMPERATURE.
2. Compare the reference temperature reading to the E-BAM reading on the LCD. If the readings are between 5 degrees Celsius, no recalibration is necessary.
3. To recalibrate, enter the reference temperature measurement into the REF:XX.X field and press calibrate.

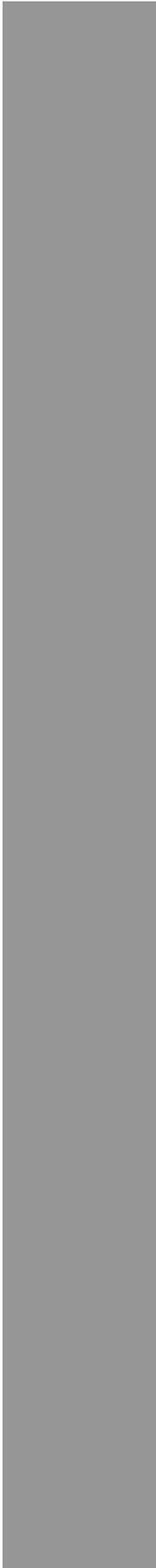
NOTE: The E-BAM has a built-in Mass Membrane Calibrator. The membrane is automatically moved into the Beta pathway to determine the 'mass' of the membrane each hour or when the filter tape advances. Each membrane has a factory verified mass and that value is stored in the E-BAM. When the hourly membrane calibration is made, the computed value is compared to the stored factory value to determine proper operation. The membrane must be withdrawn for normal measurements. Should the instrument fail to perform to specification an error is logged in memory and data is flagged.

Zero testing of blank filter paper is performed at the beginning and end of each sample period (in this case, hourly) to insure the stability of the measurement system. Zero testing is based on the ability of the E-BAM to hold a constant output when measuring blank filter paper. If the difference between the two values exceeds a preset limit a data error message is logged in the error log and the digital data value is marked.

APPENDIX D

## **Analytical Methods**

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# **STANDARD OPERATING PROCEDURE**

## **X-RAY FLUORESCENCE ANALYSIS OF PM<sub>2.5</sub> DEPOSITS ON TEFLON FILTERS**

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March 10, 1999

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## HISTORY OF REVISION

Revision	Date	Description of Change
1.0	3/10/99	New Document

# STANDARD OPERATING PROCEDURE

## X-RAY FLUORESCENCE ANALYSIS OF PM<sub>2.5</sub> DEPOSITS ON TEFLON FILTERS

### 1.0 SCOPE AND APPLICABILITY

This document addresses the application of energy dispersive X-ray fluorescence spectrometry (EDXRF) to the determination of elemental composition of PM<sub>2.5</sub> deposits on 47 mm diameter Teflon filters. The EDXRF technique is capable of quantitative analysis of elements with atomic numbers 11 (sodium) through 92 (uranium). This operating procedure discusses the determination of elements listed in Table 1.

**Table 1. Elements and their Atomic Symbols Analyzed by the EDXRF Method.**

Sodium (Na)	<b>Chromium (Cr)</b>	Strontium (Sr)	Lanthanum (La)
Magnesium (Mg)	<b>Manganese (Mn)</b>	Yttrium (Y)	Cerium (Ce)
<b>Aluminum (Al)</b>	<b>Iron (Fe)</b>	Zirconium (Zr)	Samarium (Sm)
<b>Silicon (Si)</b>	Cobalt (Co)	Niobium (Nb)	Europium (Eu)
Phosphorus (P)	<b>Nickel (Ni)</b>	Molybdenum (Mo)	Terbium (Tb)
<b>Sulfur (S)</b>	<b>Copper (Cu)</b>	Silver (Ag)	Hafnium (Hf)
<b>Chlorine (Cl)</b>	<b>Zinc (Zn)</b>	<b>Cadmium (Cd)</b>	Tantalum (Ta)
<b>Potassium (K)</b>	Gallium (Ga)	Indium (In)	Wolfram (W)
<b>Calcium (Ca)</b>	<b>Arsenic (As)</b>	Tin (Sn)	Iridium (Ir)
Scandium (Sc)	<b>Selenium (Se)</b>	Antimony (Sb)	Gold (Au)
<b>Titanium (Ti)</b>	<b>Bromine (Br)</b>	Cesium (Cs)	Mercury (Hg)
<b>Vanadium (V)</b>	Rubidium (Rb)	Barium (Ba)	<b>Lead (Pb)</b>

**Bold Elements: key elements of interest**

### 1.1 *Fundamentals of the X-ray Spectrometric Measurements*

The basis of X-ray fluorescence spectrometry is the interaction of X-ray tube-produced photons with elements in a PM<sub>2.5</sub> filter deposit. When elements in a PM<sub>2.5</sub> sample interact with photons from an X-ray tube, the photon causes the ejection of an inner shell electron of analyte elements in the deposit. Inner shell electron vacancies created by these interactions are filled by electrons from higher electron shells. These transitions result in emission of X-rays characteristic of the element. The energy of the characteristic X-ray is equal to the difference in the electron binding energies of the two electron shells involved in the transitions. Because the electron binding energies are a function of atomic number, the energy of the X-ray is characteristic of the element and used to qualitatively identify the elements present in a deposit. The number (intensity) of X-rays is used to quantify the amount of the element present by comparisons with standards. (Jenkins, 1975; Bertin, 1975, 1978; Birks, 1976; Tertiam, 1982; Williams, 1987, deVries, 1993).

A lithium-drifted silicon (Si(Li)) detector converts the energy of the characteristic X-ray photon into an electronic signal. The analyzer electronics further processes the signal and displays the X-ray energy spectrum on a personal computer. The computer software determines the energy and intensity of the characteristic X-rays and calculates the elemental concentrations.

## **1.2 Fundamentals of PM<sub>2.5</sub> Filter Analysis**

The analysis of PM<sub>2.5</sub> filter deposits is based on thin film assumptions; i.e., that the thickness of the deposit is small with respect to the analyte characteristic X-ray transmission thickness. In this case, the concentration of the analyte element is directly proportional to the concentration of analyte on a thin film standard. Thus, the concentration of analytes in an unknown PM<sub>2.5</sub> sampled is determined by first calibrating the spectrometer with thin film standards to determine sensitivity factors and then analyzing the unknown sample under identical excitation conditions as used to determine the calibration factors. In some cases, adjustments must be made to account for elemental analyte line interferences and deposit absorption corrections for light elements such as Na that might not meet the thin film assumption requirements.

## **2.0 OVERVIEW OF THE METHOD**

Filter samples are taken from archives, loaded into filter holders and logged into analysis data sheets. The samples and data sheets are taken to the X-ray fluorescence analyzer and the filters (in their holders) loaded into the analyzer in the same order as listed on the data sheet. The instrument is then prepared for analysis by inputting the filter identification numbers and sampling information. The PM<sub>2.5</sub> filter deposit analysis protocol is then initiated.

This protocol consists of the analysis of each filter along with a quality control sample six separate times using different excitation conditions. The specific excitation conditions have been previously optimized for the key elements of interest noted in Table 1. At the end of an analysis, the peak intensities are determined by a least-squares spectral deconvolution program that includes corrections for background subtraction, peak overlap and elemental interferences. The resulting net analyte peak intensities are multiplied by element sensitivity factors, and the elemental concentrations reported both as ng/cm<sup>2</sup> and ng/m<sup>3</sup>.

Instrument calibration is based on thin film Micromatter standards (Micromatter, 1998), and standards from the U. S. National Institutes of Standards and Technology (NIST). Recalibration of the instrument is required only when the quality control and/or quality assurance sample results fall outside their respective limits, or when the detector or tube is replaced. Typical recalibration frequency is on the order of once every six to twelve months. After the analysis, the samples are removed from their holders and returned to archives.

## 3.0 DEFINITIONS

- 3.1 **Accuracy.** The agreement between an experimentally determined value and the accepted reference value.
- 3.2 **Analyte.** Element of interest in the analysis.
- 3.3 **Analyte Line.** X-ray emission line used to quantify the analyte.
- 3.4 **Attenuation.** Reduction of amplitude or change in wave form due to energy dissipation or distance with time.
- 3.5 **Calibration.** The process of comparing a standard or instrument with one of greater accuracy (smaller uncertainty) for the purpose of obtaining quantitative estimates of the actual values of the standard being calibrated, the deviation of the actual value from a nominal value, or the difference between the value indicated by an instrument and the actual value.
- 3.6 **EDXRF.** Energy dispersive x-ray fluorescence
- 3.7 **Interference.** An undesired positive or negative output caused by a substance other than the one being measured.
- 3.8 **NIST.** National Institute of Standards and Technology.
- 3.9 **Precision.** The degree of mutual agreement between individual measurements, namely repeatability and reproducibility.
- 3.10 **Shape.** The actual shape of a background corrected pulse height spectrum for an element.
- 3.11 **Spectrum Deconvolution.** The process of assigning count rate intensity which includes background and interference corrections.
- 3.12 **SRM.** Standard Reference Materials.
- 3.13 **Standard.** A concept that has been established by authority, custom, or agreement to serve as a model or rule in the measurement of quantity or the establishment of a practice or procedure.
- 3.14 **Teflo™.** Trade name of a Teflon filter.
- 3.15 **Traceability to NBS.** A document procedure by which a standard is related to a more reliable standard verified by the National Institute of Standards Technology (NIST)
- 3.16 **Uncertainty.** An allowance assigned to a measured value to take into account two major components of error (1) the systematic error, and (2) the random error attributed to the imprecision of the measurement process.
- 3.17 **Unknown.** A sample submitted for analysis whose elemental concentration is not known.
- 3.18 **XRF.** X-ray fluorescence

## 4.0 HEALTH AND SAFETY WARNINGS

There are no specific health and safety concerns associated with this method under normal operation. However, good laboratory practices should be used at all times, and the following cautions should be noted:

- KevexSpectrace QuanX operators are protected from accidental exposure to X-rays by a lid lock and front and back door interlocks. If any problems arise with the X-ray on indicator light on the front panel or with the interlock system contact your KevexSpectrace Instruments service engineer.
- Beryllium windows are present on the X-ray tube and detector. Do not allow any sample or debris to fall on the window, and avoid puncturing it. If a window should break, note the beryllium metal is poisonous. Use extreme caution when collecting broken beryllium pieces. Consult a KevexSpectrace service engineer for advice on cleanup of broken windows.

## 5.0 ANALYTICAL CAUTIONS

This analysis assumes that PM<sub>2.5</sub> is uniformly deposited on the filter, and the filter position is the same as the standards. As such, it is important that care be taken when loading filters into sample holding rings that the deposit is not scraped, smudged, or smeared in any way. Care also needs to be taken to assure that the filter is placed flat in the filter holding rings, and that these holders rest flat on the instrument's sample positioning wheel. This latter condition can be tested by turning the ring about 90 degrees after placing the ring in the sample wheel.

## 6.0 INTERFERENCES AND INTENSITY CORRECTIONS

Potential sources of errors in this procedure include spectral interferences, deposit absorption and particle size effects (Cooper, 1976).

### 6.1 *Spectral Interferences.*

Spectral interferences with analyte line intensity determination include elemental peak overlap, escape peak and sum peak interference (Cooper, 1976). These interferences are accounted for in the CES-PM25 program that deconvolutes the entire spectrum for each excitation condition with a least squares algorithm which uses experimentally determined elemental shape functions. No action is required by the operator. The quality of the spectral deconvolution is evaluated in the QC/QA program.

### 6.2 *Attenuation*

Attenuation of X-ray intensities due to self absorption in the PM<sub>2.5</sub> deposit is relatively small for most elements. However, the intensities are adjusted for this attenuation in the CES-PM25 program based on the typical ambient composition of PM<sub>2.5</sub>. The calculations are described below in the calculation section and evaluated in the QC/QA program. No action is required of the operator for routine operation of the instrument (Cooper, 1987; Dzubay and Nelson, 1975; Dzubay, 1975).

### **6.3 Particle Size Effects.**

PM<sub>2.5</sub> particle size effects are substantially less than 1% for most elements. The correction for S in the PM<sub>2.5</sub> particle size range is typically 1% (Loo, 1977), and substantially less than this for higher atomic number elements. However, because of uncertainties in the particle size distribution of lower atomic number elements, no corrections are made for this effect. No action is required by the analysts.

## **7.0 PERSONNEL QUALIFICATIONS**

The procedure and instrument described should be used by a scientist with a minimum of 5 years of energy dispersive X-ray fluorescence analysis of atmospheric aerosols and its associated data processing; or should have an advanced degree in the physical sciences. CES's Quality Assurance Manager has a masters degree with over eight years experience, and our laboratory director has over thirty years experience in X-ray fluorescence analysis of aerosol deposits on filters.

## **8.0 INSTRUMENT**

The X-ray fluorescence analyzer used in this procedure is a KevexSpectrace QuanX. It is a floor standing, laboratory grade, energy dispersive X-ray fluorescence (EDXRF) spectrometer. The instrument utilizes a high flux rhodium [Rh] anode X-ray tube which is positioned to direct X-rays onto the sample. Standard equipment includes an electronically cooled lithium drifted silicon [Si(Li)] solid state X-ray detector, seven position X-ray filter wheel, and pulse processing electronics to communicate spectral data to a personal computer. The personal computer displays and processes spectral information, and outputs elemental concentration data. The analyzer contains the following major components:

- KevexSpectrace QuanX cabinet containing the detector, tube, sample changer and electronics for system control and signal processing.
- Personal computer with EDXRF.EXE software and CES-PM25 software.
- Vacuum pump and port for He flush.
- Pinter for analysis reports

### **Precautions**

- KevexSpectrace QuanX operators are protected from accidental exposure to X-rays by a lid lock and front and back door interlocks. If any problems arise with the X-ray on indicator light on the front panel or with the interlock system contact your Spectrace Instruments service engineer.
- Beryllium windows are present on the X-ray tube and detector. Do not allow any sample or debris to fall on the window avoid puncturing it. If a window should break note the beryllium metal is poisonous. Use extreme caution when collecting broken beryllium pieces. Consult a KevexSpectrace service engineer for advice on cleanup of broken windows.

## **Instrument Maintenance**

- Detector cooling. Electronically cooled detector requires free flow of air around the spectrometer cabinet. Additionally, a supply of uninterruptable 110V AC power is recommended.
- Keep debris and dust wiped from the chamber. Do not use forced air to dust the chamber because the detector beryllium window is fragile.

## **Site location**

The instrument must be placed so that free air flow around the instrument is supplied. Operate at temperatures of 50 -90 degrees Fahrenheit and relative humidity between 20 - 80% only. Power requirements are 115 V AC, 14 amps and a power conditioner/filter is recommended. Power both the Spectrace QuanX instrument and the personal computer from the same AC line.

## **9.0 INSTRUMENT CALIBRATION**

### **9.1 Standards**

Standards used for energy calibration consist of thin film standards from Micromatter (Micromatter, 1999) and NIST standards. The Micromatter standards are thin film vapor deposits on Nuclepore filters. The deposit mass is uniformly distributed across the filter surface and the mass determined gravimetrically. The NIST standards are SRM 3171a and SRM 3171b which have been deposited and dried on Nuclepore filters.

### **9.2 Calibration Frequency**

Calibration is performed only when the QC/QA limits are exceeded, or if there is a change in the excitation conditions, such as a change in the tube, detector, X-ray filters, or signal processor. Calibrations are typically valid for 6 months to a year.

### **9.3 Reference Peak Shape Spectra**

Acquisition of reference spectra is required only during the normal calibration. As long as no pulse processing modules have changed, these peak shape references remain valid. This procedure consists of placing the standards in the instrument and acquiring elemental spectra in a subdirectory labeled \REFS with each of the analytical conditions used. These reference spectra are used in the deconvolution or the unknown spectra.

### **9.4 Standardization of the Method**

Standardization of the QuanX uses thin film Micromatter and NIST certified reference materials standards. By using these materials, the EDXRF standardization then are traceable to NIST certificates. A multielement QC sample is run during the standardization and with every ten filter samples to insure instrument stability. The standardization procedure consists of the following steps:

- Set up standards file
- Set up reference and spectrum processing file
- Set up analysis menu
- Set up acquisition conditions
- Run analysis of standards
- Validate standardization with sensitivity curves

## **10.0 FILTER HANDLING AND STORAGE**

Upon receipt, filters are logged in and placed in archives. The appropriate chain of custody forms are completed and returned. The filters remain in archives until they are scheduled for analysis.

## **11.0 FILTER PREPARATION AND ANALYSIS**

### **11.1 *Preparation***

Filters scheduled for analysis are removed from archives and analysis data sheets filled out. The filters are transferred to numbered filter holders and placed in appropriate sequence in the QuanX. No other preparation of the samples is require.

### **11.2 *Instrument Setup***

After loading the filters and filter holders into the analysis chamber, the chamber lid is closed and the analysis conditions set. The analysis conditions are set the same as those used to calibrate and standardize the instrument. These conditions are retained in the software program CES-PM25, which automatically sets the conditions, analysis times, and chamber atmosphere when the program is called up. All that is required of the operator is to key in the appropriate filter identification codes. The program automatically establishes the same conditions as used for the standard procedure.

### **11.3 *Analysis***

Cross check the input data and filter identification codes. After making any required adjustments, start the analysis by initiating the ANALYZE program, and note the time of day the analysis started and the approximate time when the analysis should be complete.

### **11.4 *Operator Quality Control Check***

After the analysis is complete, perform the following QC checks.

- Note the actual time of analysis completion on the analysis data sheet next to the estimated completion time.
- Note any automatic analysis program flags on data sheet.

- Note the QC sample results on the analysis data sheet and update the QC plots. Notify supervisor if any of the QC elements is outside its control limits.
- Review run-time parameters for QC sample and note exceedances.
- Check sample identification codes with the filter positions in the analysis wheel.
- Make an electronic copy of the data and place in data archives.
- Remove the filters from the filter holders and cross check identification codes.
- Report any significant deviations to supervisor.

### 11.5 *Return Filters to Archives*

Return all filters to archives except one, randomly selected filter, which is place in the replicate analysis container for future replicate analysis.

## 12.0 DATA ACQUISITION AND CALCULATIONS

The CES-PM25 program reports measured elemental concentrations and uncertainties in both ng/cm<sup>2</sup>, and ng/m<sup>3</sup> if the volume information is provided. The elemental concentrations reported are based on the instruments analyte line intensity determinations using a least squares spectral deconvolution routine that yields net elemental intensities after background subtraction and removal of elemental spectral interferences (McCarthy, 1980). The calculations used to convert these analyte line intensities to concentration and uncertainty results are outlined in the following subsections.

### 12.1 *Analyte Concentration Calculation*

The aerial density of the i<sup>th</sup> element (M<sub>i</sub><sup>x</sup>) in ng/cm<sup>2</sup> is calculated from the following equation:

$$M_i^x = \frac{I_i^x \cdot C_i}{A_i^x} \quad \text{Equation 12.1}$$

where:

- I<sub>i</sub><sup>x</sup> = analyte peak intensity of the i<sup>th</sup> element in the unknown sample.
- A<sub>i</sub><sup>x</sup> = attenuation factor for the i<sup>th</sup> element in the unknown sample.
- C<sub>i</sub> = calibration factor.

The attenuation factors used are discussed in Cooper, 1987. The calibration factors are determined for each excitation condition using thin film standards and the following equation:

$$C_i = \frac{A_i^s \cdot M_i^s}{I_i^s} \quad \text{Equation 12.2}$$

where:

- $A_i^s$  = attenuation factor for the  $i^{\text{th}}$  element in the standard.  
 $M_i^s$  = aerial mass density of the  $i^{\text{th}}$  element in the standard.  
 $I_i^s$  = measured analyte peak intensity for the  $i^{\text{th}}$  element when analyzed under the same conditions as the unknown sample.

## 12.2 Calculation of Analyte Concentration Uncertainty

The uncertainty in the analyte concentration is given by the following equations where the  $\sigma$  prefix indicates standard deviation in the parameter:

$$\sigma M_i^x = M_i^x \left[ \left( \frac{\sigma C_i}{C_i} \right)^2 + \left( \frac{\sigma I_i^x}{I_i^x} \right)^2 \right]^{0.5} \quad \text{Equation 12.3}$$

And

$$\sigma C_i = C_i \left[ \left( \frac{\sigma M_i^s}{M_i^s} \right)^2 + \left( \frac{\sigma I_i^s}{I_i^s} \right)^2 \right]^{0.5} \quad \text{Equation 12.4}$$

$\frac{\sigma M_i^s}{M_i^s} = 0.02$  per manufacturers specifications and CES standard validation curves.

## 12.3 Attenuation Factor Calculations

The attenuation factors used in CES-PM25 are those discussed in Cooper, 1987; Dzubay, 1986; and Dzubay and Nelson, 1975.

## 12.4 Detection Limit Calculation

The detection limit,  $C_D$ , is defined as the smallest concentration that a particular measurement can detect. Using the definition developed by Currie (p. 289 of X-Ray Fluorescence Analysis of Environmental Samples, ed. By T.G. Dzubay, Ann Arbor Science, 1978), detection may be considered reliable when the probability of detecting a concentration  $C_D$  is sufficiently large (95%), while the probability of incorrectly detecting a blank is sufficiently small (5%). Using this definition of detection and the selected probabilities (95% and 5%), it can be shown that

$$C_D = 3.29 \sigma_0 \quad \text{Equation 12.5}$$

where  $\sigma_0$  is the standard deviation of the observed result when the true concentration is zero; i.e. standard deviation of the blank. The standard deviation is given by:

$$\sigma_0 = \sigma_b \sqrt{k} \quad \text{Equation 12.6}$$

where  $\sigma_b$  is the standard deviation of the blank ( $\sigma_b = B_i^{0.5}$ ) and k is 2 if paired observations (at equal counting times) of sample and blank are made and 1 if the background (or blank) is well characterized. The k term is derived from the uncertainty in the net peak intensity for the interference-free situation; i.e.,

$$\sigma_n^2 = \sigma_g^2 + \sigma_b^2$$

where  $\sigma_n$ ,  $\sigma_g$ , and  $\sigma_b$  are the uncertainties in the net, gross, and background (or blank) counts, respectively.

If the background (or blank) is well characterized, for example, by replicate measurements, then

$$\overline{\sigma_b^2} \ll \sigma_g^2 \cong \sigma_b^2 \quad \text{And} \quad \sigma_n^2 = \sigma_b^2 = B_I \quad \text{Equation 12.7}$$

where  $\overline{\sigma_b}$  is the mean uncertainty as determined by replicate measurements, and  $B_I$  refers to the background (or blank) counting rate determined over the same counting interval as the gross sample counts or the mean counts as determined by repeated measurements over the same counting interval as used to measure a sample.

If the background is determined by a paired measurement equal to the time interval used to measure a sample, then  $\sigma_g^2 \cong \sigma_b^2$  and

$$\sigma_o^2 = 2\sigma_b^2 = 2B_I$$

CES assumes that the blank standard deviation can be minimized through replicate measurements of the blank and uses a  $3.29 B_i^{0.5}$  as the interference-free minimum detection limit defined by the 95%/5% probabilities. However the detection limits listed in this document are one sigma limits to be consistent with those used by the EPA in IO-3.3. These detection limits are listed in Table 2 for the excitation conditions noted in Table 3.

### 13.0 COMPUTER PROGRAMS AND DATA REPORTING

The analysis of samples and reporting of results is performed with the CES-PM25 program. This program sets the analysis conditions, controls the analysis of the filter samples and calculates the analyte peak intensities and elemental concentrations. The calculations performed by this program are outline above in Section 12.0. Examples or the reports provided by this program are provided in Tables 4 and 5.

**Table 2. One Sigma Teflo Filter Interference Free  
Detection Limits<sup>a</sup>**

Element	Limit (ng/m <sup>3</sup> ) <sup>b</sup>	Element	Limit (ng/m <sup>3</sup> ) <sup>b</sup>	Element	Limit (ng/m <sup>3</sup> ) <sup>b</sup>
Na	6	Ni	0.5	Sn	9
Mg	3	Cu	0.5	Sb	10
Al	2	Zn	0.5	Cs	25
Si	2	Ga	0.6	Ba	30
P	2	As	0.7	La	40
S	2	Se	0.7	Ce*	4
Cl	2	Br	0.6	Sm*	3
K	2	Rb	0.6	Eu*	3
Ca	2	Sr	0.6	Tb*	3
Sc	1	Y	0.7	Hf*	2
Ti	1	Zr	0.8	Ta*	3
V	0.5	Nb	0.8	Ir*	1
Cr	0.8	Mo	2	W*	4
Mn	0.8	Ag	7	Au*	1
Fe	0.6	Cd	5	Hg*	1
Co	0.5	In	7	Pb*	1

a) These limits are provided only for comparison purposes.

Concentrations detectable at the 95% confidence level are 3.29 times those listed in this table (Currie/Dzubay,1978).

b) Assumes 1.74 m<sup>3</sup>/cm<sup>2</sup> sampled.

\* L<sub>α</sub> analyte line

**TABLE 3. Summary of Analysis Conditions**

(Cond.) Filter	(1) None	(2) Cellulose	(3) Al	(4) Thin Pd	(5) Thick Pd	(6) Cu
kV	5	11	15	30	35	50
Na	X					
Mg	X					
Al	X	X				
Si	X	X				
P	X	X				
S	X	X	X			
Cl	X	X	X			
K	X	X	X			
Ca	X	X	X			
Sc		X	X			
Ti		X	X	X		
V		X	X	X		
Cr			X	X		
Mn			X	X		
Fe			X	X		
Co				X		
Ni				X	X	
Cu				X	X	
Zn				X	X	
Ga				X	X	
Ge				X	X	
As				X	X	
Se				X	X	
Br				X	X	X
Rb				X	X	X
Sr				X	X	X
Y				X	X	X
Zr				X	X	X
Mo				X	X	X
Rh						X
Pd						X
Ag						X
Cd						X
Sn						X
Sb						X
Te						X
I						X
Cs						X
Ba						X
La						X
W				X	X	X
Au				X	X	X
Hg				X	X	X
Pb				X	X	X

## 14.0 DATA AND RECORDS MANAGEMENT

All data including analysis conditions are electronically stored on floppy disks and a Zip drive. The floppy disk is stored along with the hard copy in report archive files. The Zip drive data sets are stored in separate files and used for preparing client summary reports.

## 15.0 QUALITY CONTROL AND ASSURANCE

Procedures used to maintain and assure that quality goals follow CES's Quality Management Plans. The procedures are summarized in the following subsections.

### 15.1 *General Procedures*

General quality is controlled by precise control of filter handling, calibration, and instrumental stability as detailed below.

#### 15.1.1 *Filter Handling*

The filters are loaded and unloaded into specially machined holders. Filter loading is done in a clean area and the filters themselves are handled with forceps, out of the analysis and deposit area. The loaded filters are transported to and from the analyzer in covered sample cases, and the filter holders are cleaned between each use. These procedures prevent the possibility of sample contamination and disturbance of the deposit.

To prevent confusion in identification of the samples when they are out of their ID coded filter containers, a sample position number versus ID number relationship is used. This relationship is established by the XRF LOG before the filters are removed from their containers, and it is verified after each handling step in the procedure.

In addition, one out of every ten samples are resubmitted for analysis at a later date. These are reanalyzed and a replicate report is generated. Control limits for precision vary based on elemental concentration in the sample. Acceptable precision at samples approaching instrumental detection limits is  $\pm 50\%$ . Otherwise, a precision goal of  $\pm 10\%$  has been set.

#### 15.1.2 *Instrument Stability*

For each XRF analysis batch of ten (10) samples, a quality control standard is analyzed. Measured concentrations of the quality control standard, which contains Al, Ni, Pb, and Cd are compared with concentrations determined at the time of calibration. Precision must be within  $\pm 5\%$ , or all samples of that run must be reanalyzed. The average value of the quality control standard is computed over the period of time of interest. Once a week the NIST standards are rerun to confirm calibration. The average of these runs are also reported. If any experimental values differ from the certified concentration by more than 3 sigma, a detailed investigation of the calibration is performed. The long term stability of the instrument is monitored by plotting

the results for the five QC elements over a period of weeks. Systematic shifts in this analysis alert the operator that the instrument needs to be recalibrated.

Most of the key elements are measured under more than one of the excitation conditions normally used for each run. Results for these elements are compared for each of the excitation conditions under which they are measured. If agreement is outside two times the calculated uncertainties, the sample must be reanalyzed.

### **15.2      *Quality Control Standard***

The quality control standard is a multi-element Micromatter standard containing nominally 10  $\mu\text{g}/\text{cm}^2$  of Al, V, Ni, Cd and Pb.

### **15.3      *Run-Time Quality Control***

Run-time quality control parameters such as peak centroid, resolution, and baseline are monitored and controlled by the instrument. The effects of possible deviations not controlled by the instrument are noted in variations of the QC sample results which cover the entire energy range used in the analysis.

### **15.4      *Determination of Acceptable QC Precision Limits***

Acceptability of analytical precision limits is determined by the operator at the end of each analysis by comparing the results for each element in the QC sample with the results obtained at the time of calibration. These results are also input to control charts that are reviewed weekly by the laboratory supervisor.

### **15.5      *Quality Assurance with Standard Reference Material***

Thin film standards are no longer available from NIST. As such, NIST liquid standards evaporated on thin films are used to provide NIST traceable results. These samples are analyzed weekly.

### **15.6      *Acceptance Criteria and Procedures for Corrective Action***

These procedures are described above in Subsection 15.1.

### **15.7      *Field Data Entry Quality Checks***

The field data entered into the analysis log and subsequently entered into the analyzer at the time of analysis is confirmed at the end of the analysis by comparing the field data on the hard copy of the analytical report when the samples are returned to their original containers.

### **15.8      *Control Charts***

Control charts are prepared for the analytical results for each of the elements in the control sample and the NIST SRM sample. In addition, replicate analysis control charts are prepared and monitored for Si, S, Ca, Fe, Zn and Pb.

## **15.9 Self-Consistency Checks**

Calibration of the instrument is performed by analyzing thin film standards prepared by Micromatter, Inc. The aerial density of these standards is determined gravimetrically. These standards are verified through graphical correlation techniques. To insure stability of the instrument, a quality control standard is run at the time of calibration with every ten samples and during analysis.

Interlaboratory comparisons and NIST SRM standards are used periodically to check the accuracy of the instrument calibration.

The internal consistency of these standards is evaluated for each excitation condition by graphical evaluation of the relationship between the standards.

**Table 4. Example of Data Report Format**

## CES-PM25 Elemental Concentrations Data Report

Lab ID: C21-A1375

Client ID: HS022199

Receipt Date: 20599

Analysis Date/Time: 022899/1435

Sample Volume: 23.75 m<sup>3</sup>Deposit Area: 6.57 cm<sup>2</sup>

Valid QC Sample? Yes

Operator: JAC

Element	Flags	Concentration (ng/m <sup>3</sup> )		Concentration (ng/cm <sup>2</sup> )	
NA		320.0	+/- 100.7	1156.7	+/- 27.4
MG		643.6	+/- 104.6	2326.7	+/- 33.5
AL		248.2	+/- 102.3	897.1	+/- 238.1
SI		266.8	+/- 32.7	964.3	+/- 263.0
P		15.5	+/- 26.5	56.0	+/- 15.3
S		3661.7	+/- 268.2	13236.7	+/- 109.5
CL		1071.2	+/- 92.0	3872.5	+/- 32.7
K		288.5	+/- 21.0	1042.8	+/- 7.5
CA		46.0	+/- 5.7	166.3	+/- 14.6
SC		4.1	+/- 6.4	14.7	+/- 2.4
TI		23.6	+/- 7.3	85.3	+/- 12.4
V		4.0	+/- 2.9	14.3	+/- 1.6
CR		2.5	+/- 1.0	9.0	+/- 1.6
MN		13.2	+/- 2.2	47.7	+/- 1.7
FE		280.3	+/- 30.0	1013.1	+/- 88.4
CO		3.7	+/- 1.6	13.5	+/- 1.6
NI		3.5	+/- 1.3	12.6	+/- 0.3
CU		18.4	+/- 2.8	66.7	+/- 1.7
ZN		216.1	+/- 12.7	781.1	+/- 6.0
GA		3.5	+/- 1.2	12.7	+/- 0.0
GE		4.3	+/- 1.3	15.5	+/- 0.0
AS		34.8	+/- 3.4	125.9	+/- 1.9
SE		5.0	+/- 0.8	18.2	+/- 0.8
BR		27.8	+/- 3.6	100.6	+/- 1.1
RB		1.4	+/- 0.7	5.1	+/- 0.4
SR		3.7	+/- 1.3	13.2	+/- 0.5
Y		18.6	+/- 8.4	67.2	+/- 2.8
ZR		4.2	+/- 5.8	15.2	+/- 3.1
MO		11.0	+/- 4.3	39.6	+/- 2.4
RH		0.0	+/- 3.0	0.0	+/- 2.2
PD		0.0	+/- 2.9	0.0	+/- 2.6
AG		0.0	+/- 3.6	0.0	+/- 2.9
CD		10.5	+/- 5.1	38.1	+/- 1.1
SN		81.5	+/- 9.2	294.4	+/- 3.1
SB		0.0	+/- 10.0	0.0	+/- 0.8
TE		3.7	+/- 6.0	13.2	+/- 5.8
I		35.8	+/- 10.6	129.2	+/- 5.3
CS		0.0	+/- 14.8	0.0	+/- 5.8
BA		0.0	+/- 11.6	0.0	+/- 9.3
LA		0.0	+/- 47.3	0.0	+/- 27.0
W		0.0	+/- 2.4	0.0	+/- 2.1
AU		0.0	+/- 1.9	0.0	+/- 0.3
HG		0.0	+/- 2.8	0.0	+/- 1.1
PB		305.8	+/- 27.0	1105.5	+/- 9.5

Report Date: 022999

**Table 5. Example of Replicate Sample Analysis Report Format**

## CES-PM25 Elemental Concentrations Data Report

Lab ID:	C21-A1375	Replicate ID:	C21-A1375R
Receipt Date:	250599	Analysis Date/Time:	022899/01435
Sample Volume:	23.75 m3	Deposit Area:	6.57 cm2
Valid QC Sample?	Yes	Operator:	JAC

Element	Flags	Original (ng/cm <sup>2</sup> )	Flags	Replicate (ng/cm <sup>2</sup> )	D>3U
NA		320.0 +/- 100.7		344.9 +/- 91.6	
MG		643.6 +/- 104.6		695.1 +/- 100.1	
AL		248.2 +/- 102.3		224.6 +/- 94.6	
SI		266.8 +/- 32.7		242.2 +/- 30.4	
P		15.5 +/- 26.5		16.4 +/- 24.7	
S		3661.7 +/- 268.2		3811.8 +/- 243.0	
CL		1071.2 +/- 92.0		982.3 +/- 89.6	
K		288.5 +/- 21.0		289.3 +/- 19.3	
CA		46.0 +/- 5.7		46.3 +/- 5.3	
SC		4.1 +/- 6.4		3.7 +/- 6.1	
TI		23.6 +/- 7.3		23.4 +/- 7.0	
V		4.0 +/- 2.9		4.2 +/- 2.7	
CR		2.5 +/- 1.0		2.7 +/- 0.9	
MN		13.2 +/- 2.2		12.2 +/- 2.0	
FE		280.3 +/- 30.0		262.6 +/- 29.3	
CO		3.7 +/- 1.6		4.1 +/- 1.6	
NI		3.5 +/- 1.3		3.5 +/- 1.1	
CU		18.4 +/- 2.8		20.3 +/- 2.6	
ZN		216.1 +/- 12.7		230.1 +/- 11.9	
GA		3.5 +/- 1.2		3.2 +/- 1.1	
GE		4.3 +/- 1.3		4.5 +/- 1.3	
AS		34.8 +/- 3.4		34.6 +/- 3.2	
SE		5.0 +/- 0.8		5.2 +/- 0.8	
BR		27.8 +/- 3.6		25.8 +/- 3.3	
RB		1.4 +/- 0.7		1.3 +/- 0.7	
SR		3.7 +/- 1.3		3.9 +/- 1.2	
Y		18.6 +/- 8.4		17.5 +/- 7.5	
ZR		4.2 +/- 5.8		4.6 +/- 5.4	
MO		11.0 +/- 4.3		10.8 +/- 3.9	
RH		0.0 +/- 3.0		0.0 +/- 2.9	
PD		0.0 +/- 2.9		0.0 +/- 2.7	
AG		0.0 +/- 3.6		0.0 +/- 3.4	
CD		10.5 +/- 5.1		10.2 +/- 4.8	
SN		81.5 +/- 9.2		78.1 +/- 8.5	
SB		0.0 +/- 10.0		0.0 +/- 9.9	
TE		3.7 +/- 6.0		3.9 +/- 5.9	
I		35.8 +/- 10.6		32.8 +/- 9.8	
CS		0.0 +/- 14.8		0.0 +/- 13.5	
BA		0.0 +/- 11.6		0.0 +/- 11.3	
LA		0.0 +/- 47.3		0.0 +/- 44.5	
W		0.0 +/- 2.4		0.0 +/- 2.2	
AU		0.0 +/- 1.9		0.0 +/- 1.8	
HG		0.0 +/- 2.8		0.0 +/- 2.7	
PB		305.8 +/- 27.0		291.1 +/- 24.7	

Report Date: 022999

D=Replicate Differen U = Uncertainty in difference

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

**Compendium Method IO-3.3**

**DETERMINATION OF METALS  
IN AMBIENT PARTICULATE  
MATTER USING  
X-RAY FLUORESCENCE (XRF)  
SPECTROSCOPY**

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

June 1999

## Method IO-3.3

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

**Method IO-3.3**  
**Determination of Metals in Ambient Particulate Matter Using**  
**X-Ray Fluorescence (XRF) Spectroscopy**

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## **Chapter IO-3 CHEMICAL SPECIES ANALYSIS OF FILTER-COLLECTED SPM**

### **Method IO-3.3 DETERMINATION OF METALS IN AMBIENT PARTICULATE MATTER USING X-RAY FLUORESCENCE (XRF) SPECTROSCOPY**

#### **1. Scope**

**1.1** During a span of more than two decades, the U. S. Environmental Protection Agency (EPA) has developed and applied x-ray fluorescence (XRF) to the analysis of ambient and source aerosols using both energy and wavelength dispersive spectrometers. Inorganic Compendium Method IO-3.3 briefly describes the agency's experience with XRF and informs the reader of its capability in elemental aerosol analysis and attempts to give a brief account of what is involved in its application. The procedures described have been in a continual state of evolution beginning with those in use on a special purpose spectrometer designed by Lawrence Berkeley Laboratory (LBL) and eventually applied to a commercially available instrument manufactured by Kevex. It is for the Kevex spectrometer to which this method applies.

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

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

**1.4** The most commonly used device for sampling TSP in ambient air is the high-volume sampler, which consists essentially of a blower and a filter, and which is usually operated in a standard shelter to collect a 24-hour sample. The sample is weighed to determine concentration of TSP and is usually analyzed chemically to determine concentration of various inorganic compounds. When EPA first regulated TSP, the National Ambient Air Quality Standard (NAAQS) was stated in terms of SPM with aerodynamic particle size of < 100 Fm captured on a filter as defined by the high-volume TSP sampler. Therefore, the high-volume TSP sampler was the reference method. The method is codified in 40CFR50, Appendix B.

**1.5** More recently, research on the health effects of TSP in ambient air has focused increasingly on particles that can be inhaled into the respiratory system, i.e., particles of aerodynamic diameter of < 10 Fm. These particles are referred to as PM<sub>10</sub>. It is now generally recognized that, except for toxic materials, it is this PM<sub>10</sub> fraction of the total particulate loading that is of major significance in health effects. The reference method for PM<sub>10</sub> is codified in 40CFR50, Appendix J and specifies a measurement principle based on extracting an ambient air sample with a powered sampler that incorporates inertial separation of PM<sub>10</sub> size range particles and collection of these particles on a filter for a 24-hour period. Again, the sample is weighed to determine concentration of PM<sub>10</sub> and is usually analyzed chemically to determine concentration of various inorganic compounds.

**1.6** Further research now strongly suggests that atmospheric particles commonly occur in two distinct modes, the fine ( $< 2.5 \mu\text{m}$ ) mode and the coarse (2.5 to 10.0  $\mu\text{m}$ ) mode. The fine or accumulation mode (also termed the respirable particles) is attributed to growth of particles from the gas phase and subsequent agglomeration, whereas the coarse mode is made up of mechanically abraded or ground particles. Because of their initially gaseous origin, the fine range of particle sizes includes inorganic ions such as sulfate, nitrate, and ammonium as well as combustion-form carbon, organic aerosols, metals, and other combustion products. Coarse particles, on the other hand, normally consist of finely divided minerals such as oxides of aluminum, silicon, iron, calcium, and potassium. Samplers which separate SPM into two size fractions of 0-2.5  $\mu\text{m}$  and 2.5-10  $\mu\text{m}$  are called dichotomous samplers. In 1997, the EPA promulgated a new standard with fine particles. The new  $\text{PM}_{2.5}$  standard replaced the previously NAAQS for  $\text{PM}_{10}$ .

**1.7** Airborne particulate materials retained on a sampling filter, whether TSP,  $\text{PM}_{10}$ ,  $\text{PM}_{2.5}$ , or dichotomous size fractions, may be examined by a variety of analytical methods. This method describes the procedures for XRF analysis as the analytical technique. The XRF method provides analytical procedures for determining concentration in  $\text{ng}/\text{m}^3$  for 44 elements that might be captured on typical filter materials used in fine particle or dichotomous sampling devices. With the sample as a thin layer of particles matrix effects substantially disappear so the method is applicable to elemental analysis of a broad range of particulate material. The method applies to energy dispersive XRF analysis of ambient aerosols sampled with fine particle ( $< 2.5 \mu\text{m}$ ) samplers, dichotomous and VAPS (versatile air pollution sampler) samplers with a 10  $\mu\text{m}$  upper cut point and  $\text{PM}_{10}$  samples.

**1.8** The analysis of ambient aerosol samples captured on filterable material should be performed by a scientist that has been trained in energy dispersive x-ray fluorescence spectroscopy and its associated data processing system. The training should be performed by a scientist with an advance degree in the physical sciences with a minimum of 5 years experience in x-ray spectroscopy.

## **2. Applicable Documents**

### **2.1 ASTM Documents**

- D4096 Application of High Volume Sample Method For Collection and Mass Determination of Airborne Particulate Matter.
- D1356 Definition of Terms Related to Atmospheric Sampling and Analysis.
- D1357 Practice For Planning the Sampling of the Ambient Atmosphere.

## 2.2 U.S. Government Documents

- U.S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I: A Field Guide for Environmental Quality Assurance*, EPA-600/R-94/038a.
- U.S. Environmental Protection Agency, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition)*, EPA-600/R-94/038b.
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- "Reference Method for the Determination of Particulate Matter in the Atmosphere (PM<sub>10</sub> Method)," *Code of Federal Regulations*, 40 CFR 50, Appendix J.
- "1978 Reference Method for the Determination of Lead in Suspended Particulate Matter Collected From Ambient Air." *Federal Register* 43 (194):46262-3.
- *Test Methods for Evaluating Solid Waste, Method 9022*, EPA Laboratory Manual, Vol. 1-A, SW-846.

## 2.3 Other Documents

- KeveX XRF TOOLBOX II Reference Manual
- KeveX 771-EDX Spectrometer User's Guide and Tutorial

## 3. Summary of Method

*[Note: This method was developed using the KeveX spectrometer. EPA has experience in the use of the KeveX spectrometer associated with various field monitoring programs involving analysis of filterable particulate matter for metals over the last two decades. The use of other manufacturers of x-ray spectrometers should work as well as long as the quality assurance and quality control specifications identified in Sections 12 through 14 of Method 10-3.3 are met. However, modifications to Compendium Method IO-3.3 procedures may be necessary if another commercial x-ray spectrometer is used.]*

The method described is x-ray fluorescence applied to PM<sub>10</sub>, fine (< 2.5 μm) and coarse (2.5-10 μm) aerosols particles captured on membrane filters for research purposes in source apportionment. The samplers which collect these particles are designed to separate particles on their inertial flow characteristics producing size ranges which simplify x-ray analysis. The instrument is a commercially available KeveX EDX-771 energy dispersive x-ray spectrometer which utilizes secondary excitation from selectable targets or fluorescers and is calibrated with thin metal foils and salts for 44 chemical elements. Spectra are acquired by menu-driven procedures and stored for off-line processing. Spectral deconvolution is accomplished by a least squares algorithm which fits stored pure element library spectra and background to the sample spectrum under analysis. X-ray attenuation corrections are tailored to the fine particle layer and the discrete coarse particle fraction. Spectral interferences are corrected by a subtractive coefficient determined during calibration. The detection limits are determined by propagation of errors in which the magnitude of error from all measured quantities is calculated or estimated as appropriate. Data are reported in ng/m<sup>3</sup> for all samples. Comprehensive quality control measures are taken to provide data on a broad range of parameters, excitation conditions and elements.

**4. Significance**

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

**4.2** Inhalable ambient air particulate matter ( $< 10 \mu\text{m}$ ) can be collected on Teflon® filters by sampling with a dichotomous sampler and analyzed for specific metals by X-ray fluorescence. The dichotomous sampler collects particles in two size ranges - fine ( $< 2.5 \mu\text{m}$ ) and coarse ( $2.5\text{-}10 \mu\text{m}$ ). The trace element concentrations of each fraction are determined using the nondestructive energy dispersive X-ray fluorescence spectrometer.

**4.3** The detectability and sensitivity of specific elements may vary from instrument to instrument depending upon X-ray generator frequency, multichannel analyzer sensitivity, sample interferences, etc.

## **5. Definitions**

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

**5.1 Accuracy.** The agreement between an experimentally determined value and the accepted reference value.

**5.2 Attenuation.** Reduction of amplitude or change in wave form due to energy dissipation or distance with time.

**5.3 Calibration.** The process of comparing a standard or instrument with one of greater accuracy (smaller uncertainty) for the purpose of obtaining quantitative estimates of the actual values of the standard being calibrated, the deviation of the actual value from a nominal value, or the difference between the value indicated by an instrument and the actual value.

**5.4 10  $\mu\text{m}$  Dichotomous Sampler.** An inertial sizing device that collects suspended inhalable particles ( $< 10 \mu\text{m}$ ) and separates them into coarse ( $2.5\text{-}10 \mu\text{m}$ ) and fine ( $< 2.5 \mu\text{m}$ ) particle-size fractions.

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

**5.6 Filter.** A porous medium for collecting particulate matter.

**5.7 Fluorescent X-Rays (Fluorescent Analysis).** Characteristic X-rays excited by radiation of wavelength shorter than the corresponding absorption edge.

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

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

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

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

**5.12 Traceability to NIST.** A documented procedure by which a standard is related to a more reliable standard verified by the National Institute of Standards and Technology (NIST).

**5.13 Uncertainty.** An allowance assigned to a measured value to take into account two major components of error: (1) the systematic error, and (2) the random error attributed to the imprecision of the measurement process.

**5.14 Chi-square.** A statistic which is a function of the sum of squares of the differences of the fitted and measured spectrum.

**5.15 Fluorescer.** A secondary target excited by the x-ray source and in turn excites the sample.

**5.16 FWHM.** Full width at half maximum, a measure of spectral resolution.

**5.17 NIST.** National Institute of Standards and Technology.

**5.18 Shape.** The actual shape of a background corrected pulse height spectrum for an element.

**5.19 SRMs.** Standard reference materials.

**5.20 Teflo®.** Trade name of a Teflon filter.

**5.21 Unknown.** A sample submitted for analysis whose elemental concentration is not known.

**5.22 XRF.** X-ray fluorescence.

## **6. Description of Spectrometer**

The x-ray analyzer is a Kevex EDX-771 energy dispersive spectrometer with a 200 watt rhodium target tube as an excitation source. The machine has multiple modes of excitation including direct, filtered direct, and secondary which utilizes up to 7 targets or fluorescers. To minimize radiation damage to delicate aerosol samples only the secondary mode is used. Table 1 provides a listing of the fluorescers and the elements which they excite associated with energy dispersive spectrometers. Analysis atmospheres are selectable with choices of helium, vacuum or air; helium is used for all targets except Gd where air is employed because it gives a lower background. The detector is cryogenically cooled lithium-drifted silicon with a 5  $\mu\text{m}$  Be window and a resolution of 158 eV at Fe K $\alpha$  and comes with two manually changeable collimators. A 16 position rotating wheel accommodates the samples and provides sample changing.

The machine is operated by procedure files (or programs) written in Kevex's proprietary Job Control Language (JCL) which runs in a Windows 3.1 environment and provides setting of the analytical conditions and data acquisition. Using the JCL language, procedures have been written in-house to perform all the

functions necessary to acquire spectra and to assign to them file names in a structured manner to facilitate future spectral processing. These procedures are invoked in menu form.

## **7. Caveats**

**7.1** The type of samplers mentioned in Section 1.7 must be operated in accordance with Inorganic Compendium Method IO-2.2 Sampling for Suspended Particulate Matter in Ambient Air Using a Dichotomous Sampler, or severe errors in x-ray analysis may occur. For example, errors in flow rate will not only give erroneous volumes but will cause a more serious condition of altering the cut points upon which the coarse particle x-ray attenuations are based. If samples are intended for x-ray analysis then the sampling protocol must conform to the constraints inherent within this method. Furthermore, the type of filter on which the sample is collected is very important. In general, thin membrane filters (Teflo® and Nuclepore®) are required so that the background is low and penetration of particles into the matrix of the filter is small. Thick depth filters such as quartz or glass fiber not only have high background but also allow particles to penetrate into the matrix of the filter - a condition which the spectral processing program cannot accommodate.

**7.2** Some internal contaminations consisting of Sn, Ni, Cu and Fe are present which sometimes appear in blanks. Routine analysis of blanks with samples will give the magnitude of the correction necessary to compensate for this.

**7.3** In general the elements analyzed by the Gd fluorescer have higher detection limits than the other fluorescers (see Table 2). The reason for this is due to limitations in the upper voltage limit of the x-ray tube power supply and the use of rhodium instead of a heavier element such as tungsten as a target material for the x-ray tube. As a secondary consequence of this, there are also higher detection limits for many of the elements below chromium because they overlap the elements analyzed by Gd.

**7.4** An inherent problem with a helium atmosphere is the diffusion of He through the detector window causing detector degradation and necessitating replacement. A lifetime of 3 to 4 years is expected.

**7.5** Due to an x-ray leak around the anode area of the x-ray tube the head must be shielded with additional lead cladding to prevent unwanted excitation of internal parts. This leak posed no threat to personnel but caused high background when operating at the maximum voltage. The additional shielding proved very effective at improving detection limits.

**7.6** Experience with wavelength dispersive spectrometers (WDXRF) has shown good agreement with energy dispersive instruments (EDXRF) over a broad range of elements. In spite of this agreement and the simpler spectral processing requirements of wavelength machines the preference remains with energy dispersive equipment for a variety of reasons. The very low power tubes in EDXRF machines leaves the sample intact and unaffected whereas in WDXRF the high power excitation embrittles the filter itself after 15 - 30 min exposure raising the possibility of altering particle morphology. This is a concern if electron microscopy is considered. Also, the vacuum environment, necessary for WDXRF, causes loss of some volatile materials.

**8. Sample Preparation**

**8.1** Sample preparation begins with the correct operation of the samplers employed. Inorganic Compendium Method IO-2.2, Sampling for Suspended Particulate Matter in Ambient Air Using a Dichotomous Sampler, covering the operation of the samplers in the field and subsequent collection of ambient air particles on 37-mm Teflon® filter for XRF analysis. One of the greatest advantages of analyzing aerosols by XRF is that the sample can, in theory, be collected in a manner most advantageous to XRF by sampling for a duration that produces an ideal mass loading on the filter. An approximate maximum target mass is about 100 µg/cm<sup>2</sup> although much less is often collected in many environments.

**8.2** The types of filters used for aerosol sampling are 37-mm or 47-mm Teflo® with a pore size of 2 microns and, if electron microscopy is planned for the coarse fraction, then a 0.6 micron pore size Nuclepore® filter is used. The sample should be collected on the side of the Teflo® filter with the supporting ring to maintain the proper distance between the sample and detector during analysis. A properly collected sample will be a uniform deposit over the entire collection area of at least 25-mm in diameter. Samples which are not uniformly deposited over the whole collection area are not quantitatively analyzable.

**8.3** All filter samples received for analysis are removed with tweezers from their container and are checked for any invalidating conditions such as holes, tears, or a non-uniform deposit which would prevent quantitative analysis. If such a condition is found the sample is noted as invalid on the XRF data entry form; data from such samples are not reported. Teflo® filters are easily handled because of the supporting ring, however, Nuclepore® filters must have a supporting ring applied to them (after gravimetric assay) to help maintain their flatness and to securely hold them in the frame. The sample is then placed in a custom-designed commercially available two-part sample frame which snaps together holding the filter securely in place.

## **9. Spectral Acquisition and Processing**

**9.1** Spectra are acquired in sets of 15 samples each. Up to 7 spectra are acquired for each sample depending on how many secondary excitation targets are selected. Utilizing all seven fluorescers requires approximately 4 hours machine time for 44 elements analyzed plus atmospheric argon.

**9.2** Elemental intensities are determined by spectral deconvolution with a least squares algorithm which utilizes experimentally determined elemental shape functions instead of the mathematical Gaussian function. This approach has been successfully implemented for many years on an earlier machine and is described in Section 15, Citation 10. Since the spectral shape is not a pure Gaussian the experimental shapes are a more realistic representation of a spectrum. In addition to this library of elemental shape spectra there is also a background shape spectrum for each of the types of filters. It is assumed that the background on an unknown sample is due to the filter and not to the sample. (This is one of the reasons for avoiding heavily loaded filters.) The least squares algorithm synthesizes the spectrum of the sample under analysis by taking a linear combination of all the elemental shapes spectra and the background shape spectrum. The coefficients on the linear combination of elemental shapes and background spectra are scaling factors determined by minimizing chi-square thus producing the best fit possible by least square minimization. Values of the chi-square statistic are calculated for each sample and fluorescer to give an indication of the quality of the fit.

**9.3** X-ray attenuation corrections are performed as described in Section 15, Citation 10 and are briefly described here. The mass absorption coefficients for the layer of fine particles is based on a typical composition of ambient aerosol particles so the actual x-ray attenuations on a given sample are simply a function of the mass loading. Coarse particle attenuations are more complex in that they are based on x-ray

attenuation by spherical particles with compositions of common crustal minerals with various size distributions. An average attenuation and uncertainty for each coarse particle element is based on this broad range of crustal minerals and is therefore a one-time calculation giving an attenuation factor useable for all subsequent coarse (2.5-10  $\mu\text{m}$ ) particle analyses. This treatment assumes low coarse particle loading so that the particles do not shadow one another - yet another reason for assuring that the sample mass loading is not too high. Attenuation corrections on  $\text{PM}_{10}$  particles are deduced from elemental concentration data from samples taken with collocated  $\text{PM}_{10}$  and dichotomous samplers.

**9.4** The need for interference corrections arises from overlaps that are not deconvoluted by the least squares algorithm. This can best be illustrated by an example: Barium and titanium are analyzed by the gadolinium and iron fluorescers, respectively. The barium L x-rays overlap with the K x-rays of titanium and require an interference correction because the elements analyzed by gadolinium do not include titanium. The interference correction technique is described by Gilfrich in Section 15, Citation 29. The interference coefficient, determined during calibration, represents the fraction of the concentration of an affecting element (barium in the present example) which must be subtracted from the concentration of the affected element concentration (titanium) to compensate for the interference.

**9.5** When samples are collected by the dichotomous or other samplers using virtual impaction, an additional correction must be employed because these type of samplers do not perfectly separate the fine and coarse particles. Due to virtual impaction requirements, about 10% of the fine particle mass is deposited on the coarse filter. Therefore, the attenuation corrections used for the particles on the coarse filter "over-correct" the attenuation because of these residual fines on the coarse filter. These effects are compensated for by the flow fraction correction.

## **10. Data Reporting**

*[Note: In other Inorganic Compendium methods, the authors have provided detailed examples of calculations involving final metal concentration (in terms of  $\mu\text{g}/\text{m}^3$ ) from filterable materials. However, due to the nature of overlapping spectra which is characteristic of energy dispersive spectrometers, calculations are required to be performed by computer due to the complexity of the deconvolution of the recorded spectra which uses least square algorithm involving experimentally determined elemental shape functions instead of the mathematical Gaussian function. To perform by hand would require second order calculus and considerable time and manpower. Thus, the application of a computer is mandatory to determine elemental intensities and the elemental concentrations by a polynomial fit using a model based on the fundamentals of x-ray physics process (see Section 11 for further explanation).]*

The two most important data output files are an ASCII file which contains a recapitulation of the field data and the final sample concentrations in  $\text{ng}/\text{m}^3$  and a Lotus file with only the sample data. An example printout of a fine/coarse sample pair is shown in Table 3.

The uncertainty reported with each concentration is a 1F (68% confidence level) uncertainty and is determined by propagating the errors given in Section 12. Elements with concentrations below 3 times the uncertainty are flagged with an asterisk (\*) on the printed record. If the true elemental concentration is zero then the fitting procedure implies that negative and positive results are equally probable. Therefore, negative numbers may be reported.

## **11. Calibration**

**11.1** Calibration is performed only when a change in fluorescers or x-ray tubes or detector is made or a serious malfunction occurs requiring significant repairs. Calibration establishes the elemental sensitivity factors and the magnitude of the interference or overlap coefficients. It takes approximately 2 weeks to complete a calibration.

**11.2** Thin film standards are used for calibration because they most closely resemble the layer of particles on a filter. There are two types of calibration standards in use. One type consists of thin films deposited on Nuclepore substrates (Micromatter Co., Eastsound, WA). These standards are available for almost all the elements analyzed ranging in atomic number from 11 (Na) to 82 (Pb) with deposit masses gravimetrically determined to  $\pm 5\%$ . Another type consists of polymer films that contain known amounts of two elements in the form of organo-metallic compounds dissolved in a polymer and are not commercially available but their preparation is described in Section 15, Citation 9. These standards have been prepared for elements with atomic numbers above 21 (titanium and heavier). The same set of standards is used every time the spectrometer is calibrated. The standards are sufficiently durable to last many years, however occasionally one must be replaced due to accidents in handling. Approximately 200 calibration standards for 44 elements are in use (see Table 4.) and the acquisition of their spectra requires several days.

**11.3** The background files which are used for background fitting are created at calibration time. Thirty clean Teflo® and Nuclepore® blanks are kept sealed in a plastic bag and are used exclusively for background measurement. After acquiring spectra for all 7 fluorescers the spectra are added together to produce a single spectrum for each fluorescer. Options are available to omit a spectrum from the sum if one shows a contamination. It is these summed spectra that are fitted to the background during spectral processing.

**11.4** The shape standards are thin film standards consisting of ultra pure elemental materials for the purpose of determining the physical shape of the pulse height spectrum. For this purpose it is not necessary for the concentration of the standard to be known - only that it be pure. A slight contaminant in the region of interest in a shape standard can have serious effect on the ability of the least squares fitting algorithm to fit the shapes to the unknown. For this reason the Se and elemental As standards, whose compounds are volatile, are kept in separate plastic bags in a freezer to prevent contamination of other standards; the Au standard, which will slowly amalgamate with atmospheric Hg, is kept in a desiccator. The shape standards are acquired for sufficiently long times to provide a large number of counts in the peaks of interest. It is these elemental shape spectra that are fitted to the peaks in an unknown sample during spectral processing.

**11.5** The spectra from the calibration standards are deconvoluted to get elemental intensities as described in Section 9.2. Using these intensities and the elemental concentration in the standards the sensitivities are determined by a polynomial fit using a model based on the fundamentals of the x-ray physics process as well as measurements on the calibration standards. This approach allows the calculation of sensitivities for elements for which there are poor or no standards such as volatile ones like Se and elemental As as well as improving on elements with good standards.

**11.6** The overlap coefficients are determined during calibration and represent the extent of interference that exists between overlapping spectral peaks. During calibration an affecting element (barium, to continue with the example of Section 9.4) is measured both at the analyte line peak for barium and at the titanium peak. The coefficient is expressed as the ratio of the concentration of the affected element (titanium) to the

concentration of the affecting element (barium). All elements requiring overlap coefficient determination are calculated in this manner.

## **12. Detection Limits**

The detection limits are determined by propagation of errors. The sources of random error which are considered are: calibration uncertainty ( $\pm 5\%$ ); long term system stability ( $\pm 5\%$ ); peak and background counting statistics; uncertainty in attenuation corrections; uncertainty in overlap corrections; uncertainty in flow rate; and uncertainty in coarse fraction due to flow fraction correction (paired samples only). Table 2 outlines typical 1F (68% confidence level) detection limits on a Teflo® blank for fine particles and a Nuclepore® blank for coarse (2.5  $\mu\text{m}$ -10  $\mu\text{m}$ ) particles. These detection limits are defined in terms of the uncertainty in the blank. This ignores the effect of other elements which generally is small except for the light elements (potassium and lower) where overlapping spectral lines will increase the detection limit.

*[Note: The difference in the detection limits between the two filters in Table 2 is due more to the difference in sensitivity to fine and coarse particles and less to the difference in filter material.]*

Higher confidence levels may be chosen for the detection limits by multiplying the 1F limits by 2 for a 2F (or 95% level) or by 3 for 3F (or 99.7% level). To convert the detection limits to more useful units one can use the typical deposit areas for 37-mm and 47-mm diameter filters of 6.5  $\text{cm}^2$  and 12.0  $\text{cm}^2$  respectively.

## **13. Quality Control**

**13.1** A comprehensive quality control program is in effect consisting of many measured parameters covering all measurement conditions and automatically produces control charts for all such measurements. All plotted data are normalized to the mean to give a rapid assessment of relative change.

**13.2** Run-time quality control gives an indication of instrument performance at the time of data acquisition by measurements on stable qualitative standards. The parameters which are measured and their significance are: peak areas (monitors change in sensitivity), background areas (monitors contamination or background changes), centroid (monitors gain and zero adjustment to insure that spectra are assigned the correct channel), and FWHM, (monitors degradation of the detector resolution). These four parameters are measured for elements ranging from sodium to lead and include atmospheric argon. An example of plots of run-time QC data are illustrated in Figures 1 through 4 and Table 5, for the target and tolerance values for the parameters measured.

**13.3** In addition to the run-time quality control procedure the analysis results of Standard Reference Materials SRM1833 and SRM1832 are included in the data reports. These results provide an overall check of the spectral processing program for the elements which are certified in the standards. The sole purpose of the SRMs is to provide a quality control measure; the standards are not used for calibration. Typical results of these SRMs are documented in Tables 6 and 7, and plotted in Figure 5.

**13.4** The run-time quality control procedures serve as an indicator of possible emerging problems by flagging deviations greater than 3 tolerance units as defined for each element in Table 5. Persistently increasing trends are investigated to determine their cause(s) before they impact the results of SRM analyses.

**13.5** The acceptance criteria of results for the elements certified in the SRMs is that the uncertainty intervals for the analytical results and those of the certified values should overlap each other. If any element fails this then the run of unknowns is repeated. Repeated failures indicate the need for recalibration.

**13.6** A value for chi-square is calculated and reported with the data to indicate the quality of the fit. Chi-square values that are much larger than 1.0 indicate a problem in the fitting procedure. Changes in detector resolution or gain in the amplifier produce large values for chi-square; however such changes would be detected by the run-time quality control procedure. Also, large chi-square values can accompany results for heavily loaded filters even though the relative errors are typical. In addition, elements analyzed by the titanium and the iron fluoescers may experience large chi-square values due to interferences from overlapping elements. Chi-square is a more useful measure of goodness-of-fit for the other fluoescers for this reason.

**13.7** To acquire more information about fitting problems the fitted spectra can be viewed on the screen or a hard copy printed. Such plots can be compared to the unknown spectra, background spectra, or to the library shape standards to help elucidate the suspected problem. Various statistics such as the correlation coefficient can be calculated on the fitted and measured spectra as a additional measure of the goodness-of-fit. Fitted spectrum superposed on its measured spectrum along with the associated statistics is illustrated in Figure 6.

#### **14. Precision and Accuracy**

Precision varies with the element and concentration. At high concentrations (greater than 1  $\mu\text{g}/\text{cm}^2$ ) a precision of 7.1% can be expected for elements analyzed by one fluoescer and 5.0% can be expected for those analyzed by two. Refer to Table 1 for a listing of the elements and the fluoescers which analyze them. Based upon the analysis of NIST SRMs the accuracy is  $\pm 10\%$ .

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**TABLE 1. EXAMPLE OF FLUORESCER USAGE**

Element	Fluorescer						Gd
	Al	Ti	Fe	Ge	Ag	Zr	
Na	x						
Mg	x						
Al	x						
Si	x						
P	x	x					
S	x	x					
Cl	x	x					
Ar	x	x					
K	x	x					
Ca	x	x					
Sc	x	x					
Ti			x	x			
V			x	x			
Cr			x	x			
Mn				x		x	
Fe				x		x	
Co				x		x	
Ni				x		x	
Cu				x		x	
Zn						x	
Ga						x	
Ge						x	
As						x	
Se						x	
Br						x	
Rb					x	x	
Sr					x		x
Y					x		
Zr					x		x
Mo					x		x
Rh							x
Pd							x
Ag							x
Cd							x
Sn							x
Sb							x
Te							x
I							x
Cs							x
Ba							x
La							x
W					x	x	
Au					x	x	
Hg					x	x	
Pb					x	x	

[Note: The 'x' marks the fluoescers that analyze each element.]

TABLE 2. METHOD DETECTION LIMITS (MDL) FOR  
TEFLO® AND NUCLEPORE® BLANK FILTERS (1F)

Teflo® - fine element			Nuclepore® - coarse element		
Method Detection Limits (MDL)			Method Detection Limits (MDL)		
	ng/cm <sup>2</sup>	ng/m <sup>3</sup> 1		ng/cm <sup>2</sup>	ng/m <sup>3</sup> 2
Na	5.3	1.59	Na	17.4	47.12
Mg	3.2	0.96	Mg	7.9	21.34
Al	17.6	5.29	Al	46.7	126.48
Si	8.0	2.41	Si	21.2	50.40
P	2.6	0.78	P	4.1	11.10
S	2.6	0.78	S	6.9	16.56
Cl	4.8	1.44	Cl	5.6	13.44
K	6.3	1.89	K	5.6	15.17
Ca	9.0	2.71	Ca	8.7	23.56
Sc	1.5	0.45	Sc	1.3	3.52
Ti	16.9	5.08	Ti	18.7	42.52
V	5.3	1.59	V	5.5	14.89
Cr	3.0	0.90	Cr	3.0	8.12
Mn	.8	0.24	Mn	.8	2.17
Fe	.7	0.21	Fe	1.0	2.71
Co	.4	0.12	Co	.4	1.08
Ni	.6	0.18	Ni	.7	1.89
Cu	.7	0.21	Cu	.8	2.17
Zn	1.0	0.30	Zn	1.1	2.98
Ga	1.6	0.48	Ga	1.5	4.06
Ge	1.1	0.33	Ge	1.0	2.71
As	.8	0.24	As	.9	2.44
Se	.7	0.21	Se	.6	1.62
Br	.6	0.18	Br	.7	1.89
Rb	.7	0.21	Rb	.7	1.89
Sr	1.1	0.33	Sr	.9	2.44
Y	1.2	0.36	Y	1.1	2.98
Zr	1.2	0.36	Zr	1.1	2.98
Mo	1.6	0.48	Mo	1.5	4.06
Rh	25.9	7.79	Rh	26.5	71.70
Pd	22.9	6.89	Pd	18.7	50.65
Ag	20.2	6.02	Ag	20.3	54.98
Cd	22.0	6.62	Cd	19.2	52.00
Sn	30.5	9.18	Sn	31.5	85.31
Sb	31.4	9.45	Sb	26.7	72.31
Te	26.3	7.91	Te	27.6	66.62
I	35.5	10.68	I	34.4	93.17
Cs	48.9	14.62	Cs	50.9	137.85
Ba	51.8	15.59	Ba	58.3	157.89
La	70.6	2.12	La	68.9	186.60
W	3.4	10.23	W	3.3	8.93
Au	1.7	0.51	Au	1.5	4.06
Hg	1.5	0.45	Hg	1.4	3.79
Pb	1.5	0.45	Pb	1.5	4.06

<sup>1</sup>Based upon dichotomous sampling for 24-hrs. using a 37-mm Teflo® filter at a sampling rate of 0.9 m<sup>3</sup>/hr.

<sup>2</sup>Based upon dichotomous sampling for 24-hrs using a 37-mm Nuclepore® filter at a sampling rate of 0.1 m<sup>3</sup>/hr.

**TABLE 3. DATA REPORT FORMAT FOR A FINE/COARSE PAIRED SAMPLE**

KEVEX SUMMARY: ADOBE FLATS URBAN PARTICULATE STUDY

SITE	=	ADB	SAMPLE DATE	=	3/20/92 AND 1900 HOURS
DURATION (MIN)	=	714.0	FLOW (L/MIN)	=	37.105 +- .500
FLOW FRAC	=	.0869	XRF ID	=	999956
XRF ID	=	999906	SAMPLE ID	=	NU0033
SAMPLE ID	=	T0033			
		FINE, NG/M <sup>3</sup>			COARSE, NG/M <sup>3</sup>
MASS	77912.	+- 1962.	MASS	11347.	+- 812.
*NA	211.9	+- 71.4	*NA	53.3	+- 27.1
MG	564.6	+- 89.4	MG	443.9	+- 40.8
*AL	162.2	+- 74.1	AL	539.9	+- 173.8
SI	213.4	+- 40.4	SI	909.5	+- 232.7
* P	12.1	+- 18.5	* P	-5.5	+- 11.3
S	2653.4	+- 183.7	S	285.7	+- 84.9
CL	1164.4	+- 79.3	*CL	34.8	+- 24.6
K	193.6	+- 13.8	K	63.5	+- 8.9
CA	43.4	+- 5.6	CA	181.7	+- 13.9
*SC	3.6	+- 4.1	*SC	-1.3	+- 2.2
*TI	17.6	+- 6.6	TI	54.7	+- 9.6
* V	4.6	+- 2.3	* V	3.2	+- 1.7
*CR	2.0	+- 1.0	CR	9.8	+- 1.6
MN	10.0	+- 1.4	MN	10.1	+- 1.3
FE	243.7	+- 21.9	FE	783.5	+- 78.2
*CO	2.8	+- 1.8	*CO	4.8	+- 1.7
NI	3.8	+- 1.2	*NI	.3	+- .6
CU	14.3	+- 1.9	CU	8.8	+- 1.3
ZN	167.5	+- 14.9	ZN	27.6	+- 4.9
*GA	2.4	+- 1.0	*GA	-.0	+- .4
*GE	3.3	+- 1.3	*GE	.0	+- .6
AS	24.7	+- 3.6	*AS	1.8	+- 1.2
SE	4.7	+- .8	*SE	.7	+- .4
BR	29.0	+- 2.8	BR	7.9	+- 1.1
*RB	1.7	+- .8	*RB	1.0	+- .4
SR	2.9	+- .9	SR	2.2	+- .5
* Y	12.4	+- 6.1	* Y	3.9	+- 2.9
*ZR	2.9	+- 4.8	*ZR	4.3	+- 2.6
*MO	7.3	+- 4.8	*MO	-3.2	+- 2.2
*RH	.0	+- 3.2	*RH	-1.2	+- 1.6
*PD	-3.6	+- 3.1	*PD	-1.0	+- 1.7
*AG	-6.4	+- 3.4	*AG	1.2	+- 1.9
*CD	8.5	+- 4.5	*CD	-.7	+- 2.2
SN	54.3	+- 9.4	*SN	2.3	+- 3.9
*SB	-1.6	+- 6.4	*SB	-.6	+- 3.3
*TE	2.5	+- 7.5	*TE	-7.2	+- 3.8
* I	25.0	+- 9.6	* I	2.4	+- 4.7
*CS	-4.0	+- 11.2	*CS	12.4	+- 5.9
*BA	-7.7	+- 13.7	BA	25.1	+- 7.4
*LA	-4.8	+- 34.5	*LA	22.6	+- 17.9
* W	-1.1	+- 2.6	* W	1.5	+- 1.3

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*AU	-.9 +-	1.8	*AU	.2 +-	.9
*HG	-.4 +-	1.9	*HG	1.5 +-	1.0
PB	221.6 +-	19.7	PB	46.0 +-	6.2

\* INDICATES THAT THE CONCENTRATION IS BELOW 3 TIMES THE UNCERTAINTY.  
XRF DATE= 04/29/1992 16:35 RBK (F): 04/29/1992 20:35 RBK (C)  
SPECTRAL ANALYSIS DATE= 5/20/1992

TABLE 4. CALIBRATION STANDARDS AND CONCENTRATIONS

Standard ID	Element	µg/cm <sup>2</sup>									
CaF237	F	18.00	CuS1124	S	31.90	Cr 85	Cr	85.00	RbNO311	Rb	69.00
CaF2 29	F	14.10	CuS58.6	S	16.50	Cr 84	Cr	84.00	RbNO322	Rb	12.90
CaF2 90	F	43.80	CuS57.6	S	13.90	Cr 75	Cr	75.00	RbNO3 a	Rb	24.90
CaF2 91	F	44.30	CuS58.2	S	14.00	Cr 74	Cr	74.00	RbNO3 b	Rb	24.90
CaF2102	F	49.60	NaCl 57	Cl	34.60	Cr 122	Cr	122.00	RbNO3 c	Rb	24.90
CaF2 66	F	32.10	NaCl 87	Cl	52.80	CrCu32a	Cr	9.19	SrF2 57	Sr	39.80
CaF2 28	F	13.60	NaCl446	Cl	27.10	CrCu26g	Cr	8.14	SbSr29z	Sr	4.97
CaF2 33	F	16.10	NaCl715	Cl	43.40	MnZn24b	Mn	8.57	SrF2 50	Sr	34.90
CaF2 39	F	19.00	NaCl497	Cl	30.20	Mn 57	Mn	57.00	SbSr31y	Sr	5.14
CaF2 54	F	26.30	NaCl501	Cl	30.40	Mn 183	Mn	183.00	SrF2137	Sr	95.60
CaF2291	F	14.10	NaCl 51	Cl	31.00	MnZn27x	Mn	9.10	SrF2184	Sr	12.80
CaF2 30	F	14.60	NaCl512	Cl	31.10	Mn 43	Mn	43.00	SrF2 92	Sr	64.20
CaF2 52	F	25.30	NaCl519	Cl	31.50	Mn 46.9	Mn	46.90	SrF2103	Sr	71.80
CaF2 48	F	23.40	KCl 45	Cl	21.40	Mn 44.5	Mn	44.50	YF3 46	Y	28.00
CaF2 45	F	21.90	KCl53.3	Cl	25.40	Mn 46.6	Mn	46.60	ZrCd24c	Zr	9.85
CaF2 36	F	17.50	KCl 70	Cl	33.30	Mn 43.7	Mn	43.70	ZrCd20w	Zr	10.77
CaF2134	F	65.20	KCl 49	Cl	23.30	Mn 69	Mn	69.00	MoO3145	Mo	96.70
CaF2110	F	53.50	KCl48.7	Cl	23.20	FePb37y	Fe	7.72	MoO3106	Mo	70.70
NaCl 57	Na	22.40	KCl47.9	Cl	22.80	Fe 107	Fe	107.00	MoO3110	Mo	73.30
NaCl 87	Na	34.20	KCl 48	Cl	22.80	Fe 127	Fe	127.00	MoO3 59	Mo	39.30
NaCl446	Na	17.60	KCl47.6	Cl	22.60	Fe 46	Fe	46.00	MoO3 54	Mo	36.00
NaCl715	Na	28.10	KCl 45	K	23.60	Fe 88	Fe	88.00	Rh 16	Rh	16.00
NaCl497	Na	19.60	KCl53.3	K	28.00	FePb38y	Fe	7.71	Pd 33	Pd	33.00
NaCl501	Na	19.70	KCl 70	K	36.70	Co 45a	Co	45.00	Pd 198	Pd	198.00
NaCl 51	Na	20.10	KCl 49	K	25.70	Co 45b	Co	45.00	Ag 35	Ag	35.00
NaCl512	Na	20.10	KCl48.7	K	25.50	RbCo29c	Co	7.43	Ag 132	Ag	132.00
NaCl519	Na	20.40	KCl47.9	K	25.10	RbCo25b	Co	7.65	Cd 83	Cd	83.00
Mg 81	Mg	81.00	KCl 48	K	25.20	Ni 54	Ni	54.00	ZrCd20w	Cd	9.15
Mg 41	Mg	41.00	KCl47.6	K	25.00	Ni 88	Ni	88.00	ZrCd24c	Cd	8.38
Mg 41.3	Mg	41.30	CaF2 37	Ca	19.00	NiV 21c	Ni	5.77	Cd 77	Cd	77.00
Mg 43	Mg	43.00	CaF2 29	Ca	14.90	Ni 101	Ni	101.00	Sn 40	Sn	40.00
Mg 43.8	Mg	43.80	CaF2 90	Ca	46.20	Cu 96	Cu	96.00	Sn 185	Sn	185.00
Mg 60.2	Mg	60.20	CaF2 91	Ca	46.70	Cu 104	Cu	104.00	Sn 97a	Sn	97.00
Al 57	Al	57.00	CaF2102	Ca	52.40	Cu 128	Cu	128.00	Sn 97b	Sn	97.00
Al 37.9	Al	37.90	CaF2 66	Ca	33.90	CrCu26g	Cu	7.65	Sn 79	Sn	79.00
Al 37.4	Al	37.40	CaF2 28	Ca	14.40	CrCu32a	Cu	8.63	Sb 194	Sb	194.00
Al 29	Al	29.00	CaF2 33	Ca	16.90	Cu 38	Cu	38.00	Sb 47	Sb	47.00
Al 43.2	Al	43.20	CaF2 39	Ca	20.00	Zn 51	Zn	51.00	Sb 147	Sb	147.00
Al 62	Al	62.00	CaF2 54	Ca	27.20	Zn 125	Zn	125.00	Sb 42	Sb	42.00
Al 75	Al	75.00	CaF2291	Ca	14.90	MnZn27x	Zn	8.46	SbSr29z	Sb	5.01
SiO 46	Si	29.30	CaF2 30	Ca	15.40	MnZn24b	Zn	7.97	SbSr31y	Sb	5.18
SiO 47	Si	29.90	CaF2 52	Ca	26.70	GaP 34	Ga	23.50	Te 53	Te	53.00
SiO 51a	Si	32.50	CaF2 48	Ca	24.60	GaP 40	Ga	27.70	KI 46	I	35.20
SiO 51b	Si	32.50	CaF2 45	Ca	23.10	GaP 70	Ga	48.50	CsBr 53	Cs	33.10
SiO 56	Si	35.70	CaF2 36	Ca	18.50	GaP 105	Ga	72.70	CsBr 54	Cs	33.70
SiO 80	Si	51.00	CaF2134	Ca	68.60	Ge 37	Ge	37.00	CsBr 51	Cs	31.90
SiO27.6	Si	17.60	CaF2110	Ca	56.50	TiGe33d	Ge	6.22	BaF2108	Ba	84.60
SiO46.1	Si	29.40	ScF3 57	Sc	25.10	TiGe29x	Ge	5.94	BaF2 48	Ba	37.60
SiO72.2	Si	46.00	Ti 39	Ti	39.00	Ge 140	Ge	140.00	BaF2 60	Ba	47.00
GaP 34	P	10.50	Ti 95	Ti	95.00	BaAs23y	As	5.60	BaF2 57	Ba	44.70
GaP 40	P	12.30	TiGe33d	Ti	2.46	BaAs36w	As	5.52	BaF2143	Ba	112.00
GaP 70	P	21.50	TiGe29x	Ti	2.36	CsBr 53	Br	19.90	BaF2114	Ba	89.40
GaP 105	P	32.30	V 45	V	45.00	CsBr 54	Br	20.30	BaAs23y	Ba	4.98

TABLE 4. (continued)

Standard ID	Element	$\mu\text{g}/\text{cm}^2$									
CuS1052	S	30.80	V 53	V	53.00	CsBr 51	Br	19.10	BaAs36w	Ba	4.91
CuS 48	S	13.00	NiV 21c	V	6.64	RbNO346	Rb	26.60	LaF3157	La	111.30
CuS 136	S	33.00	Cr 30	Cr	30.00	RbCo25b	Rb	7.88	LaF3 62	La	44.00
CuS39.6	S	10.20	Cr 53	Cr	53.00	RbCo29c	Rb	7.65			

TABLE 5. TARGET AND TOLERANCE VALUES FOR QC RESULTS

(TARGET VALUES)									
FILE: 0:QCBEGTGT					FILE: 0:QCENDTGT				
STDEL ID	EL	AREA (cts)	CENTROID (keV)	FWHM (ev)	STD ID	EL	AREA (cts)	CENTROID (keV)	FWHM (ev)
1833	Pb	31112.12	10.5449	207.4653	1832	Cu	17548.85	8.0411	174.1389
1833	Zn	31772.52	8.6306	179.6835	1832		5303.84	6.9247	167.1478
1833	Fe	313475.41	6.3935	159.4537	1832	Mn	86202.33	5.8891	154.6347
1833	Ti	216978.09	4.5037	142.4946	1832	Ca	217562.00	3.6847	135.3520
1833	Si	69021.60	1.7322	121.7406	1832	V	99761.96	4.9443	146.1904
1833	K	220344.80	3.3069	132.4137	1832	Al	16562.45	1.4779	119.5793
BLKt	Sn	111.52	0.0000	0.0000	1832	Si	67688.42	1.7319	118.4960
BLKt	Pb	85.82	0.0000	0.0000	1832	Na	10332.21	1.0256	114.4485
BLKt	Cu	497.06	0.0000	0.0000	BLKt	Ba	183.14	0.0000	0.0000
BLKt	Sr	72.92	0.0000	0.0000	BLKt	W	241.42	0.0000	0.0000
BLKt	Ni	648.99	0.0000	0.0000	BLKt	Zn	148.48	0.0000	0.0000
BLKt	Fe	459.10	0.0000	0.0000	BLKt	Sr	83.00	0.0000	0.0000
BLKt	S	266.76	0.0000	0.0000	BLKt	Ni	654.44	0.0000	0.0000
BLKt	Al	396.30	0.0000	0.0000	BLKt	Fe	603.55	0.0000	0.0000
BLKt	Ar	747.74	0.0000	0.0000	BLKt	S	3047.53	0.0000	0.0000
BLKt	Na	120.85	0.0000	0.0000	BLKt	Si	936.48	0.0000	0.0000
BaNa	Na	27711.44	1.0278	107.2698	BLKt	Ar	751.18	0.0000	0.0000
BaNa	Ba	7369.12	32.0701	670.6336	BLKt	Mg	3622.12	0.0000	0.0000
					BaSr	Sr	210871.20	14.1410	227.8625
					BaSr	Ba	7464.85	32.0692	671.0372
(TOLERANCE UNITS in %)									
FILE: 0:QCBEGTOL					FILE: 0:QCENDTOL				
STDEL ID	EL	AREA (cts)	CENTROID (keV)	FWHM (ev)	STD ID	EL	AREA (cts)	CENTROID (keV)	FWHM (ev)
1833	Pb	1.66	0.0313	0.9901	1832	Cu	1.66	0.0104	1.9331
1833	Zn	1.66	0.0131	1.7328	1832	Co	1.70	0.0308	2.4345
1833	Fe	1.66	0.0224	0.9361	1832	Mn	1.66	0.0198	1.3536
1833	Ti	1.66	0.0259	0.9768	1832	Ca	1.66	0.0253	1.1311
1833	Si	1.66	0.0616	1.4120	1832	V	1.66	0.0243	1.1031
1833	K	1.66	0.0323	0.9235	1832	Al	2.02	0.1173	3.3722
BLKt	Sn	12.98	0.0000	0.0000	1832	Si	1.66	0.0481	0.8888
BLKt	Pb	8.93	0.0000	0.0000	1832	Na	1.78	0.1560	1.5333
BLKt	Cu	4.95	0.0000	0.0000	BLKt	Ba	9.92	0.0000	0.0000
BLKt	Sr	17.61	0.0000	0.0000	BLKt	W	8.20	0.0000	0.0000
BLKt	Ni	3.81	0.0000	0.0000	BLKt	Zn	11.45	0.0000	0.0000
BLKt	Fe	7.57	0.0000	0.0000	BLKt	Sr	10.88	0.0000	0.0000
BLKt	S	8.71	0.0000	0.0000	BLKt	Ni	6.55	0.0000	0.0000
BLKt	Al	7.23	0.0000	0.0000	BLKt	Fe	5.63	0.0000	0.0000
BLKt	Ar	17.39	0.0000	0.0000	BLKt	S	2.88	0.0000	0.0000
BLKt	Na	16.00	0.0000	0.0000	BLKt	Si	6.75	0.0000	0.0000
BaNa	N	1.66	0.1103	1.2599	BLKt	Ar	22.14	0.0000	0.0000
BaNa	Ba	2.53	0.0979	3.9782	BLKt	Mg	5.64	0.0000	0.0000
					BaSr	Sr	1.66	0.0073	0.4538
					BaSr	Ba	1.86	0.0279	2.8094

TABLE 6. EXAMPLE PRINTOUT OF SRM 1833

KEVEX SUMMARY: TEFLON® BLANKS LOT #457803 (NEW TUBE)

SITE =  
 DURATION (MIN) = .0      SAMPLE DATE = 99/99/99 AND 9999 HOURS  
 FLOW FRAC = .0000      FLOW (L/MIN) = .000 +- .200  
 XRF ID = 112141  
 SAMPLE ID = SRM1833

FINE, NG/CM <sup>2</sup>			NIST CERTIFIED VALUES		
MASS	0.	+-	398.	MASS	15447
*NA	-801.2	+-	326.4	NA	.0 +- .0
MG	161.3	+-	18.2	MG	.0 +- .0
AL	1027.5	+-	102.2	AL	.0 +- .0
SI	34806.8	+-	3023.4	SI	33366.0 +- 2163.0
P	79.8	+-	19.9	P	.0 +- .0
*S	-28.2	+-	782.8	S	.0 +- .0
*CL	-68.6	+-	113.8	CL	.0 +- .0
K	16734.7	+-	1018.7	K	17147.0 +- 1699.0
*CA	-3.9	+-	61.4	CA	.0 +- .0
*SC	-17.1	+-	5.4	SC	.0 +- .0
TI	12852.9	+-	822.1	TI	12821.0 +- 1854.0
*V	46.0	+-	52.2	V	.0 +- .0
CR	108.2	+-	12.7	CR	.0 +- .0
MN	13.8	+-	2.9	MN	.0 +- .0
FE	14332.4	+-	872.4	FE	14212.0 +- 463.0
*CO	-2.6	+-	2.9	CO	.0 +- .0
NI	62.5	+-	4.6	NI	.0 +- .0
*CU	3.8	+-	1.5	CU	.0 +- .0
ZN	3800.9	+-	327.7	ZN	3862.0 +- 309.0
*GA	-30.9	+-	7.7	GA	.0 +- .0
*GE	5.9	+-	3.6	GE	.0 +- .0
*AS	5.7	+-	14.6	AS	.0 +- .0
*SE	-2.0	+-	2.6	SE	.0 +- .0
*BR	-2.3	+-	2.5	BR	.0 +- .0
*RB	.5	+-	1.4	RB	.0 +- .0
*SR	-5.0	+-	2.9	SR	.0 +- .0
*Y	-2.6	+-	7.5	Y	.0 +- .0
*ZR	-7.6	+-	3.5	ZR	.0 +- .0
MO	45.4	+-	5.6	MO	.0 +- .0
*RH	156.7	+-	69.5	RH	.0 +- .0
*PD	79.2	+-	67.1	PD	.0 +- .0
*AG	114.0	+-	69.7	AG	.0 +- .0

TABLE 6. (continued)

FINE, NG/CM <sup>2</sup>				NIST CERTIFIED VALUES			
*CD	24.7	+-	66.3	CD	.0	+-	.0
*SN	-1496.1	+-	188.1	SN	.0	+-	.0
*SB	88.2	+-	96.2	SB	.0	+-	.0
*TE	240.8	+-	93.8	TE	.0	+-	.0
*I	134.8	+-	107.5	I	.0	+-	.0
*CS	-209.3	+-	106.6	CS	.0	+-	.0
*BA	-5098.1	+-	517.8	BA	.0	+-	.0
*LA	-1416.4	+-	202.2	LA	.0	+-	.0
W	59.9	+-	17.6	W	.0	+-	.0
*AU	8.7	+-	6.8	AU	.0	+-	.0
*HG	-30.6	+-	5.9	HG	.0	+-	.0
PB	16886.2	+-	1028.1	PB	16374.0	+-	772.0

\* INDICATES THAT THE CONCENTRATION IS BELOW 3 TIMES THE UNCERTAINTY.

XRF DATE= 28-SEP-93 10:58:37 RBK

SPECTRAL ANALYSIS DATE= 12/14/1993

TABLE 7. EXAMPLE PRINTOUT OF SRM 1832

KEVEX SUMMARY: TEFLON® BLANKS LOT #457803 (NEW TUBE)

SITE =  
 DURATION (MIN) = .0 SAMPLE DATE = 99/99/99 AND 9999 HOURS  
 FLOW FRAC = .0000 FLOW (L/MIN) = .000 +- .200  
 XRF ID = 112191  
 SAMPLE ID = SRM1832

FINE, NG/CM <sup>2</sup>				NIST CERTIFIED VALUES			
MASS	0.	+-	398.	MASS	16431		
NA	11891.5	+-	1035.0	NA	11173.0	+-	.0
MG	92.2	+-	13.0	MG	.0	+-	.0
AL	15856.5	+-	1373.2	AL	14953.0	+-	986.0
SI	34398.8	+-	2964.2	SI	35491.0	+-	1150.0
P	492.0	+-	32.1	P	.0	+-	.0
S	402.1	+-	27.3	S	.0	+-	.0
CL	156.8	+-	15.9	CL	.0	+-	.0
*K	18.5	+-	18.0	K	.0	+-	.0
CA	20011.7	+-	1218.2	CA	19225.0	+-	1315.0
*SC	-21.8	+-	5.6	SC	.0	+-	.0
*TI	-4.7	+-	130.6	TI	.0	+-	.0
V	4593.6	+-	281.1	V	4272.0	+-	493.0
*CR	7.4	+-	7.3	CR	.0	+-	.0
MN	4959.3	+-	302.4	MN	4437.0	+-	493.0
FE	30.5	+-	3.9	FE	.0	+-	.0
CO	1055.1	+-	64.7	CO	970.0	+-	66.0
*NI	-6.8	+-	1.8	NI	.0	+-	.0
CU	2400.1	+-	146.3	CU	2300.0	+-	164.0
ZN	9.3	+-	2.7	ZN	.0	+-	.0
*GA	2.1	+-	2.1	GA	.0	+-	.0
*GE	.3	+-	2.4	GE	.0	+-	.0
*AS	-3.7	+-	2.2	AS	.0	+-	.0
*SE	1.0	+-	1.2	SE	.0	+-	.0
BR	10.7	+-	1.8	BR	.0	+-	.0
*RB	-.2	+-	.9	RB	.0	+-	.0
*SR	2.8	+-	2.3	SR	.0	+-	.0
*Y	-5.0	+-	1.6	Y	.0	+-	.0
*ZR	-6.5	+-	1.8	ZR	.0	+-	.0
MO	26.8	+-	4.2	MO	.0	+-	.0
*RH	25.2	+-	58.2	RH	.0	+-	.0

TABLE 7. (continued)

	FINE, NG/CM <sup>2</sup>			NIST CERTIFIED VALUES			
*PD	-69.0	+-	54.7	PD	.0	+-	.0
*AG	151.2	+-	63.4	AG	.0	+-	.0
*CD	24.2	+-	58.2	CD	.0	+-	.0
*SN	-640.8	+-	138.6	SN	.0	+-	.0
*SB	-73.5	+-	81.3	SB	.0	+-	.0
*TE	-9.3	+-	73.9	TE	.0	+-	.0
*I	-46.6	+-	91.6	I	.0	+-	.0
*CS	3.6	+-	96.7	CS	.0	+-	.0
*BA	-2352.9	+-	328.6	BA	.0	+-	.0
*LA	-509.9	+-	156.5	LA	.0	+-	.0
W	40.0	+-	12.9	W	.0	+-	.0
*AU	-5.6	+-	2.5	AU	.0	+-	.0
*HG	-5.4	+-	3.0	HG	.0	+-	.0
*PB	-10.4	+-	4.2	PB	.0	+-	.0

\* INDICATES THAT THE CONCENTRATION IS BELOW 3 TIMES THE UNCERTAINTY.

XRF DATE= 29-SEP-93 13:27:55 RBK

SPECTRAL ANALYSIS DATE= 12/14/1993

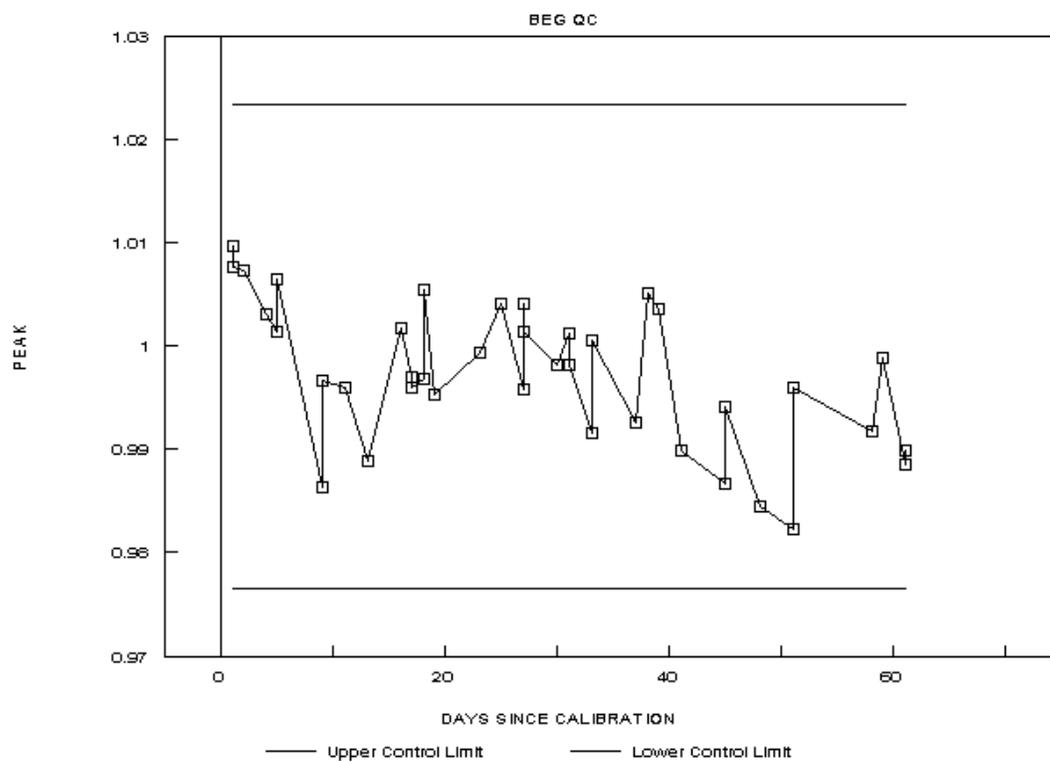


Figure 1. Quality control indicator associated with Fe peak area.

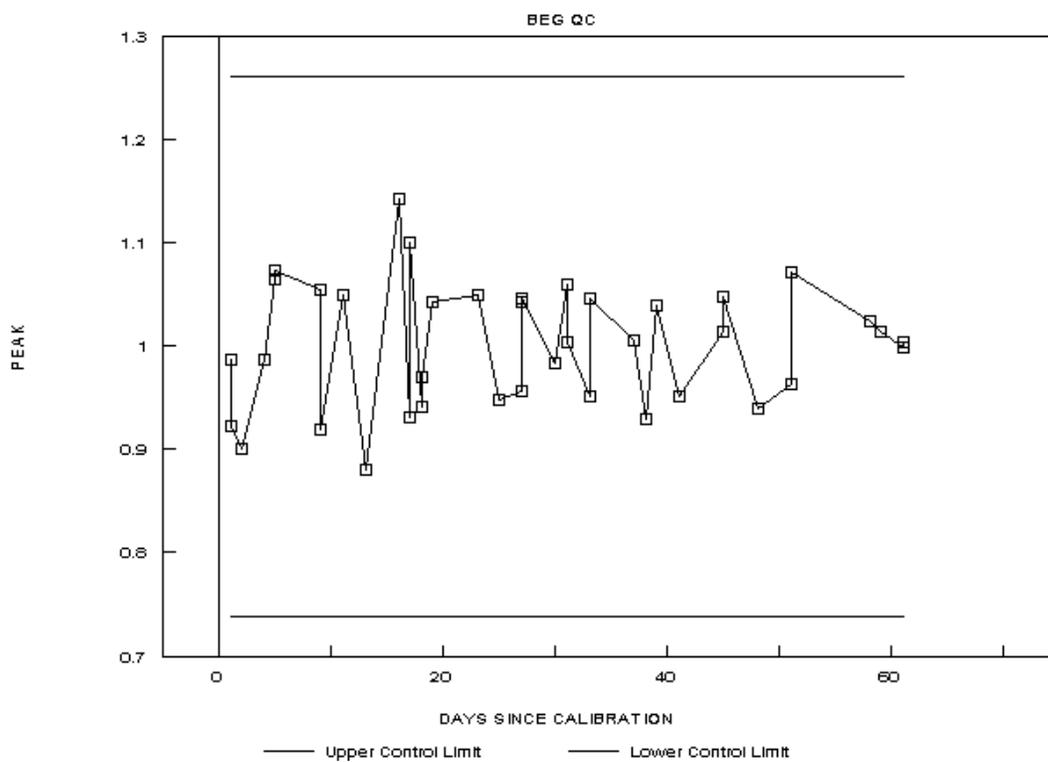


Figure 2. Quality control indicator associated with S background area.

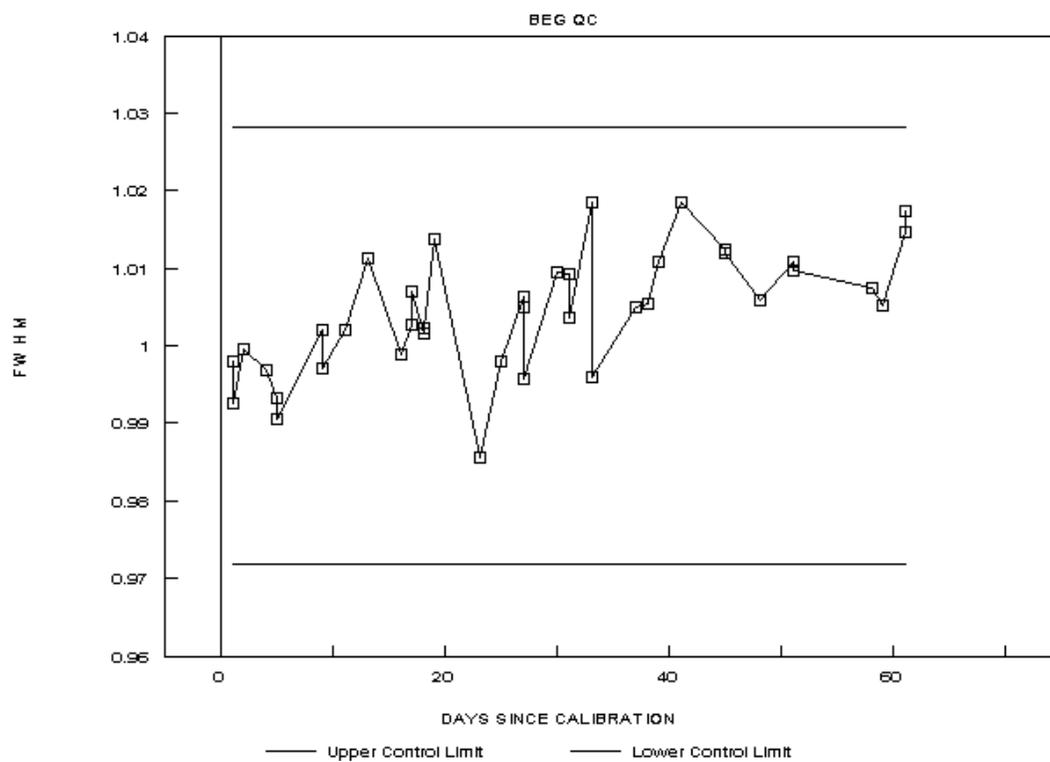


Figure 3. Quality control indicator associated with Fe FWHM.

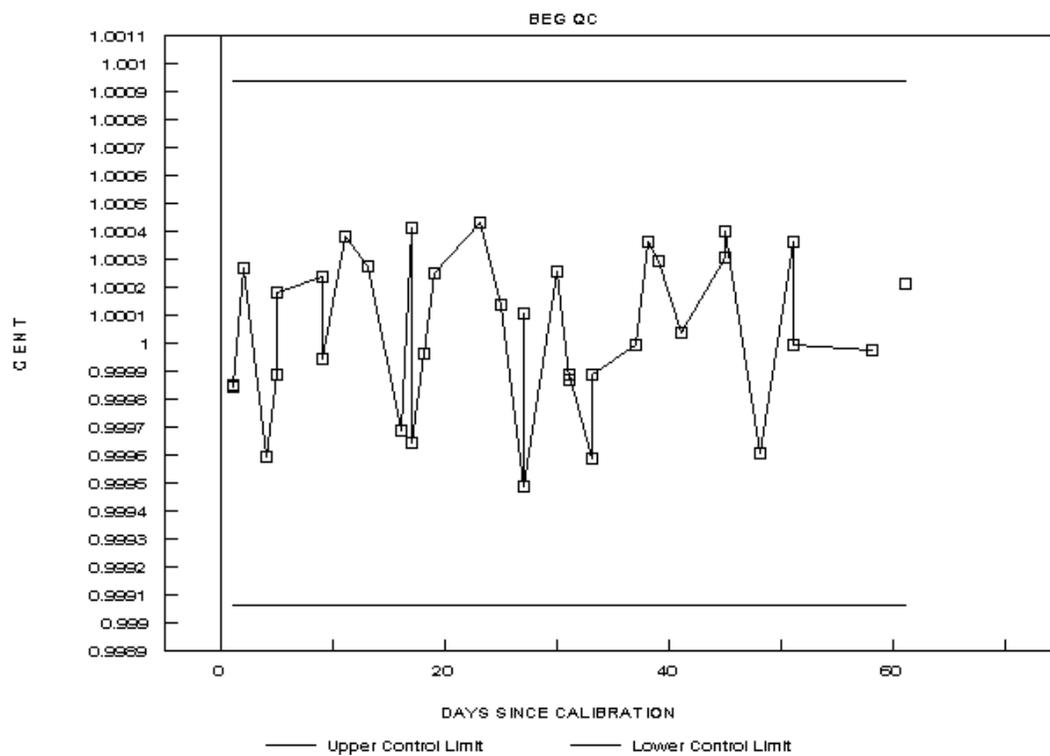


Figure 4. Quality control indicator associated with Pb centroid.

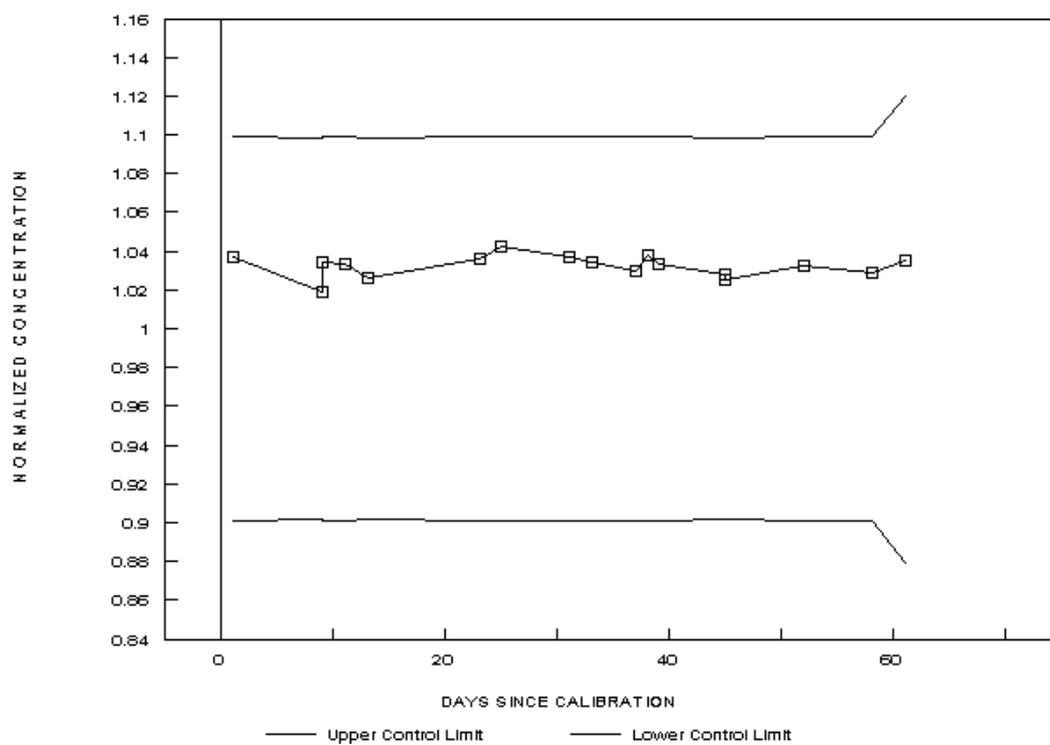


Figure 5. Quality control indicator associated with Pb in SRMs.

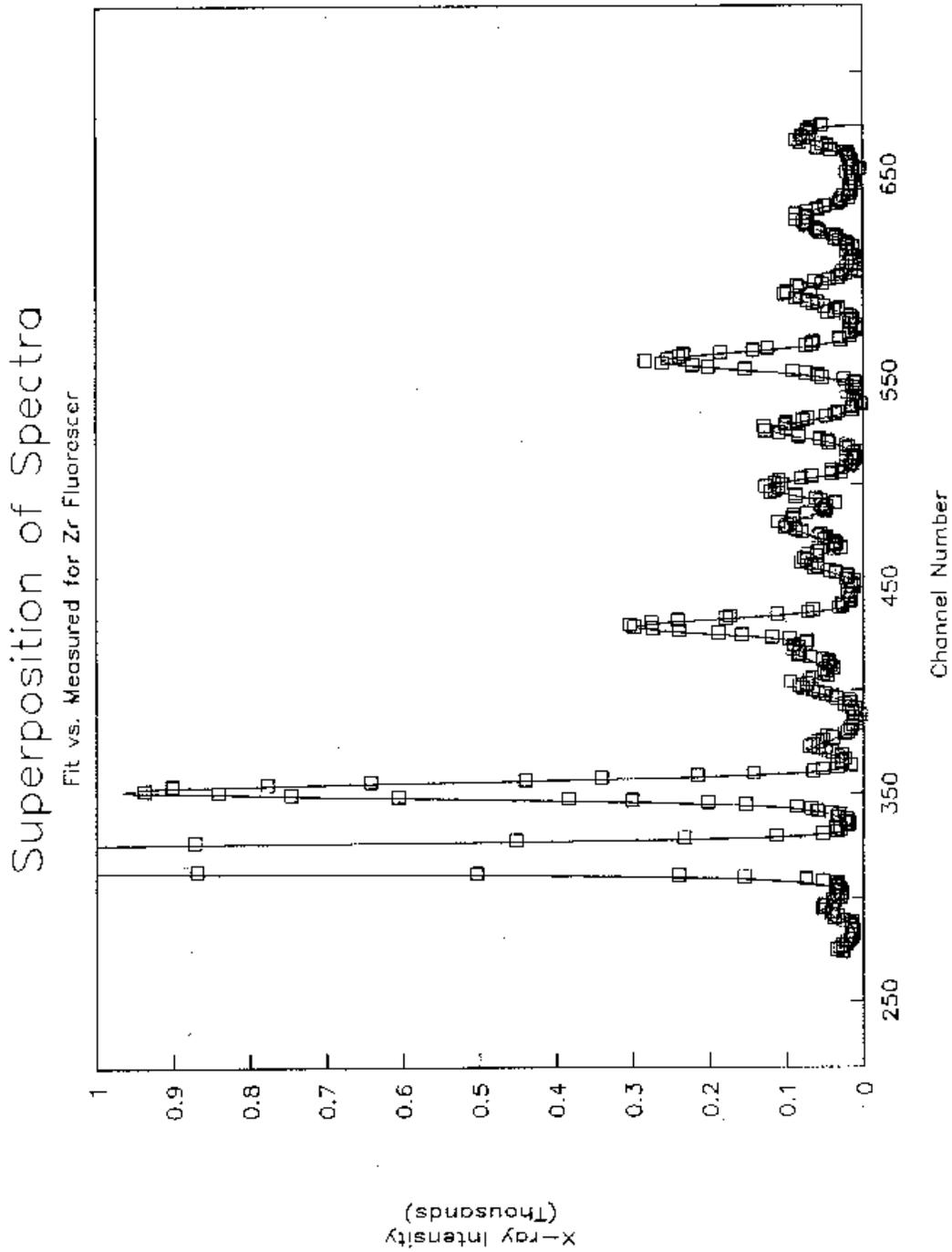


Figure 6. Quality control indicator associated with superposition of fitted and measured spectrum.

## METHOD 8095

### EXPLOSIVES BY GAS CHROMATOGRAPHY

#### 1.0 SCOPE AND APPLICATION

1.1 Method 8095 may be used to determine the concentrations of various explosives in water and soil using capillary column gas chromatography with an electron capture detector (GC/ECD). The compounds are nitroaromatics, nitramines, and nitrate esters, which are used as explosives, are byproducts of the manufacture of explosives, or are the transformation products of explosives. The method has also been successfully used to determine the commonly found explosives in acetonitrile extracts from soil prepared by the extraction procedure in Method 8330.

Compound	Abbreviation	CAS Number*
2-Amino-4,6-dinitrotoluene	2-Am-DNT	35572-78-2
4-Amino-2,6-dinitrotoluene	4-Am-DNT	1946-51-0
3,5-Dinitroaniline	3,5-DNA	618-87-1
1,3-Dinitrobenzene	1,3-DNB	99-65-0
2,4-Dinitrotoluene	2,4-DNT	121-14-2
2,6-Dinitrotoluene	2,6-DNT	606-20-2
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX	121-82-4
Nitrobenzene	NB	98-95-3
Nitroglycerine	NG	55-63-0
2-Nitrotoluene	2-NT	88-72-2
3-Nitrotoluene	3-NT	99-08-1
4-Nitrotoluene	4-NT	99-99-0
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX	2691-41-0
Pentaerythritoltetranitrate	PETN	78-11-5
1,3,5-Trinitrobenzene	1,3,5-TNB	99-35-4
2,4,6-Trinitrophenylmethylnitramine	Tetryl	479-45-8
2,4,6-Trinitrotoluene	2,4,6-TNT	118-96-7

\* Chemical Abstracts Service Registry number.

1.2 Aqueous samples to be analyzed by this procedure are extracted using the solid-phase extraction techniques in Method 3535. Solid samples are extracted using the extraction techniques in Method 8330.

1.3 The method is capable of detecting the target compounds in a range from 0.003 to 0.5 µg/L and is capable of quantitative analysis in a range of 0.03 to 5 µg/L, depending on the sensitivity of the analyte to electron capture detection.

1.4 Method 8095 requires special precautions in the operation of the gas chromatograph because of the thermal lability of many of these compounds, especially the nitramines.

1.5 Prior to employing this method, analysts are advised to consult the base method for each type of procedure that may be employed in the overall analysis (e.g., Methods 3500, 3600, 5000, and 8000) for additional information on quality control procedures, development of QC acceptance criteria, calculations, and general guidance. Analysts also should consult the disclaimer statement at the front of the manual and the information in Chapter Two, Sec. 2.1, for guidance on the intended flexibility in the choice of methods, apparatus, materials, reagents, and supplies, and on the responsibilities of the analyst for demonstrating that the techniques employed are appropriate for the analytes of interest, in the matrix of interest, and at the levels of concern.

In addition, analysts and data users are advised that, except where explicitly specified in a regulation, the use of SW-846 methods is *not* mandatory in response to Federal testing requirements. The information contained in this method is provided by EPA as guidance to be used by the analyst and the regulated community in making judgments necessary to generate results that meet the data quality objectives for the intended application.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of GC/ECD, skilled in the interpretation of chromatograms, and trained in the handling of environmental samples that may contain explosives. Also, each analyst must demonstrate the ability to generate acceptable results with this method.

NOTE: Refer to Section 5.0 for additional information on safety.

## 2.0 SUMMARY OF METHOD

2.1 Samples are extracted using either the solid-phase extraction techniques provided in Method 3535 (aqueous samples) or the ultrasonic extraction techniques described in Method 8330 (solid samples), using acetonitrile as the solvent. No further concentration of the extract is performed unless lower detection limits are required.

2.2 Acetonitrile extracts are injected into the heated inlet of a gas chromatograph equipped with an electron capture detector. The analytes are resolved on a short wide-bore fused-silica capillary column coated with polydimethylsiloxane.

## 3.0 DEFINITIONS

Definitions related to this method are found in Chapter One.

## 4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interference by the analysis of reagent blanks.

4.2 In addition to nitrogenated organics, the ECD will respond to other electrophores such as halogenated and oxygenated compounds. Interference by phthalate esters introduced during sample preparation can pose a major problem. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents prior to use.

4.3 The injection port liner must be deactivated to prevent adsorption of several analytes. After several injections of sample extracts, deposition of non-volatile residues may result in peak tailing and a decline in the response for HMX. Each time the septum is replaced, the injection port liner must be deactivated again or a commercially-available deactivated liner must be used. Analysts should expect to replace or deactivate the liner after every 50 injections, unless data demonstrating acceptable performance can be generated for HMX and all other analytes of interest.

## 5.0 SAFETY

5.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses.

5.2 The target analytes for this method are explosive materials. Analysts must be trained in proper handling techniques for explosive-containing material in order to appropriately use this method, or supervised by those who have received such training. Caution should be exercised during the sampling of possible explosive-contaminated material. Solid material must be carefully inspected prior to extraction and care should be employed when handling the analytical standard neat material of the explosives. Observe the precautions described in the warning in Sec. 7.4.1 regarding drying the neat materials at ambient temperatures. Additional caution should be exercised if the material has a dry appearance or a grayish look that is characteristic of raw munitions.

## 6.0 EQUIPMENT AND SUPPLIES

6.1 Gas chromatograph - An analytical system complete with gas chromatograph, equipped with a temperature-programmable oven, an electron capture detector (ECD), and suitable for on-column injection. If a split-splitless injection port is used, deactivated direct injection port liners must be used to avoid degradation of the nitramines (especially HMX). Other necessary accessories include syringes, analytical columns, gases, and recorder/integrator or data system.

**NOTE:** It is recommended that deactivated direct injection port liners be purchased from a commercial vendor (Restek #20964, #20965, #20966, or equivalent).

6.2 GC columns - The columns listed in this section were the columns used to develop the method performance data. The analyst should select one primary and one confirmation column from either the recommendations listed below or based on other sources. The mention of these columns in this method is not intended to exclude the use of other columns. Laboratories may use other columns provided that they document method performance data (e.g., chromatographic resolution, analyte breakdown, and sensitivity that are appropriate for the intended application).

## 6.2.1 Recommended primary columns

6.2.1.1 6 m x 0.53-mm ID fused-silica, coated with 5% diphenyl - 95% dimethylsiloxane (HP-5, or equivalent), 1.5- $\mu$ m film thickness.

6.2.1.2 6 m x 0.53-mm ID fused-silica, coated with 100% polydimethylsiloxane (DB-1 or equivalent), 1.5- $\mu$ m film thickness.

## 6.2.2 Recommended confirmatory columns

6.2.2.1 6 m x 0.53-mm ID fused-silica, coated with 100% trifluoropropyl methylpolysiloxane (Restek RTX-200 or equivalent), 1.5- $\mu$ m film thickness.

6.2.2.2 6 m x 0.53-mm ID fused-silica, coated with 50% cyanopropylmethyl - 75% phenyl methylpolysiloxane (Restek RTX-225 or equivalent), 1.5- $\mu$ m film thickness.

6.3 Autosampler with ability to refrigerate vials (HP 6890 or equivalent) - The use of an autosampler is optional, and injections may be made manually. However, if an autosampler is employed, it MUST be able to refrigerate the vials to  $4 \pm 2^\circ\text{C}$  to avoid the degradation of the analytes of interest while the vials are sitting in the autosampler tray.

6.4 Refrigerated circulating bath (Neslab Endocal or equivalent) - For use with the refrigerated autosampler in Sec. 6.3. Must be capable of maintaining the autosampler tray at a temperature of  $4 \pm 2^\circ\text{C}$ .

6.5 Disposable cartridge filters, 0.45-micron (Millex SR or equivalent).

6.6 Disposable syringes, Plastipak, 3-mL (or equivalent).

6.7 Vacuum desiccator, glass.

6.8 Volumetric Flasks, Class A

## 7.0 REAGENTS AND STANDARDS

7.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination or causing interferences.

7.2 Extraction/exchange solvents - All solvents must be pesticide quality or equivalent.

7.2.1 Acetonitrile ( $\text{CH}_3\text{CN}$ )

7.2.2 Acetone ( $\text{CH}_3\text{COCH}_3$ )

7.2.3 2-Propanol ( $(\text{CH}_3)_2\text{CHOH}$ )

#### 7.2.4 Methanol (CH<sub>3</sub>OH)

7.3 Analytical standards - Calibration standards are commercially available from several sources including Supelco, AccuStandard and Radian, as both solutions and neat materials. It is **highly recommended** that commercially-prepared stock standard solutions be obtained rather than handling neat materials.

7.4 Stock standard solution (1000 mg/L) - Can be purchased as certified solutions or may be prepared from pure standard analytical reference material. Store purchased standards in the dark at  $\leq 4^{\circ}\text{C}$ , or as recommended by the standard manufacturer. It is **highly recommended** that commercially-prepared stock standard solutions be obtained rather than handling pure explosives and propellant material. However, if the laboratory routinely handles these types of compounds, they may be prepared as follows:

7.4.1 Dry about 0.15 g of the standard for each solid analyte to a constant weight in a vacuum desiccator in the dark at ambient temperature.

**WARNING:** HMX, RDX, Tetryl, PETN, and 2,4,6-TNT are explosives and the neat material must be handled carefully. HMX, RDX, and Tetryl neat materials are shipped under water. Drying at ambient temperature in a vacuum desiccator requires several days. **DO NOT DRY AT ELEVATED TEMPERATURES!**

**NOTE:** The NG standard is a solution and should not be dried.

7.4.2 Place about 0.100 g (weighed to 0.0001 g) of a single analyte into a 100-mL volumetric flask and fill to volume with acetonitrile. The NG standard is a solution of NG in acetone, which should be diluted with acetonitrile.

7.4.3 Invert the flask several times until the analyte is dissolved. Store this stock solution in a refrigerator at  $\leq 4^{\circ}\text{C}$  in the dark. Stock solutions may be used for up to one year.

7.4.4 Calculate the concentration of the stock solution from the actual weight used (nominal concentration = 1000 mg/L).

#### 7.5 Preparation of intermediate and working standard solutions

7.5.1 The actual analytes used in the calibration mixture should be tailored to what is expected at the site being investigated. Calibration standard A, in Sec. 7.5.2, contains the analytes that are most commonly found. If the analysis of all method analytes is necessary, then the standards should be prepared as described in Sec. 7.5.2. The known co-eluting pairs for the two recommended primary GC columns are listed below:

GC Column	Co-eluting Compounds
HP-5	DNA and 4-Am-DNT
DB-1	DNA and 4-Am-DNT RDX and PETN

Any combination of analytes other than those listed in Sec. 7.5.2. should avoid having these pairs in the same standard. These coeluting compounds may be reported as the total of two compounds, unless one of the compounds is known not to be present at a site.

7.5.2 Prepare two intermediate standard solutions by combining appropriate volumes of the various stock solutions. The ECD response is dependent on the number of nitro groups. The response is greatest for 2,4,6-TNT and least for the nitrotoluenes (see Table 2). These solutions should be stored in a freezer at  $\leq -10^{\circ}\text{C}$ .

Intermediate Stock Solution A		Intermediate Stock Solution B	
Analyte	Concentration ( $\mu\text{g/L}$ )	Analyte	Concentration ( $\mu\text{g/L}$ )
1,3-DNB	1000	NB	5000
2,6-DNT	1000	3-NT	5000
2,4-DNT	1000	2-NT	5000
1,3,5-TNB	1000	4-NT	5000
2,4,6-TNT	1000	NG	5000
RDX	1000	PETN	5000
4-Am-DNT	1000	3,5-DNA	1000
2-Am-DNT	1000		
Tetryl	1000		
HMX	1000		

7.5.3 Prepare at least five calibration standard solutions covering a range from 0.5 to 10  $\mu\text{g/L}$  for solution A, and 2.5 to 50  $\mu\text{g/L}$  for solution B, or other project-specific range, by diluting the two intermediate stock solutions with acetonitrile. The calibration standards should be stored in a freezer at  $\leq -10^{\circ}\text{C}$ , and may be used for up to 30 days. Suggested concentrations for solution A are 0.5, 1.0, 2.0, 5.0, 10.0  $\mu\text{g/L}$  and for solution B are 2.5, 5.0, 10, 25, 50  $\mu\text{g/L}$ . It may be necessary or expedient to extend the calibration concentrations higher and/or lower depending on the linear calibration range of the specific detector in use. However, the analyst must also ensure that the calibration curve extends low enough to include all project-specific quantitation limits.

**NOTE:** The analytes 1,3,5-TNB, 2,4,6-TNT, and tetryl are unstable at low concentrations in some brands of acetonitrile. If the response for these analytes declines with time, another brand of acetonitrile should be used.

## 7.6 Preparation of surrogate spiking solution

The analyst should monitor the performance of the extraction and analytical system, as well as the effectiveness of the method in dealing with each sample matrix, by spiking each sample, standard, and reagent water blank with one or two surrogates (i.e., analytes not expected to be in the sample but having properties similar to the target analytes). Suggested surrogates are 2,5-DNT and 3,4-DNT. It is recommended that both be used. This will minimize quantitation problems caused by interference peaks since one peak should be resolved sufficiently for accurate quantitation. 2-Methyl-4-nitroaniline has also been

suggested as a possible surrogate compound. Prepare a surrogate standard concentration of 0.25 g/L. Spike 1 liter of reagent water with 1.0 mL of this standard. This will result in a concentration of 0.05 g/L in the final 5.0 mL acetonitrile eluate (membrane filter method) assuming 100% recovery. Store standards in the dark at  $\leq -10^{\circ}\text{C}$ , or as recommended by the standard manufacturer.

#### 7.7 Preparation of matrix spiking solutions

It is recommended that two matrix spiking solutions be prepared in acetonitrile such that the concentration in the sample is:

0.2  $\mu\text{g/L}$  for 1,3-DNB, 2,6-DNT, 2,4-DNT, 1,3,5-TNB, 2,4,6-TNT, RDX, 4-Am-DNT, 2-Am-DNT, tetryl, and DNA

1.0  $\mu\text{g/L}$  for NB, 3-NT, 2-NT, 4-NT, NG, and PETN

2.0  $\mu\text{g/L}$  for HMX

All target analytes for a given project should be included in the matrix spiking solutions. Because RDX and PETN co-elute on the DB-1 column, these analytes should be in separate spiking solutions (A and B) when the DB-1 column is employed. Follow the same guidance outlined in Secs. 7.5.1 and 7.5.2 to determine what analytes to include in which matrix spiking solution. It would be expected that the same analytes in the calibration standard(s) are also present in the matrix spike standard(s). Store standards in the dark at  $\leq -10^{\circ}\text{C}$ , or as recommended by the standards manufacturer.

### 8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

8.1 Follow the conventional sampling and sample handling procedures for semivolatile organics described in Chapter Four, Organic Analytes, Sec. 4.1.

8.2 Sample extracts should be stored in the dark at  $\leq 4^{\circ}\text{C}$ .

8.3 Soil samples may be contaminated, and should therefore be considered hazardous and handled accordingly. See Sec. 5.0 for additional safety considerations.

### 9.0 QUALITY CONTROL

9.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation techniques can be found in Method 3500. Each laboratory should maintain a formal quality assurance program. The laboratory should also maintain records to document the quality of the data that are generated.

9.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification, and chromatographic analysis of samples.

### 9.3 Initial demonstration of proficiency

9.3.1 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

9.3.2 It is suggested that the spiking solution used to prepare the samples for this demonstration of proficiency contain each analyte of interest at the concentrations listed for the matrix spike in Sec. 7.7. See Method 8000, Sec. 8.0, for additional information on how to accomplish this demonstration.

9.3.3 Calculate the average recovery and the standard deviation of the recoveries of the analytes in each of the four QC reference samples. Refer to Sec. 8.0 of Method 8000 for procedures for evaluating method performance.

9.4 Sample quality control for preparation and analysis - The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy). At a minimum, this includes the analysis of QC samples including a method blank and a laboratory control sample (LCS) in each analytical batch, the addition of surrogates to each field sample and QC sample, and routine analyses of matrix spike and matrix spike duplicate aliquots.

9.4.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratories should use a matrix spike and matrix spike duplicate pair. The compounds in solution A (see Sec. 7.5.2) are more commonly found at arsenals that work with the finished product. Therefore, unless the site is expected to contain compounds found in solution B, use the compounds in solution A to prepare routine matrix spike/matrix spike duplicate pairs (see Sec. 7.7).

9.4.2 A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

9.4.3 Surrogate recoveries - The laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0, for information on evaluating surrogate data and developing and updating surrogate limits.

9.4.4 In-house method performance criteria should be developed using the guidance found in Sec. 8.0 of Method 8000.

9.5 It is recommended that the laboratory adopt additional quality assurance/quality control practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

## 10.0 CALIBRATION AND STANDARDIZATION

10.1 The GC column should be baked at the injection port temperature until the baseline is stable. (The injection port temperature should not be set higher than the maximum column temperature recommended by the column manufacturer).

NOTE: Because of the thermal lability of some of these analytes, reliable quantitation is very dependent on the condition of the GC system. The injection port and column should always be cleaned prior to performing the initial calibration. This is especially true for the nitramines and for HMX in particular.

10.2 Prepare calibration standards as described in Sec. 7.5. All standards should be kept refrigerated at *all* times, *including* while the standards are in the autosampler. If a refrigerated autosampler is used, it should be maintained at  $4 \pm 2^{\circ}\text{C}$ . If manual injections are used, keep the standards refrigerated until just before the injection is made.

NOTE: 1,3,5-TNB, 2,4,6-TNT and tetryl are unstable at low concentrations in some brands of acetonitrile. If the responses for these analytes decline with time, another brand of acetonitrile should be used.

10.3 Establish the GC operating conditions appropriate for the GC column being utilized and the target analytes specified in the project plan. Optimize the instrumental conditions for resolution of the target analytes and sensitivity. Suggested operating conditions are given below for the columns recommended in Sec. 6.2. Table 1 presents examples of analyte retention times at different linear velocities for some of the primary and confirmation columns. Figures 1 through 3 illustrate the potential effects of flow rate on the separation and retention times of the target analytes. Use these figures as guidance in selecting the appropriate linear velocity for the target analytes in the project plan. If all target analytes are to be included, then the lowest linear velocity is recommended.

NOTE: Once established, the same operating conditions must be used for both calibrations and sample analyses.

### 10.3.1 Suggested GC operating conditions for the recommended primary columns

#### HP-5 column

---

Injection Port Temperature:	250°C
Injection volume:	1 µL
Carrier gas:	Hydrogen (flow rate 15 mL/min)
Makeup gas:	Nitrogen (flow rate 30 mL/min)
Detector temperature:	300°C
Temperature Program	
Initial temperature:	100°C for 2 minutes
1st temperature ramp:	10°C per minute to 200°C
2nd temperature ramp:	20°C per minute to 250°C
Final hold:	5.5 minutes

#### DB-1 column

---

Injection Port Temperature:	250°C
Injection volume:	1 µL
Carrier gas:	Hydrogen (linear velocity 40-125 cm/sec)
Makeup gas:	Nitrogen (flow rate 38 mL/min)
Detector temperature:	300°C
Temperature Program	
Initial temperature:	100°C for 2 minutes
1st temperature ramp:	10°C per minute to 200°C
2nd temperature ramp:	20°C per minute to 250°C
Final hold:	5 minutes

**NOTE:** Peak resolution is greatest at low linear velocity, but GC response for some analytes is greatest at high linear velocity (see Table 2). The linear velocity should be chosen based on the objectives of the analysis. A mid-range linear velocity of about 80 cm/s will be suitable for most analyses unless nitrobenzene and the nitrotoluenes are to be included and solvent peak broadening interferes.

**NOTE:** Other carrier gases used routinely with an ECD are acceptable. However, the use of hydrogen provides the best peak resolution. The retention times, chromatograms, and data presented in this method were developed with hydrogen.

10.3.2 Suggested GC operating conditions for the recommended confirmation columns

RTX-200 column (low linear velocity option)

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Injection Port Temperature:	250°C
Injection volume:	1 µL
Carrier gas:	Hydrogen (linear velocity 40 cm/sec)
Detector temperature:	290°C
Temperature Program	
Initial temperature:	100°C for 1.2 minutes
1st temperature ramp:	5°C per minute to 140°C
2nd temperature ramp:	1°C per minute to 160°C
3rd temperature ramp:	20°C per minute to 250°C

RTX-200 column (high linear velocity option)

---

Injection Port Temperature:	270°C
Injection volume:	1 µL
Carrier gas:	Hydrogen (linear velocity 122 cm/sec)
Detector temperature:	290°C
Temperature Program	
Initial temperature:	150°C for 1 minute
Temperature ramp:	20°C per minute to 250°C and hold

RTX-225

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Injection Port Temperature:	220°C
Injection volume:	1 µL
Carrier gas:	Hydrogen (linear velocity 108 cm/sec)
Detector temperature:	250°C
Temperature Program	
Initial temperature:	100°C for 2 minutes
Temperature ramp:	10°C per minute to 220°C
Final hold:	8 minutes

10.4 Because of the low concentration of standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more. Therefore, the GC column should be primed (or deactivated) by injecting a standard mixture approximately 20

times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

10.5 A 1- $\mu$ L injection volume of each calibration standard is recommended.

#### 10.6 Initial calibration

Inject each of the calibration standards, in order from lowest to highest concentration, and obtain peak heights or peak areas for each analyte. Follow the calibration procedures outlined in Method 8000, Sec. 7.0. Normally, since this is an ECD method, external standard calibration is used along with the linear calibration model. However, other calibration models may be used, as appropriate. Calculate calibration factors (CFs) and ensure that all target analytes meet the recommended calibration criterion of  $\leq 20\%$  RSD, or other criteria appropriate for the specific project.

#### 10.7 Calibration verification

10.7.1 See Method 8000, Sec. 7, for detailed instructions on calibration verification. Include a mid-level calibration standard after each group of 20 samples in the analysis sequence as a calibration check. (It is *recommended* that a calibration standard be included after every 10 samples to minimize the number of repeat injections). Injections of method blanks, matrix spike samples, and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total. If all target analytes are included in a given project, it will be necessary to inject two calibration standards in order to verify the calibration of all the analytes. If the injection of both calibration standards is necessary, alternate the two calibration standards by injecting one set after the first ten samples and the second set after the second 10 samples.

**NOTE:** As mentioned in Sec. 7.5.1, two calibration standards are used because DNA and 4-Am-DNT coelute on both the HP-5 and the DB-1 columns and RDX and PETN co-elute on the DB-1 column. These coeluting compounds may be reported as the total of two compounds, unless one of the compounds is known not to be present at a site.

10.7.2 If there has been a break in analyses since initial calibration was performed, begin by injecting a mid-level calibration standard containing the target analytes. As noted above, this may require the injection of two calibration standards. Ensure that the recommended calibration verification criterion of  $\pm 15\%$  is met before beginning the injection of samples. If the results for the verification standard are close to the control limits, it is highly recommended that injection port and column maintenance be performed and the verification standard be successfully reanalyzed prior to proceeding with sample injections. This should be done to avoid having to reinject all samples if the next calibration verification standard exceeds the criterion. This is especially true if HMX is a target analyte and is close to exceeding control limits.

10.7.3 The calibration factors for the verification should be within  $\pm 15\%$  of the mean calibration factors from the initial calibration (see Sec. 7.0 of Method 8000 for more details). When this calibration verification standard falls out of this acceptance window, the laboratory should stop analyses and take corrective action as outlined in Method 8000, Sec. 7.0, and recalibrate, if necessary.

10.7.4 If quantitation is accomplished using an internal standard, internal standards must be evaluated for acceptance. The measured area of the internal standard must be no more than 50 percent different from the average area calculated during calibration. When the internal standard peak area is outside the limit, all samples that fall outside the QC criterion must be reanalyzed.

10.8 Retention time windows - Absolute retention times are used for compound identification. Retention time windows are crucial to the identification of target compounds, and should be established by one of the approaches described in Method 8000, Sec. 7.0.

10.8.1 Before establishing the retention time windows, make sure the gas chromatographic system is operating within optimum conditions.

10.8.2 The widths of the retention time windows are defined as described in Method 8000. However, the experience of the analyst should weigh heavily in the interpretation of the chromatograms.

## 11.0 PROCEDURE

### 11.1 Sample Extraction

Three procedures for sample extraction are provided elsewhere in SW-846, two for aqueous samples and one for solid samples.

11.1.1 Consult Method 3535 for the solid-phase extraction procedures utilizing either SPE cartridges or SPE disks.

11.1.2 Consult Method 8330 for an ultrasonic procedure for the extraction of soil and solid waste samples.

NOTE: When performing ultrasonic extraction of explosives, it is important to keep the ultrasonic bath at or below ambient laboratory temperature, in order to minimize the thermal degradation of the analytes of interest. This may be accomplished by several means, including by placing a cooling coil in the bath.

11.1.3 Store sample extracts in the freezer until analysis.

### 11.2 Sample analysis by GC/ECD

11.2.1 Sample extracts must be kept refrigerated at all times, in order to minimize the degradation of the analytes of interest. If an autosampler is used, it must be capable of refrigerating the vials (see Sec. 6.3) at  $4 \pm 2^{\circ}\text{C}$ . When using an autosampler, allow the sample extracts to equilibrate to the temperature of the autosampler tray before beginning analyses. If manual injections are used, keep the extracts refrigerated until just before the injection is made.

11.2.2 Deactivate the GC column if the GC has not been used during the past day or so (see Sec. 10.4).

11.2.3 Perform initial calibration (Sec. 10.6) or calibration verification (Sec. 10.7). Beginning an analysis run with calibration verification is acceptable if the calibration verification criterion can still be met. Subsequent calibration verification includes the analysis of additional calibration standards interspersed with sample extracts (see Sec. 10.7). Consult Sec. 7 of Method 8000 for approaches to initial calibration and calibration verification.

11.2.4 Inject about 1  $\mu\text{L}$  of the sample extract into the GC and record the exact volume injected. The same GC operating conditions used for the initial calibration must be employed for the analysis of samples.

11.2.5 Sample injections may continue for as long as the calibration verification standards and the standards interspersed with the samples meet instrument QC criteria (see Sec. 10.7).

## 12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Compare the retention time of each analyte in the calibration standard with the absolute retention time windows established in Sec. 10.8. As described in Method 8000, the center of the absolute retention time window for each analyte is its retention time in the mid-concentration standard analyzed during the initial calibration. Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

12.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. Each tentative identification should be confirmed using either a second GC column of dissimilar stationary phase or using another technique such as GC/MS or HPLC/UV (see Method 8000, Sec. 7).

12.3 For the options on reporting confirmation results, see Sec. 7 of Method 8000.

12.4 For guidance on verification of external standard calibrations, see Sec. 10.7 of this method and Sec. 7 of Method 8000. When performing external standard calibration and a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent misquantitations and possible false negative results. Reinjection of the sample extracts may be required. More frequent analyses of standards will minimize the number of sample extracts that would have to be reinjected if the QC limits are violated for the standard analysis.

However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e., >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the verification standard has demonstrated that the analyte would have been detected if it were present. In contrast, if an analyte above the QC limits was detected in a sample extract, then reinjection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% *below* the initial calibration response, then reinjection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present (i.e., a false negative result).

12.5 The target compounds in this method are known to decompose if the injection port of the GC becomes activated. Therefore, when a calibration verification standard fails, the analyst must evaluate whether the calibration has drifted out the acceptable range due to normal calibration drift or active sites in the injection port causing degradation of the target compounds. Degradation of the target compounds would be expected to lead to lower responses.

When reviewing the results of the calibration verification standard, the analyst should keep in mind the number of samples run and/or whether some very dirty samples may have contaminated the injection port. As noted in Sec. 4.3, the injection port liner must be deactivated again or a commercially-available deactivated liner should be inserted each time the septum is replaced or at least after every 50 injections.

12.6 For guidance on calculating results from internal and external standard calibrations, see Sec. 7.0 of Method 8000. The nominal final extract volumes are 4.0 mL for the cartridge SPE method and 5.0 mL for the disk SPE method, unless the eluate was concentrated further. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined.

12.7 As mentioned in Sec. 7.5.1, DNA and 4-Am-DNT coelute on both the HP-5 and the DB-1 columns and RDX and PETN co-elute on the DB-1 column. These coeluting compounds may be reported as the total of two compounds, unless one of the compounds is known not to be present at a site.

### 13.0 METHOD PERFORMANCE

13.1 Tables 1 and 2 present example retention times and calibration factors obtained on various GC columns and using several different carrier gas velocities. Example chromatograms are presented in Figures 1 through 3 (Reference 3). These data are provided only as examples. Each laboratory must develop retention time data and calibration factors for their application of the procedure.

13.2 Using the disk and cartridge SPE procedures outlined in Method 3535, single-laboratory MDLs for the analytes in solution A (Sec. 7.5.2) ranged from approximately 0.003 to 0.02 µg/L, based on a spiked sample concentration of 0.01 µg/L. For the analytes in solution B, the MDLs ranged from approximately 0.2 to 0.5 µg/L for the analytes spiked at 1 µg/L, and was approximately 0.06 µg/L for 3,5-DNA, which was spiked at 0.2 µg/L.

13.3 Table 3 presents single-laboratory recovery and precision data for the analytes spiked into 500 mL of reagent water. Extraction was by the disk SPE method (see Reference 3).

13.4 Single-laboratory MDLs were determined for spiked soil samples prepared in two different soil matrices. MDLs ranged from approximately 0.7 to 3.5 µg/kg for analytes spiked at 5 µg/kg, and from approximately 10 to 25 µg/kg for analytes spiked at 50 µg/kg.

13.5 Table 4 presents single-laboratory recovery and precision data for the analytes spiked into two different soil matrices. Extraction was performed by the procedure described in Method 8330.

13.6 Tables 5 and 6 present data on the recovery and repeatability of GC/ECD and HPLC/UV determinations of the analyte concentrations in spiked water samples. The HPLC

conditions were those found in Method 8330. The samples were extracted as described in Method 3535 and the extracts were split for GC and HPLC analysis. The analytes in this study represent those most commonly found at arsenals around the country. Data from the disk SPE method are presented in Table 5 and data from the cartridge SPE method are presented in Table 6 (See Reference 2).

## 14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of a waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 For information about pollution prevention that may be applicable to laboratories and research institutions consult *Less is Better: Laboratory Chemical Management for Waste Reduction*, available from the American Chemical Society.

14.3 This method conforms with the USEPA's pollution prevention goals. The cartridge SPE method uses only 34 mL of acetonitrile per sample and the disk SPE method only uses 45 mL of solvent.

## 15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult *The Waste Management Manual for Laboratory Personnel* available from the American Chemical Society at the address listed in Sec. 14.2.

## 16.0 REFERENCES

1. Hable, M., C. Stern, C. Asowata, and K. Williams, The Determination of Nitroaromatics and Nitramines in Ground and Drinking Water by Wide-Bore Capillary Gas Chromatography, *Journal of Chromatographic Science*, 29:131-135 (1991).
2. Walsh, M.E. and T. Ranney, "Determination of Nitroaromatic, Nitramine, and Nitrate Ester Explosives in Water using Solid-phase Extraction and Gas Chromatography-electron Capture Detection: Comparison with High-performance Liquid Chromatography," *Journal of Chromatographic Science*, 36, pp. 406-416 (1998).

3. Walsh, M.E. and T. Ranney, "Determination of Nitroaromatic, Nitramine, and nitrate ester explosives in water using SPE and GC-ECD: Comparison with HPLC," CRREL Report 98-2. U.S. Army Cold Regions Research and Engineering Laboratory, Hanover, NH (1998).

#### 17.0 TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

The pages to follow contain Tables 1 through 6, Figures 1 through 3, and a flow diagram of the method procedure.

TABLE 1

EXAMPLE RETENTION TIMES FOR VARIOUS GC COLUMNS  
OBTAINED AT DIFFERENT CARRIER GAS LINEAR VELOCITIES

Compound	Retention Time (minutes)				
	DB-1 Column		RTX-200 Column		RTX-225 Column
	LV = 126	LV = 44	LV = 40	LV = 122	LV = 108
NB	0.32	1.38	2.15	ES	ND
2-NT	0.47	2.06	2.78	ES	0.95
3-NT	0.57	2.47	3.40	ES	1.20
4-NT	0.62	2.69	3.72	ES	1.40
NG	1.18	3.84	8.57	0.52	6.25
1,3-DNB	1.84	5.05	9.01	0.63	5.86
2,6-DNT	2.07	5.28	8.51	0.55	5.50
2,4-DNT	2.88	6.12	10.64	0.88	6.51
1,3,5-TNB	4.19	7.42	18.90	1.98	9.99
2,4,6-TNT	4.61	7.82	17.81	1.86	9.51
PETN	5.62	8.79	28.52	2.74	11.57
RDX	5.62	8.83	29.19	2.86	13.66
4-Am-DNT	6.77	9.92	23.80	2.45	12.65
3,5-DNA	6.83	10.07	26.08	2.65	13.32
2-Am-DNT	7.17	10.38	28.57	2.85	13.17
Tetryl	8.05	11.26	32.11	3.54	13.65
HMX	11.21	13.92	not eluted	6.29	not eluted

surr = Surrogate

LV = Linear Velocity in cm/s

ES = Elutes in solvent peak

ND = Not Determined

Data are taken from Reference 3

Chromatographic conditions are described in Sec. 10.3

All data are provided as examples only. Each laboratory must determine retention times and retention time windows for their specific application of the method.

TABLE 2

EXAMPLE CALIBRATION FACTORS OBTAINED AT DIFFERENT CARRIER GAS  
LINEAR VELOCITIES USING A DB-1 COLUMN AND 50 µg/L SOLUTIONS

Compound	Calibration Factor	
	LV = 126 cm/s	LV = 44 cm/s
2,4,6-TNT	104	109
2,6-DNT	91	92
2-Am-DNT	80	83
RDX	79	52
HMX	75	32
4-Am-DNT	71	76
DNA	69	69
2,4-DNT	58	58
Tetryl	46	59
1,3,5-TNB	45	50
1,3-DNB	30	28
NB	18	9.9
PETN	17	8.1
NG	12	4.5
3-NT	7.5	7.4
2-NT	5.9	3.6
4-NT	2.5	4.5

The calibration factor is calculated as the peak height divided by the concentration of the standard. All data are provided as examples only. Each laboratory must determine calibration factors for their specific application of the method.

Data are taken from Reference 3

TABLE 3

## RECOVERY OF TARGET ANALYTES FROM WATER USING DISK SPE PROCEDURES

Compound	Spike Conc. ( $\mu\text{g/L}$ )	Mean Recovery (%)	RSD(%)
1,3-DNB	0.2	99	9.7
2,6-DNT	0.2	93	7.1
2,4-DNT	0.2	104	7.6
1,3,5-TNB	0.2	94	7.7
2,4,6-TNT	0.2	116	8.0
RDX	0.2	88	7.2
4-Am-DNT	0.2	75	11.2
2-AM-DNT	0.2	87	11.6
Tetryl	0.2	95	8.3
DNA	0.2	74	9.2
NB	1	97	7.1
2-NT	1	93	5.1
3-NT	1	92	4.6
4-NT	1	90	5.3
NG	1	92	5.5
PETN	1	99	4.8
HMX	2	79	8.1

Mean recovery from seven replicate 500-mL water samples extracted using Empore SDB-RPS disks and with a final volume of 5 mL of acetonitrile.

Data are taken from Reference 3.

TABLE 4

## SINGLE LABORATORY PERFORMANCE DATA FOR EXPLOSIVES IN SOIL

Compound	Spike Level ( $\mu\text{g}/\text{kg}$ )	Ottawa Sand		AEC Soil	
		Mean Recovery (%)	RSD (%)	Mean Recovery (%)	RSD (%)
1,3-DNB	50	106	2.4	102	3.4
2,6-DNT	50	107	1.8	105	3.6
2,4-DNT	50	108	1.9	105	3.4
TNB	50	126	4.3	91	19.6
TNT	50	120	2.6	107	3.9
RDX	50	118	6.9	93	11.4
4-Am-DNT	50	106	4.8	99	7.1
3,5-DNA	50	111	5.6	100	8.1
2-Am-DNT	50	113	5.2	107	7.3

Seven soil samples were extracted using the approach described in Method 8330.

Sample Prep: 2.00 g soil (AEC or Ottawa Sand) spiked at either 5 or 50  $\mu\text{g}/\text{kg}$ , extracted with 10 mL acetonitrile by 18-hour ultrasonic extraction in a water-cooled ultrasonic bath. Filtration of extract through Millex SR filter unit.

TABLE 5

COMPARISON OF GC/ECD AND HPLC/UV DETERMINATIONS  
OF SPIKED WATER SAMPLES USING EMPORE SDB-RPS 47-mm SPE DISKS

Compound	Spiked Conc. ( $\mu\text{g/L}$ )	Conc. Found ( $\mu\text{g/L}$ )		Mean Rec. (%)	RPD (%)
		Replicate 1	Replicate 2		
GC/ECD result					
1,3-DNB	5.06	4.77	4.35	90	9.4
2,6-DNT	5.08	4.88	4.48	92	8.7
2,4-DNT	5.12	4.78	4.50	91	6.1
1,3,5-TNB	5.04	4.33	4.25	85	1.7
2,4,6-TNT	5.01	4.72	4.63	93	1.9
RDX	10.0	9.55	9.32	94	2.4
4-Am-DNT	5.06	4.51	4.28	87	5.1
2-Am-DNT	5.02	5.74	5.22	109	9.5
HMX	50.1	49.7	47.0	96	5.6
HPLC/UV result					
1,3-DNB	5.06	5.45	5.26	106	3.7
2,6-DNT and 2,4-DNT <sup>†</sup>	10.2	10.6	10.2	102	3.9
1,3,5-TNB	5.04	5.62	5.18	107	8.1
2,4,6-TNT	5.01	6.04	5.48	115	9.8
RDX	10.0	10.3	10.3	103	0.2
4-Am-DNT and 2-Am-DNT <sup>†</sup>	10.1	10.8	10.3	105	5.2
HMX	50.1	45.9	46.9	93	2.2

<sup>†</sup> Peaks are not resolved and the results are reported as the total of the two compounds.

Data are taken from Reference 2.

TABLE 6

COMPARISON OF GC/ECD AND HPLC/UV DETERMINATIONS OF SPIKED WATER  
SAMPLES USING WATERS SEP-PAK RDX SPE CARTRIDGES

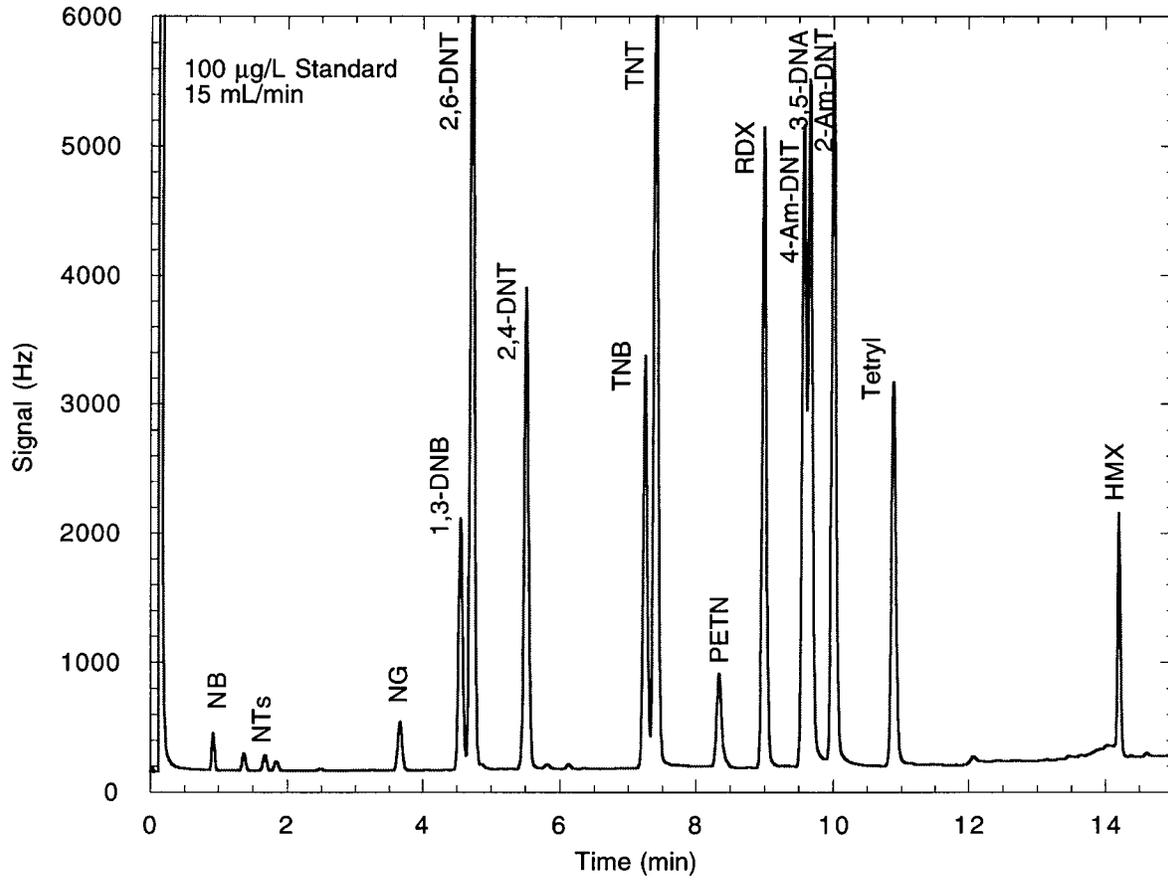
Compound	Spiked Conc. ( $\mu\text{g/L}$ )	Conc. Found ( $\mu\text{g/L}$ )		Mean Rec. (%)	RPD (%)
		Replicate 1	Replicate 2		
GC/ECD result					
1,3-DNB	5.06	5.20	4.66	98	11.1
2,6-DNT	5.08	5.29	4.87	100	8.3
2,4-DNT	5.12	5.03	4.80	96	4.6
1,3,5-TNB	5.04	4.92	4.73	96	3.8
2,4,6-TNT	5.01	5.26	5.07	103	3.7
RDX	10.0	10.8	10.6	106	1.8
4-Am-DNT	5.06	5.05	4.58	95	9.6
2-Am-DNT	5.02	5.26	4.85	101	8.1
HMX	50.1	68.8	67.7	136	1.6
HPLC/UV result					
1,3-DNB	5.06	5.76	5.70	113	1.1
2,6-DNT and 2,4-DNT <sup>†</sup>	10.16	11.0	11.0	108	0.3
1,3,5-TNB	5.04	5.71	5.67	113	0.7
2,4,6-TNT	5.01	5.97	5.99	119	0.4
RDX	10.0	12.5	12.1	123	3.3
4-Am-DNT and 2-Am-DNT <sup>†</sup>	10.1	10.6	10.6	105	0.4
HMX	50.1	55.5	56.2	111	1.3

<sup>†</sup> Peaks are not resolved and the results are reported as the total of the two compounds.

Data are taken from Reference 2.

FIGURE 1

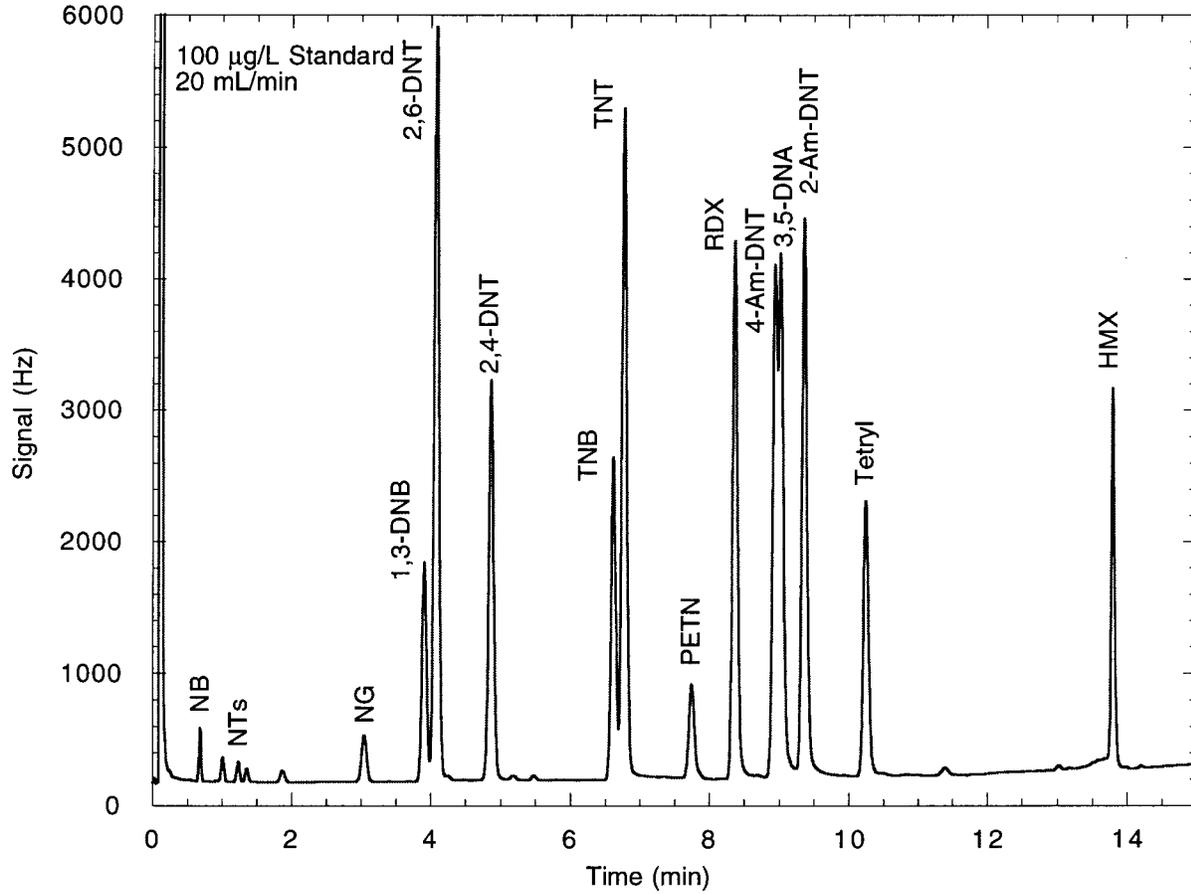
CHROMATOGRAM OF A 100 µg/L STANDARD ON AN HP-5 GC COLUMN  
AT A FLOW RATE OF 15 mL/MIN HYDROGEN CARRIER GAS



GC operating conditions are listed in Sec. 10.3.1.

FIGURE 2

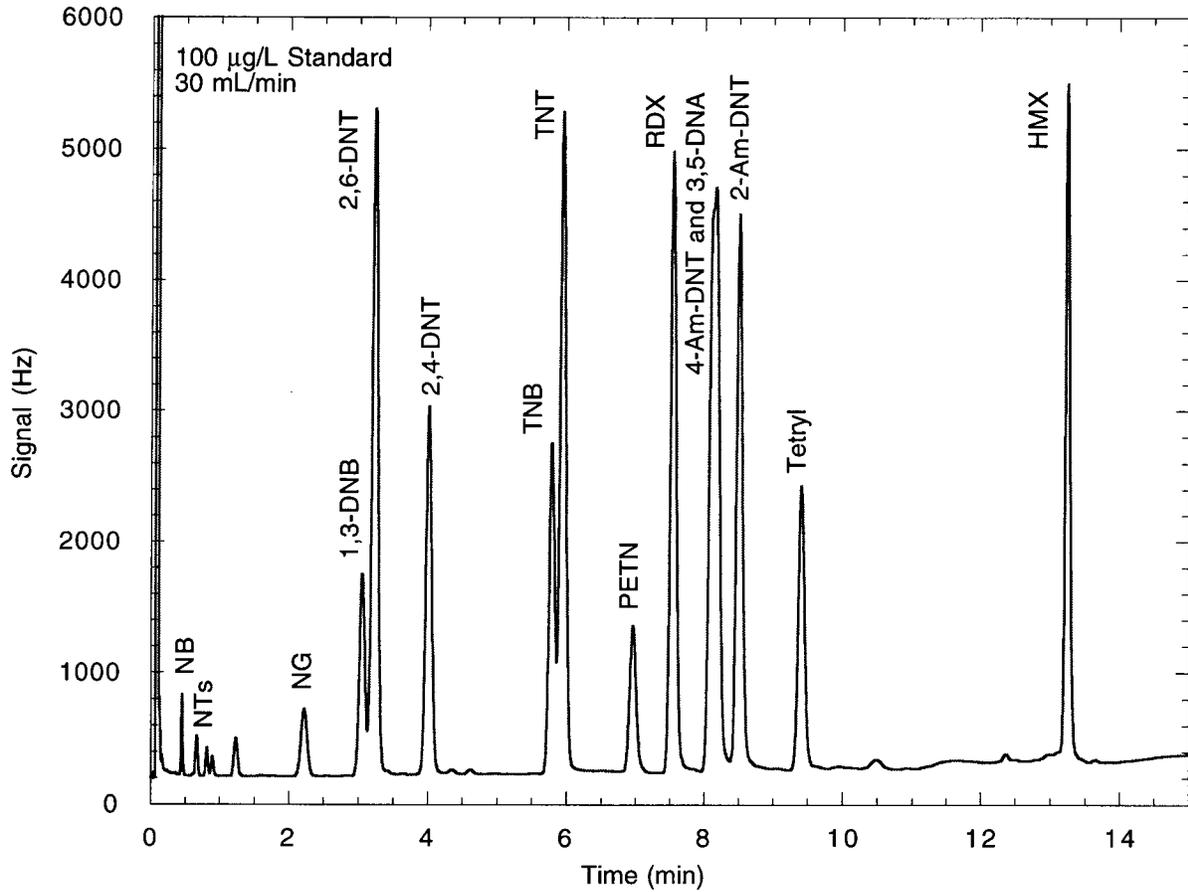
CHROMATOGRAM OF A 100 µg/L STANDARD ON AN HP-5 GC COLUMN  
AT A FLOW RATE OF 20 mL/MIN HYDROGEN CARRIER GAS



GC operating conditions are listed in Sec. 10.3.1. Note the effect of the flow rate on the separation of 4-Am-DNT and 3,5-DNA, as well as on the retention time of HMX.

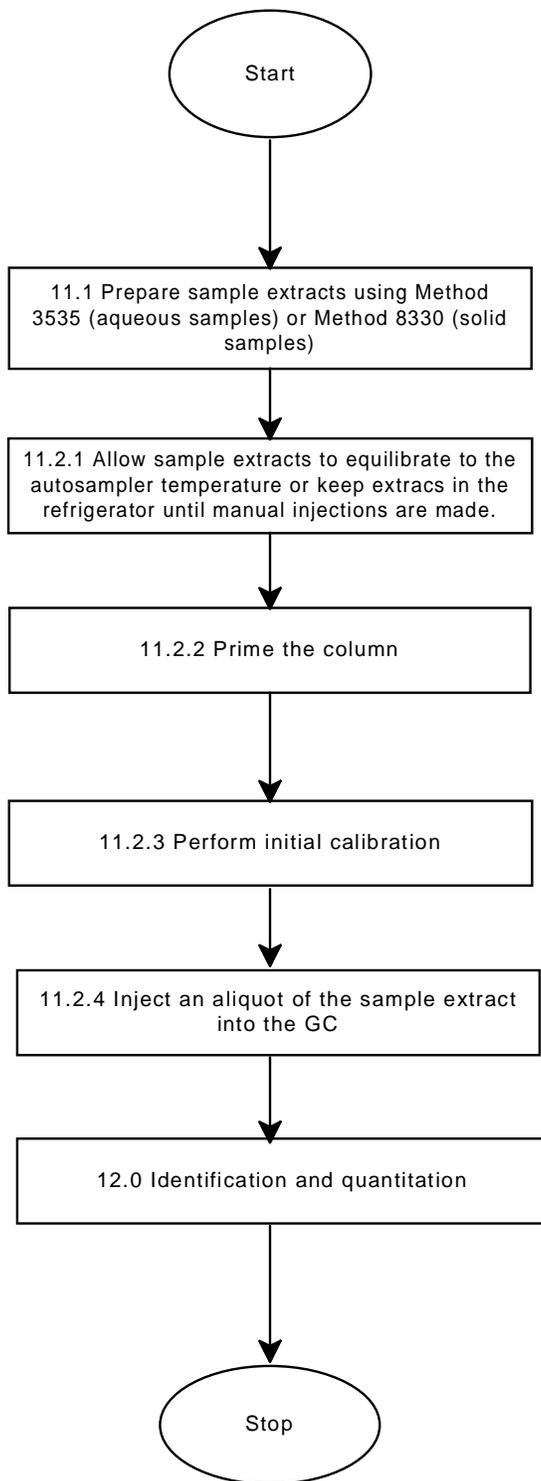
FIGURE 3

CHROMATOGRAM OF A 100 µg/L STANDARD ON AN HP-5 GC COLUMN  
AT A FLOW RATE OF 30 mL/MIN HYDROGEN CARRIER GAS



GC operating conditions are listed in Sec. 10.3.1. Note the effect of the flow rate on the separation of 4-Am-DNT and 3,5-DNA, as well as on the retention time of HMX.

METHOD 8095  
EXPLOSIVES BY GAS CHROMATOGRAPHY



APPENDIX E

## **Site Visit Log**

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# AIR MONITORING SITE VISIT LOG

FORMER VIEQUES NAVAL TRAINING RANGE

**NOTE ANY METEOROLOGICAL CONDITIONS AND/OR ACTION IN THE LIA/SIA OR REGION THAT WOULD AFFECT AIR QUALITY BELOW:**

(Examples: fire, volcanic activity, BIPs, surface activity)

## E-BAM Data

	OP-1	BOAT HOUSE	OP-5
Date			
Time			
Filter Tape Last Replaced ? Estimated remaining <small>(Annotate tape w/date&amp;time)</small>			
Tape sample removed? <small>(Annotate tape w/date&amp;time)</small>			
Battery Check <small>(If &lt; 10.5 Volts Change out)</small>			
Wind Speed			
Wind Direction			
Data Download			
Error Log Download			
Last Hour <small>(Note time)</small>			
Last Hour EBAM Concentration			
Leak Check: monthly <small>(Acceptance criteria&lt;1.5 lpm)</small>			

STANDARD	MAKE/MODEL	SERIAL NO.	CERTIFICATION DATE
Flow meter	BGI Inc, Delta-Cal	396	15-NOV-2004
Temperature	BGI Inc, Delta-Cal	396	15-NOV-2004
Pressure	BGI Inc, Delta-Cal	396	15-NOV-2004



APPENDIX F

## **Audit Data Sheet**

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E-BAM  
CALIBRATION/FLOW CHECK/AUDIT  
FORMER VIEQUES NAVAL TRAINING RANGE

<b>Calibration Date:</b>	<b>Time Started:</b>	<b>Time Finished:</b>
<b>Field Operator:</b>	<b>Auditor:</b>	
<b>Station/Site #:</b>	<b>Monitor S/N:</b>	<b>Last Cal:</b>

**INSTRUMENT CHECKS**

<b>Leak Check: (<math>\leq 1.5</math> LPM)</b>	
<b>Pressure, Temperature and Flow Transfer Standard (name &amp; model): BGI Inc, Delta-Cal</b>	
<b>S/N: 396</b>	<b>Cert. Date: 15-Nov-2004</b>
<b>True Pressure:</b>	<b>E-BAM Display:</b>
<b>True Temperature:</b>	<b>E-BAM Display:</b>
<b>True Flow:</b>	<b>E-BAM Display:</b>

**CALIBRATIONS**

<b>Temperature Sensor Calibration</b>			
E-BAM Temp. Display:	Standard Temperature:	Diff. in Degrees:	Adjusted E-BAM Temp:

Temperature criteria  $\leq \pm 2.0$  degrees

<b>Pressure Sensor Calibration</b>			
E-BAM Press Display:	Standard Pressure:	Diff. In Pressure:	Adjusted E-BAM Press:

Pressure criteria  $\leq \pm 10$  mm Hg

<b>Flow Calibration</b>			
E-BAM Flow Display:	Standard Flow:	% Difference:	Adjusted E-BAM Flow:

Flow range criteria  $\leq \pm 2.0$  %

<b>Flow Verification/Audit</b>				
E-BAM Display:	Standard Reading:	% Difference:	Verifi. Criteria	Audit Criteria
			Flow $\leq \pm 2.0$ % (16.34 to 17.00)	Flow $\leq \pm 4.0$ % (16.00 to 17.34)

<b>Comments:</b>

APPENDIX G

## Sample Data Report

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Location 5  
Name Boathouse  
SN 4353

Date	ConcRT (mg/m3)	ConcHr (mg/m3)	Flow (l/m)	WS (m/s)	WD (Deg)	AT(C)	RHx(%)	RHi(%)	BV(V)	FT(C)	Alarm
8/3/05	0.015	0.012	16.5	4.3	121	23.7	69	51	11.6	33.1	0
8/3/05	-0.005	0.012	11.9	4.8	125	28.6	72	51	11.9	33	1
8/3/05	0.015	0.012	16.4	5	122	27.3	71	49	11.7	33.9	0
8/3/05	0.097	0.012	16.4	5.1	120	26.2	69	48	11.8	34.4	0
8/3/05	-0.005	0.048	16.4	4.8	119	26.9	65	46	11.9	35.2	0
8/3/05	0.025	0.048	16.3	4.6	123	28.2	63	44	12.1	34.8	0
8/3/05	0.024	0.048	16.3	4.8	121	28	61	45	12.1	34.3	0
8/3/05	0.014	0.048	16.4	4.7	120	27.9	52	44	12.2	34.7	0
8/3/05	0.012	0.001	16.3	4.8	120	28.3	51	44	12.2	34.6	0
8/3/05	0.065	0.001	16.3	5	117	28	51	44	12.3	34.4	0
8/3/05	-0.005	0.001	16.4	5.3	116	27.8	48	44	12.3	34.3	0
8/3/05	0.01	0.001	16.4	5.4	116	27.6	49	44	12.3	34.1	0
8/3/05	0.005	0.011	16.4	5.5	117	27.3	47	44	12.3	34.1	0
8/3/05	-0.005	0.011	15.8	5.7	118	27.7	50	45	12.4	34	0
8/3/05	0.028	0.011	16.4	5.7	119	27.7	49	44	12.4	33.8	0
8/3/05	0.024	0.011	16.3	5.6	119	27.8	48	44	12.4	33.6	0
8/3/05	0.008	0.007	16.4	5.3	118	27.8	47	44	12.4	34	0
8/3/05	0.106	0.007	16.3	5.4	116	27.9	47	45	12.4	33.9	0
8/3/05	-0.005	0.007	16.4	5	116	26.8	47	45	12.3	34.8	0
8/3/05	0.057	0.007	16.3	4.9	119	28.4	49	43	12.5	34.4	0
8/3/05	0.02	0.024	16.3	5	117	28.3	48	44	12.5	34.2	0
8/3/05	0.035	0.024	16.4	4.9	114	27	47	45	12.3	34.4	0
8/3/05	-0.005	0.024	16.4	4.9	115	27.2	48	44	12.4	35.4	0
8/3/05	0.045	0.024	16.3	5.1	115	28	49	44	12.4	34.6	0
8/3/05	0.016	0.015	16.3	5.1	116	27.7	48	45	12.4	34.2	0
8/3/05	0.061	0.015	16.4	4.9	113	27.3	46	45	12.2	34.8	0
8/3/05	-0.005	0.015	16.4	4.7	113	27.2	47	45	12.3	34.9	0
8/3/05	0.035	0.015	16.3	4.6	118	28.6	47	45	12.3	34.7	0