AIR QUALITY MONITORING AND CONTROL PLAN

Kerr-McGee Chemical Corp.—Navassa Superfund Site

Navassa, North Carolina
EPA ID #NCD980557805

Prepared for
Greenfield Environmental Multistate Trust LLC
Trustee of the Multistate Environmental Response Trust

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ACRONYMS AND ABBREVIATIONS

ACM	asbestos-containing material
BaP	benzo[*a*]pyrene
BMP	best management practice
COC	constituent of concern
CQA	construction quality assurance
HASP	health and safety plan
Multistate Trust	Greenfield Environmental Multistate Trust LLC
OSHA	Occupational Safety and Health Administration
OU	operable unit
PAH	polycyclic aromatic hydrocarbon
PCP	pentachlorophenol
PID	photoionization detector
PM10	particles with an aerodynamic diameter 10 microns or smaller
ppm	part per million
$RBSL_{aa}$	risk-based screening level for ambient air
SDS	safety data sheet
Site	Kerr-McGee Chemical Corp.—Navassa Superfund site
TCDD	2,3,7,8-tetrachlorodibenzo-*p*-dioxin
TEQ	toxic equivalency
VOC	volatile organic compound

1 INTRODUCTION

This Air Quality Monitoring and Control Plan has been prepared for the Greenfield Environmental Multistate Trust LLC, Trustee of the Multistate Environmental Response Trust (Multistate Trust) in support of planned remediation activities at the Kerr-McGee Chemical Corp.—Navassa Superfund Site (Site) in the Town of Navassa, Brunswick County, North Carolina (Figure 1). The remediation work will be completed in accordance with the Operable Unit 2 (OU2) Record of Decision, and the 2011 Consent Decree and Environmental Settlement Agreement between the Multistate Trust, the U.S. Environmental Protection Agency, the North Carolina Department of Environmental Quality, and other parties, and will comply with the Comprehensive Environmental Response, Compensation and Liability Act, and relevant regulations and guidance.

The purpose of this plan is to minimize the mobilization of potential constituents of concern (COCs) (as described below) in fugitive dust and vapors, and to protect the surrounding community and the environment during remedy implementation. This plan describes how air hazard mitigation will be accomplished and documented. Major components include dust and volatile organic compound (VOC) control techniques; best management practices (BMPs); dust, VOC, and meteorological monitoring; documentation; and communications. Dust and/or VOC mitigation/control measures, BMPs, and dust and VOC monitoring will be implemented during all remedial activities that involve disturbance or handling of contaminated soil or other activities with the potential to generate fugitive dust or VOCs.

This Air Quality Monitoring and Control Plan has been prepared for the remedial action chosen for OU2. OU2 includes surface soils with COCs consisting of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) toxic equivalency (TEQ), benzo[*a*]pyrene (BaP), BaP TEQ, naphthalene, and pentachlorophenol (PCP). Remedial action objectives and cleanup levels have been developed for OU2 to prevent unacceptable risks to future residents, commercial/industrial workers, construction workers, recreators, or ecological receptors that may come into contact with surface soil. The remedial action for OU2 will involve excavation of 1.6 acres of surface soil to a depth of 1–2 ft below the ground surface and stockpiling of the soils in OU4 at the south end of the facility.

The air will be monitored during the active excavation and stockpiling phases of the OU2 remedial action to prevent exposure to COCs through airborne migration, as described in Section 3. Dust and VOC monitoring and mitigation will reduce exposure to OU2 COCs. Dust and VOC exposure threshold values that trigger mitigation and stop work requirements will be in place and will be protective of human health, as described in Section 3. The program will protect both onsite workers and offsite sensitive receptors. Because the most sensitive receptors (children) were used as the basis of action level calculations, the measures will be protective of other receptors.

2 ROLES AND RESPONSIBILITIES

Dust and VOC monitoring will be conducted by the Multistate Trust's construction quality assurance (CQA) team. A CQA representative will be present during all earthmoving and other activities capable of generating fugitive dust or VOCs to verify that the control measures are followed. The Contractor will be responsible for control measures during the active excavation and stockpiling phases of the OU2 remedial action. The CQA representative will be responsible for gauging the effectiveness of the control measures as applied throughout the workday (e.g., checking for the presence of visual dust plumes and track out, working with the appropriate construction personnel to take corrective actions as needed, and confirming that disturbed surfaces are stabilized at the end of each workday and stockpiles are covered). The CQA representative will be responsible for communication of monitoring results to the Contractor and Owner's Representative.

3 AIR AND METEOROLOGICAL MONITORING

Monitoring of airborne dust and VOCs will be conducted during the active excavation and stockpiling phases of the OU2 remedial action to monitor any migration or possible exposure to the community. Monitoring is effective at assessing potential exposure and migration of dust-borne and vapor-phase compounds, including BaP, TCDD TEQ, BaP TEQ, naphthalene, and PCP (OU2 COCs). An action level is used as a trigger for implementing additional measures to decrease dust and/or VOC generation and migration.

Monitoring techniques and action levels are described below. Action levels are used as triggers for implementing additional measures to decrease emissions and protect the community. This plan focuses primarily on protection of the community and sensitive offsite receptors. Other receptors, including onsite workers, should be protected by these measures as well. Worker exposure monitoring requirements will be included in the Contractor's worker health and safety plan (HASP), which will reference this Air Quality Monitoring and Control Plan.

In addition to the air quality monitoring described in this plan, air monitoring will be conducted by the OU2 Contractor during the removal of asbestos-containing materials (ACM) from OU2. The scope and methods for the ACM air monitoring is described in the ACM monitoring plan prepared by the OU2 Contractor.

3.1 DUST MONITORING AND SAMPLING

Real-time air monitoring will be performed at the Site to assess and monitor dust emissions. Such monitoring will document the effectiveness of measures intended to be protective of human receptors and the environment through dust suppression. Airborne particles are classified by their size, and particles with a diameter of 10 microns or smaller (PM10) pose a greater health risk because they can penetrate deeper into the respiratory system than other particles. As such, emissions will be quantified as PM10 concentrations.

3.1.1 Continuous Perimeter Air Monitoring Stations

Up to three stationary air monitoring stations will be placed at the perimeter of the remedial work areas—one upwind and two downwind—at breathing height. One downwind station will be located downwind of the exclusion zone, as close as is feasible; the second downwind station will be located at the nearest Site perimeter location (example setup location shown in Figure 2). A fourth air monitoring station will be necessary when excavation and stockpile construction are happening concurrently. The placement of air monitoring station locations will be based on proximity to the remedial activities and predominant wind direction. Locations will be identified and adjusted, as needed, following collection of wind speed and direction data (see Section 3.1.2, below).

During working hours, these stations will continuously monitor and provide documentation for potential offsite migration of Site COCs during dust-generating activities. Compliance with the PM10 action level of 150 μg/m^3 will be assessed downwind of the removal work area. Upwind monitoring will be used to assess the source of the dust.

Dust monitoring equipment, such as a TSI DustTrak II, or similar, will be used. A specification sheet for the TSI DustTrak II monitor series is provided in Appendix A. These passive aerosol monitors use single beam nephelometry to measure airborne particles. The monitors will also be capable of collecting information regarding maximum and average daily dust concentrations in a digital form through use of a data logger. Data will be documented daily (Section 5), and quality assurance and quality control will be completed as described in Section 6.

3.1.2 Meteorological Monitoring and Placement of Air Monitoring Stations

The upwind/downwind air monitoring station configuration will allow comparison of real-time conditions. The CQA representative will install an onsite meteorological (weather) station with an anemometer and direction sensor, or similar, and a wireless data-logging console will be erected in the OU4 Site entrance (Figure 2), in a location that will be at least 20 ft away from and 4 ft above area features or other obstructions that could affect ambient airflow patterns.

The CQA representative will check the wind speed and direction throughout the workday to inform the upwind/downwind placement of the continuous monitors, PID measurements, and placement of monitoring badges. Monitoring locations will be moved as necessary. In addition, existing weather station wind rose data from Wilmington International Airport for the work periods will be reviewed prior to the start of work to determine the typical predominant wind direction for the months of work. A wind rose presenting wind data from Wilmington International Airport, Wilmington, North Carolina, is shown in Figure 3. Onsite visual observations can be compared to weather station data to evaluate whether potential correlations exist (e.g., whether rain or wind significantly affects dust generation and migration). Visual observations and recorded wind data during the work period will be documented and reported with dust readings measured during the work (Section 3.1).

3.1.3 Dust Action Level and Calculation of Site-Specific Risk-Based Ambient Air Screening Levels

In the absence of local air quality criteria, potential exposure will be minimized by maintaining dust concentrations below the primary/secondary National Ambient Air Quality Standard for PM10 24-hour standard of 150 μg/m^3. The standard of 150 μg/m^3 measured at the downwind extent of a work area will serve as a stop-work trigger, at which point work will temporarily cease while additional dust control measures are selected and implemented.

The PM10 action level will be protective with respect to potential site-related COC exposures. To evaluate this scenario, Site-specific risk-based ambient air screening levels ($RBSL_{aa}$) were derived for surface soil COCs identified at OU2: BaP TEQ, BaP, TCDD TEQ, naphthalene, and PCP. The $RBSL_{aa}$ are calculated following EPA[1] methodology and assume conservative receptor assumptions. This includes potential inhalation exposure to offsite infant children aged 0 to 2 years. The schedule for OU2 remedial activities anticipates there will be approximately 35 workdays of active excavation and stockpiling. To simulate a conservate exposure scenario, the exposure duration for the calculation of the $RBSL_{aa}$ is based on the length of the intrusive activity of 60 days per year (5 days per week for 12 weeks) for 10 hours per day.[2] The $RBSL_{aa}$ assume a target risk of 1.0×10^{-6} and a hazard quotient of 1.0. Further details regarding how the $RBSL_{aa}$ calculations are derived are found in Appendix B, Table B-1. The maximum surface soil COC concentration at OU2 is conservatively assumed to be equivalent to the COC concentration of the PM10 in ambient air, which is then multiplied by the North Carolina and National Ambient Air Quality 24-hour Standard for PM10 of 150 $\mu g/m^3$ (see Appendix B, Table B-2). This conservative assumption follows the Department of Toxic Substances Control[3] methodology for community air monitoring.

A comparison of the Site COC concentration in ambient air to the $RBSL_{aa}$ for offsite children results in COC concentrations well below the standard. The results are summarized below, and further details are provided in Appendix B, Table B-2.

Ambient Air Screening Summary

		Worst Case Exposure (Based on Maximum COC Concentration in Soil)				
Parameter	Unit	BaP TEQ	BaP	TCDD TEQ	Naphthalene	PCP
Estimated COC concentration in ambient air containing soil particulates	$\mu g/m^3$	0.016	0.010	4.1E-08	3.7E-04	1.7E-04
Site-specific $RBSL_{aa}$ for offsite children (0-2yrs)	$\mu g/m^3$	0.17	0.029	2.7E-05	30	200
Does COC concentration exceed $RBSL_{aa}$?		No	No	No	No	No

Notes:
COC = constituent of concern
$RBSL_{aa}$ = risk-based screening level for ambient air

[1] USEPA. 2024. Regional screening levels (RSLs) – equations. Available at: https://www.epa.gov/risk/regional-screening-levels-rsls-equations.
[2] OU2 contractors anticipate active excavation and stockpile construction will be less than 40 working days.
[3] DTSC. 2020. Community air monitoring plan guidance. California Environmental Protection Agency, Department of Toxic Substances Control. January.

3.1.4 Exceedance of Dust Action Level and Response Actions

If dust monitoring indicates greater than 150 µg/m^3 PM10 average sustained for more than 15 minutes at the downwind remedial work area monitoring station, the following response actions will be implemented:

- Suspending work activities
- Assessing emission source, nature, and concentration, and prevailing weather conditions
- Upgrading worker personal protective equipment as appropriate
- Implementing construction BMPs to control dust (Section 4.1).
- If necessary, working with the Owners' Representative to inform affected adjacent property owners, residents, and the Town of Navassa of potential temporary construction emissions and appropriate precautionary measures.

Additional details regarding these response actions are provided in Section 4.

3.1.5 Particulate Sampling

The CQA team will conduct one dust sampling event in the initial weeks of excavation to confirm assumptions inherent to the dust monitoring approach. Samples will be collected for analysis of PM10 dust and PAHs from the station downwind of the active removal area and downwind of the nearest Site perimeter location.

PM10 will be collected and analyzed following Modified NIOSH 0500 using a personal sampling pump.
Samples will be collected following EPA Method TO-13A for PAHs. A high volume air sample pump (or equivalent) will be set to collect a minimum of 100 m^3 of ambient air over the full workday (8–10 hours). After sample collection is complete, the filter and cartridge will be placed in clean, sealed containers and submitted to Eurofins for analysis. Sample collection procedures and analytical methods are provided in Appendix C.

3.2 VOC MONITORING DURING EXCAVATION AND STOCKPILE CONSTRUCTION

The CQA representative will conduct VOC monitoring in the work area during active excavation and stockpile construction activities. A handheld MiniRae 3000 (or similar) photoionization detector (PID) will be used to monitor for total VOCs. The PID will have a detector lamp with the appropriate ionization energy to detect the Site COCs (e.g., 10.6 eV lamp).

The monitoring will include a minimum of a measurement every hour during active excavation or soil stockpiling. PID monitoring will be also conducted if a chemical odor unrelated to vehicle or generator exhaust is detected by any worker during active excavation or if grossly contaminated material[4] is encountered during OU2 excavation activities. The PID measurements will be recorded from the breathing zone height (~5 ft above the ground surface) within the exclusion zone[5] of the excavation area or stockpile construction area and from a location upwind of the work area to provide ambient background conditions.

The action level for VOCs (avoiding to the extent possible, those associated with equipment emissions) will be 1 part per million (ppm) above background. The 1 ppm action threshold is a minimum value that potentially represents elevated VOC concentrations that can be detected using standard PIDs. If the above-described PID monitoring detects VOCs at 1 ppm or greater, sustained (continuous) monitoring of VOC concentrations within and upwind of the exclusion zone will be initiated. If VOC concentrations in the exclusion zone are sustained above 1 ppm over background for greater than 1 minute, the following actions will be taken:

1. Work will stop and the workers will retreat in an upwind direction.
2. Monitoring badges will be mounted at 5 ft above the ground surface on a post positioned in the exclusion zone, on a post positioned at the Site perimeter downwind of remedial activities, and a post set at an upwind location to record naphthalene and benzene concentrations over a 24-hour period. After 24 hours, the badges will be submitted for laboratory analysis of naphthalene and benzene concentrations. The badges will be replaced each 24-hour period until PID measurements indicate that sustained total VOCs are below 1 ppm. PID measurements will also be recorded at the Site perimeter downwind of remedial activities. The result of the badge measurements will be compared against OSHA permissible exposure limits for worker safety. Information for the monitoring badges is provided in Appendix C.
3. The work operations will be reviewed, and steps will be taken to reduce emissions such that work can continue safely. Steps that may be taken to reduce emissions include placing tarps over excavated material within trucks and stockpiles, and applying water or dust suppressants[6] over some or all of the open work areas and stockpiles in areas of open excavation to control and/or disperse emissions to safe levels. Should concentrations remain elevated, additional personal protective equipment such as

[4] Grossly contaminated material includes nonaqueous-phase liquid (e.g., creosote tar, oil), heavily stained soils, or a sheen.
[5] The location of the exclusion zone will be activity dependent. The area around the active excavation will be considered the exclusion zone during excavation activities, and the stockpile and the area around the stockpile will be considered the exclusion zone during stockpile construction.
[6] The safety data sheet (SDS) for the dust suppressing additive identified by OU2 contractors is provided in Appendix D.

respirators may be used in accordance with the Contractor's HASP, and the areas will continue to be retested with the PID.

4. Work will resume once PID monitoring demonstrates that sustained VOC concentrations are below 1 ppm over background.

3.3 BASELINE MONITORING AND SAMPLING

Baseline monitoring and sampling for dust and VOCs will be conducted prior to the start of potential dust generating activities such as haul road improvements for OU2 remedial activities. Baseline data will be collected from two stations:

- One station located central of OU2 removal areas
- One station located at the OU4 Site entrance, as shown on Figure 2.

The CQA representative will be responsible for baseline monitoring and sampling, which will be conducted using the methods described above. The following data will be collected:

- Dust
  - Dust monitoring at the air monitoring stations for PM10 for 24 hours. The monitoring stations will be checked by the CQA representative each hour during working hours.
  - Two 8-hr baseline particulate samples, to be analyzed for dust (PM10) and PAHs per the methods described in Section 3.1.5.
- VOCs
  - Instantaneous PID readings will be recorded each hour during working hours.
  - Monitoring badges for naphthalene and benzene will be placed at the baseline monitoring locations for 24 hours.

Baseline monitoring and sample results will be used to establish baseline ambient air conditions and inform decision-making during remedial activities.

4 DUST AND VOC EMISSIONS CONTROL

The OU2 remediation activities have the potential to generate fugitive dust. This dust may include airborne COCs. In addition, although the concentrations of VOCs in OU2 soils are low, there is a potential for VOCs to be released to the air during the OU2 remediation activities.

Provisions to prevent generation of airborne COCs and to minimize offsite migration are described in this plan. The overall approach and various mitigation (i.e., control) measures and BMPs that are required or recommended are described in this section. Monitoring, action levels, and instrumentation are outlined in Section 3. Dust and VOC monitoring will be conducted daily by the CQA representative, as described in Section 3. If action levels are exceeded, additional mitigation steps will be taken, as necessary, to further control dust and/or VOC emissions.

The potential for offsite dust or VOC migration, as well as worker exposure, is minimized by measures that include, but are not limited to excavation and loading techniques, maintenance of soil moisture, effective equipment decontamination, and onsite speed limits. Prevention of all dust generation and project-related emissions is not possible, but the Contractor will be required to minimize dust or other emissions during the workday to the maximum extent possible.

BMPs and risk reduction measures to limit dust and VOC production and exposure will be implemented during any construction activity that involves disturbance or handling of soil. The Contractor will be responsible for dust and VOC emission control measures during these construction activities. BMPs for dust and VOC emissions control for anticipated OU2 remedial activities are described below.

4.1 ALL CONSTRUCTION ACTIVITIES

This Air Quality Monitoring and Control Plan will be posted in a visually prominent area at the entrance to the worksite and is to be provided to all construction site personnel (e.g., foreman, equipment operators, water truck operator). The following practices will be adhered to during all construction activities.

Equipment and staffing will be provided by the Contractor during normal working hours for watering of all exposed or disturbed soil surfaces sufficiently to suppress dust plumes and VOC emissions; the wetting will be limited so as to avoid the generation of runoff.

The Contractor conducting earth-moving activities shall:

- Adequately wet to the depth of earth-moving activity and allow time for penetration
- Adequately wet at frequencies to prevent the generation of visible dust plumes.

Dust suppressant additives in the water, which can be a small amount of ordinary liquid detergent or may include use of a calcium-chloride based product,[7] will be used if necessary and in accordance with other state and local regulations.

If necessary, odor suppressing additives[8] will be used to reduce VOC emissions during excavation and handling of the soils.

Adjacent streets will be swept of all soil and debris generated from the remedial activities, as necessary, although avoidance of track-out is preferred.

Inactive areas that have exposed soil surfaces will be covered with plastic sheeting and weighted down on an as-needed basis to avoid the generation of dust or runoff.

Earth-moving or other dust-producing activities will be suspended during periods of high winds whenever dust control measures are unable to prevent visible dust plumes. The Contractor shall cease earth-moving activities if the wind speed is greater than 15 mph averaged over a 15-minute period or where there are instantaneous wind speeds that exceed 25 mph.

If earth-moving activities will not occur for 3 or more consecutive days, exposed stockpiles will be covered with plastic sheeting and other exposed surfaces will be covered with straw or erosion control matting to avoid generation of dust or runoff.

4.2 LOADING AND UNLOADING

During the course of loading and unloading activities, Contractor shall:

- Minimize drop heights and load and unload slowly to help prevent plumes of dust and release of VOCs.
- Use water to control dust generation during transport and handling of materials. Dust-proof chutes may be used to load debris into trucks to help control dust emissions.
- Maintain at least 6 in. of space between the soil and the top of the truck bed while transporting within the Site.
- Empty the loader bucket slowly so that no dust plumes are generated.
- Completely tarp the truck and trailer prior to leaving the Site.
- Empty the trailers slowly so that no dust plumes are generated.

[7] The SDS for the dust suppressing additive identified by OU2 contractors is provided in Appendix D.
[8] The SDS for the odor suppressing additive identified by OU2 contractors is provided in Appendix D.

4.3 DEMOLITION, EXCAVATION, AND GRADING

During the course of demolition, excavation and grading activities, Contractor shall:

- Use water to control dust generation during any demolition of structures or break-up of concrete.
- Use odor suppressing additives[9] as necessary to reduce VOC emissions during excavation and handling of the soils.
- Cover all trucks hauling demolition debris.
- Water all active excavation areas as needed and more often during windy periods; active areas adjacent to the existing land uses shall be kept damp at all times or shall be treated with a non-toxic stabilizer or dust palliative.[10]
- Control water application to minimize runoff.

4.4 HAULING AND HAUL ROUTES

During the course of hauling activities, Contractor shall:

- Post signs at all entrances of the Site to designate the speed limit as 15 mph.
- Stabilize the surface of all vehicular traffic and parking areas by applying gravel.
- Water unpaved access roads as needed and more often during windy periods. Unpaved access roads shall be kept damp at all times or shall be treated with a non-toxic stabilizer or dust palliative.[9]
- Maintain haul routes to help prevent migration of material using methods that include applying crushed rock to cover routes through non-paved areas and laying down plastic sheeting or geotextile to contain material where necessary.
- Implement measures so that haul trucks that may leave the Site will not travel through/within impacted areas.
- Load haul trucks at locations adjacent to the identified impacted excavation areas.
- Immediately remove any spilled soil along haul roads.

[9] The SDS for the odor suppressing additive identified by OU2 contractors is provided in Appendix D.
[10] The SDS for the dust suppressing additive identified by OU2 contractors is provided in Appendix D.

4.5 TRACKOUT

Trackout is any dirt, mud or other debris tracked onto a paved public roadway by vehicles leaving a constructions site. The contractor shall not cause or allow trackout of soils onto an adjacent paved roadway or paved roadway shoulder to the extent practicable.

Contractor shall:

- Remove any trackout each day using a ride-on road sweeper;
- Clean the soil from the exterior of trucks, trailers, and tires prior to the truck leaving the site; and
- Establish a construction entrance/exit maintained in a clean condition, consisting of 4- to 6-in. quarry spalls to a depth of 8 in., and extending at least 20 ft wide and at least 50 ft long.

Minimization of dust generation is the Contractor's responsibility. Excess sources of dust, such as track out of soil onto roadways, will be controlled and removed from the roads as soon as possible. These techniques are expected to control dust generation and offsite migration, as well as to minimize worker exposure.

4.6 STOCKPILING

Excavated soil from OU2 will be directly loaded to trucks and transported to the OU2 soil stockpile areas located in OU4. However, in the event that temporary stockpiling of soil is needed within the excavation areas to facilitate loading and transport of excavated soils, stockpiling will be performed in compliance with the Contract Documents. For all stockpiles, the Contractor shall:

- Maintain stockpiles to avoid steep sides or faces that exceed the angle of repose.
- Maintain individual temporary stockpiles to not contain greater than 400 cubic yards of soil and to not be greater in height than the perimeter fencing and windscreen.
- Segregate clean import material stockpiles from contaminated soil stockpiles.
- Wet down stockpiles during the day as necessary to prevent dust.
- Completely cover stockpiles at the end of each working day with 6-mm-thick plastic sheeting that overlaps a minimum of 24 in. The plastic sheeting shall be anchored and secured so that no portion of the soil is exposed to the atmosphere.
- Inspect covered stockpiles daily. For a covered stockpile, such inspections shall include a visual inspection of all seams and plastic cover surfaces. Immediately restabilize or repair any holes, tears, or any other potential sources of fugitive toxic air contaminant emissions.

5 RECORD KEEPING

Air quality monitoring data will be recorded with instantaneous and continuous measurements during construction activities. Data sheets for documenting air monitoring activities will include the following information:

- Operator name
- Instrument type
- Date/time of last calibration
- General weather conditions (wind speed and direction, temperature, precipitation, cloud cover)
- Location and measurement of background concentration
- Start and end time of monitoring
- Summary of samples collected for analysis
- Location and description of odor observations
- Location and stabilized measurement for elevated readings
- Summary of contractor activities and suspected source of odors or elevated readings (only needed if odors or elevated readings are encountered)
- Follow-up response actions.

At the end of each day, the CQA representative will download logged data from the monitoring stations; data will be included in the remedial action completion report. The CQA representative will securely store monitoring equipment, and charge monitoring equipment in preparation for the following workday.

6 QUALITY ASSURANCE/QUALITY CONTROL

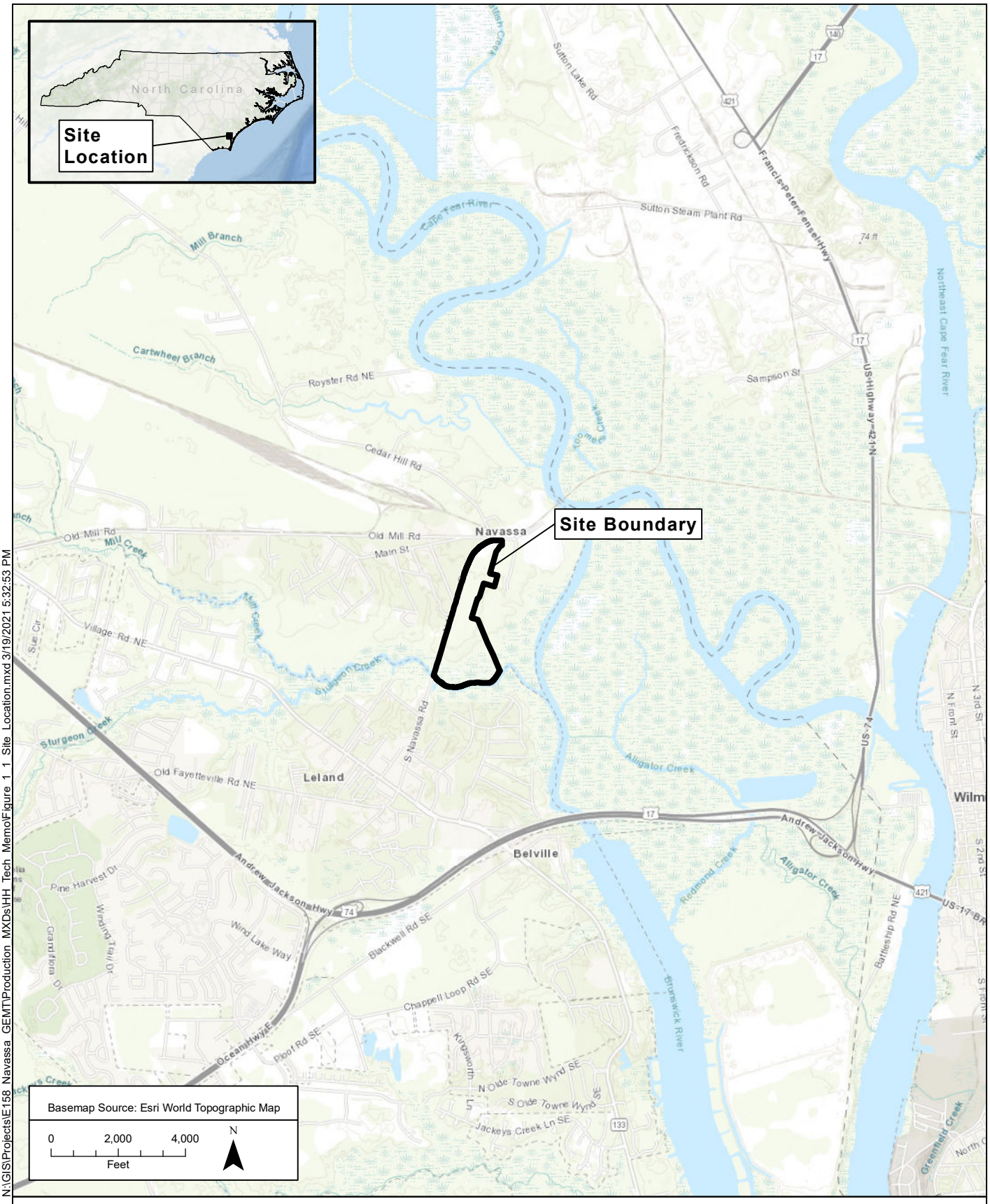
Air monitoring equipment will be calibrated according to manufacturer's instructions and at the recommended frequency, including zeroing sensors with pure air provided by the manufacturer. Calibration results will be documented after each calibration event. Equipment will be fully charged and functioning properly each day. Recalibration may be conducted midday to account for equipment malfunction or changes in environmental parameters such as ambient temperature and humidity. Prior to each use, the monitor response and battery charge will be checked.

7 POINTS OF CONTACT FOR THE COMMUNITY

The main points of contact for any community concerns regarding project environmental issues, including air quality, are provided in the table below. These points of contact will be provided on Site signage procured and installed by the Contractor. Signage will be posted on the outside of the Site informing the public of the Site activities and control measures taking place.

Organization	Personnel	Responsibility	E-mail	Phone
Multistate Trust	Ngozi Ibe	Senior Project Manager	ni@g-etg.com	(919) 695-9582
Multistate Trust	Claire Woods	Director of Environmental Justice Policies and Programs and Senior Attorney	cw@g-etg.com	(323) 204-6943

Figures

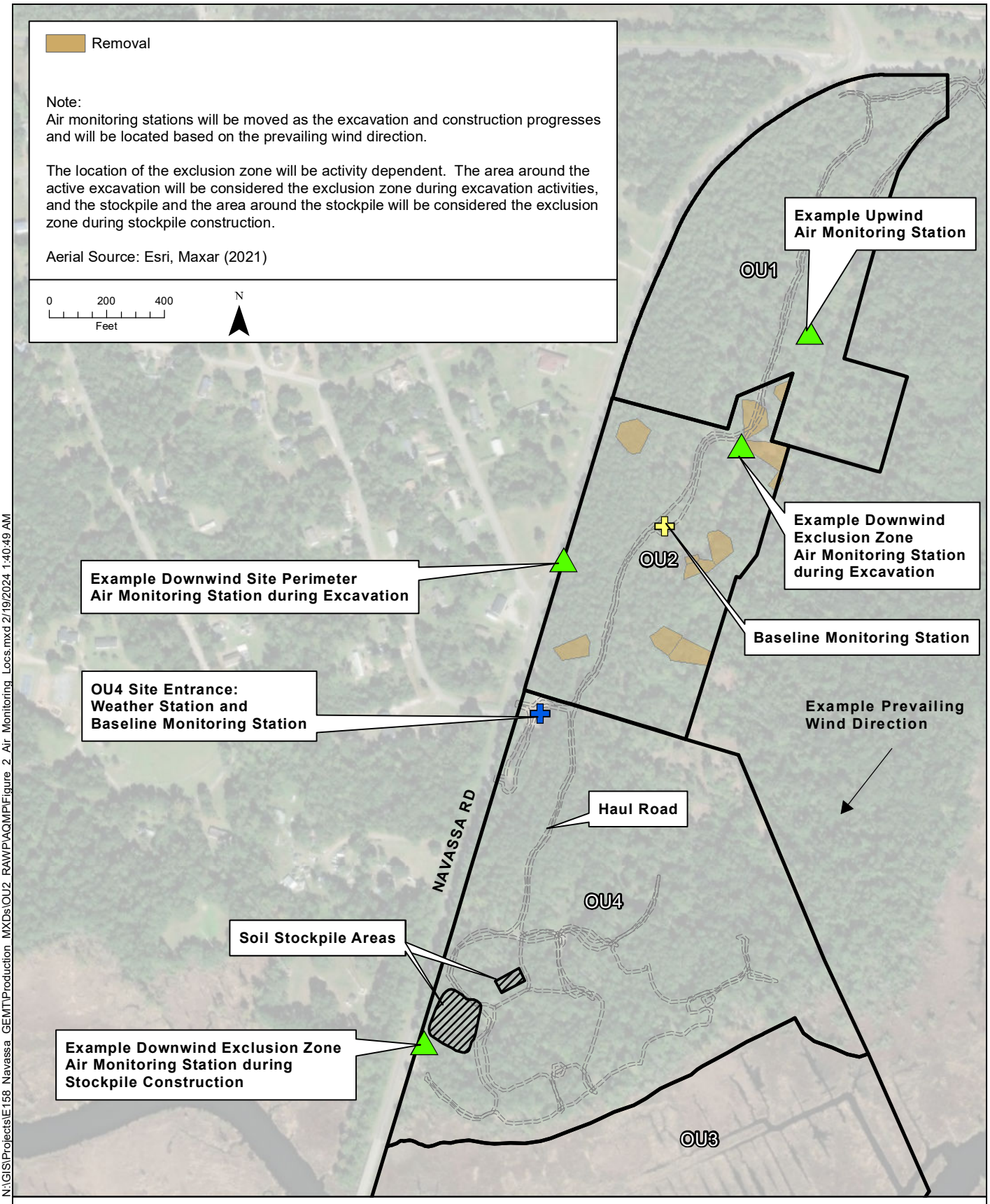


N:\GIS\Projects\E158 Navassa GEMT\Production MXDs\HH Tech Memo\Figure 1_1 Site Location.mxd 3/19/2021 5:32:53 PM

Prepared for: Greenfield Environmental Multistate Trust LLC
Trustee of the Multistate Environmental Response Trust

Prepared by: integral engineering pc

Figure 1.
Site Location
Kerr-McGee Chemical Corp. - Navassa Superfund Site
Navassa, North Carolina
February 2024

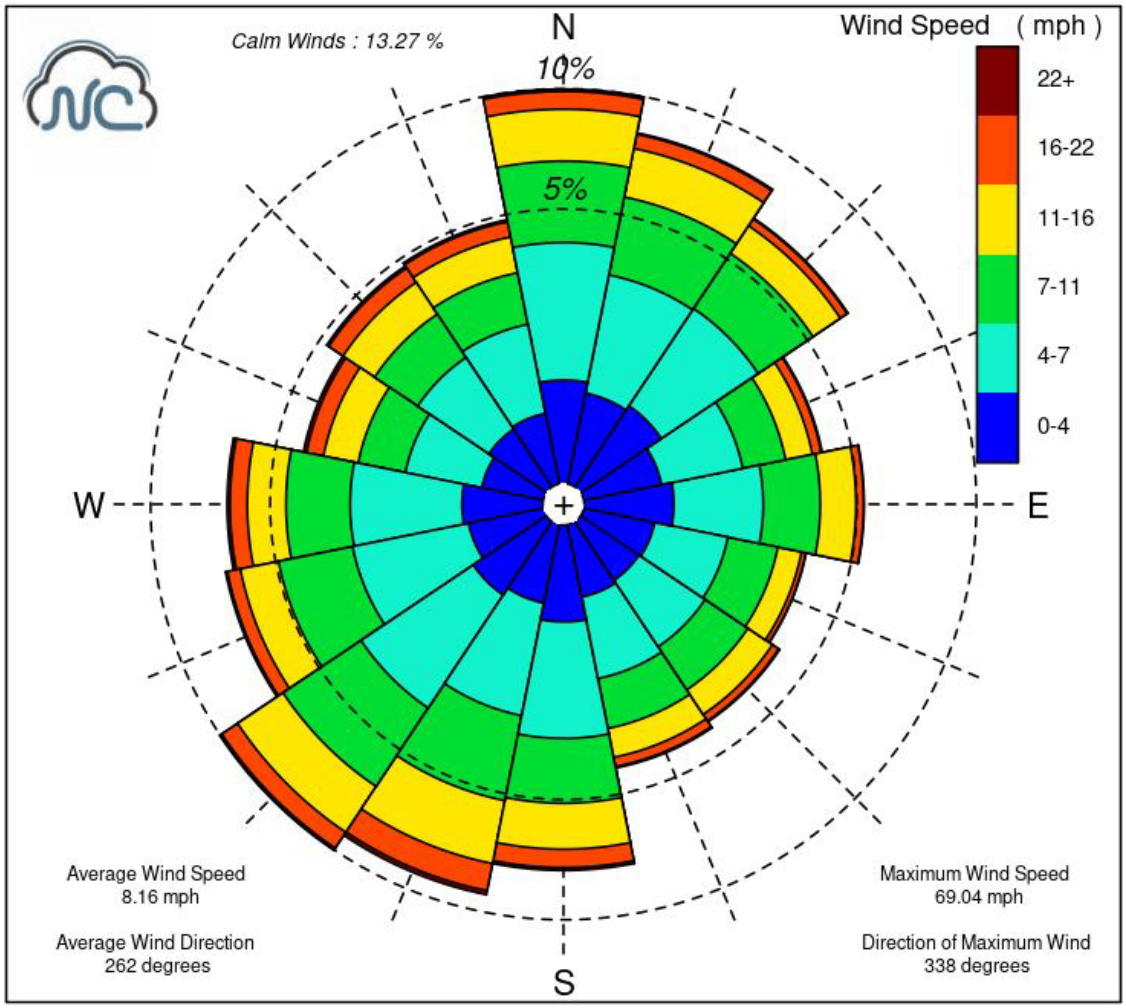


N:\GIS\Projects\E158 Navassa GEMT\Production MXDs\OU2 RAWPAQMP\Figure 2 Air Monitoring Locs.mxd 2/19/2024 1:40:49 AM

Prepared for: Greenfield Environmental Multistate Trust LLC
Trustee of the Multistate Environmental Response Trust

Prepared by: integral engineering pc.

Figure 2.
Air Monitoring Locations
Kerr-McGee Chemical Corp. - Navassa Superfund Site
Navassa, North Carolina
February 2024



Notes:
Wind rose generated from data collected at Wilmington International Airport, August 6, 1948 to August 6, 2021, as plotted using the North Carolina State University Climate Office wind rose generator tool, available at https://legacy.climate.ncsu.edu/windrose

Prepared for: Greenfield Environmental Multistate Trust LLC
Trustee of the Multistate Environmental Response Trust

Prepared by: integral engineering

Figure 3.
Wind Rose for Wilmington International Airport
Kerr-McGee Chemical Corp. - Navassa Superfund Site
Navassa, North Carolina
February 2024

Appendix A

Dust Monitoring Equipment Specifications

Knowledge Beyond Measure.

DustTrak™ II Aerosol Monitors

Models 8530, 8530EP and 8532

The DustTrak™ II monitors measure aerosol contaminants such as dust, smoke, fumes and mists. Desktop or Handheld units for any environment, any application.

DustTrak™ II Aerosol Monitors are battery-operated, data-logging, light-scattering laser photometers that give you real-time aerosol mass readings. They use a sheath air system that isolates the aerosol in the optics chamber to keep the optics clean for improved reliability and low maintenance. From desktop and desktop with external pump models to a handheld model, the DustTrak II offers a suitable solution for harsh industrial workplaces, construction and environmental sites and other outdoor applications, as well as clean office settings.

Features and Benefits

All Models

- Real-time mass concentration readings and data-logging allow for data analysis during and after sampling
- Measure aerosol concentrations corresponding to PM1, PM2.5, Respirable, and PM10 size fractions, using a variety of inlet conditioners
- Easy-to-use graphical user interface with color touch-screen for effortless operation

Handheld Model (8532)

- Long life internal pump for continuous sampling
- Single-point data collection for walk through surveys
- Lightweight design with ergonomic handle for portable applications

Desktop Models (8530 and 8530EP)

- Energy-efficient, long lasting external pump for continuous, unattended, 24/7, outdoor monitoring applications (Model 8530EP only)
- Long life internal pump for shorter work-shift or IAQ sampling applications (Model 8530)
- Gravimetric reference sampling capability for custom reference calibrations
- Automatic zeroing (with optional zero module) to minimize the effect of zero drift
- STEL alarm setpoint for tracking 15-minute average mass concentrations
- Environmental protected and tamper-proof secure (with an optional environmental enclosure)
- Inlet sample conditioning (with optional heated inlet sample conditioner) to reduce the effect of humidity on photometric mass measurements (for use with an environmental enclosure)

Desktop Models: Ideal for Long-Term Surveys and Remote Monitoring Applications

The DustTrak™ II is offered as a standard desktop (Model 8530), as well as a desktop with external pump (Model 8530EP.) Both models have manual and programmable data logging functions, making them ideal for unattended applications. The standard desktop model is most suitable for indoor, continuous monitoring, while the desktop with external pump is designed for 24/7 unattended, remote monitoring outdoors.

The DustTrak™ II desktop models come with USB (device and host), Ethernet, and analog and alarm outputs allowing remote access to data. User adjustable alarm setpoints for instantaneous or 15-minute short-term excursion limit (STEL) are also available on desktop models. The alarm output with user-defined setpoint alerts you when upset or changing conditions occur.

The DustTrak™ II desktop monitors have several unique features:

- Measure aerosols in high concentrations up to 400 mg/m³.
- External pump (Model 8530EP) with low power consumption for continuous, unattended monitoring in remote outdoor locations.
- Gravimetric sampling capability using a 37-mm filter cassette which can be inserted in-line with the aerosol stream allowing you to perform an integral gravimetric analysis for custom reference calibrations.
- Zeros automatically using the external zeroing module. This optional accessory is used when sampling over extended periods of time. By zeroing the monitor during sampling, the effect of zero drift is minimized.
- STEL alarm feature for tracking 15-minute average mass concentrations when alarm setpoint has been reached for applications like monitoring fugitive emissions at hazardous waste sites.
- Provide for environmental protection and tamper-proof security using an environmental enclosure. This optional accessory encloses the instrument within a waterproof, lockable, custom-designed case.
- Condition the sample air stream before entering the instrument optics using a heated inlet sample conditioner (designed for use with an environmental enclosure.) This optional accessory is used in humid environments. By conditioning the sample, the humidity and water vapor are minimized, reducing elevated measurements.

Handheld Models: Perfect for Walk-Through Surveys and Single-Point Data Collection Applications

The DustTrak II Handheld Model 8532 is lightweight and portable. It is perfect for industrial hygiene surveys, point source location monitoring, indoor air quality investigations, engineering control evaluations/validation, and for baseline trending and screening. Like the desktop models, it has manual and programmable data logging functions. In addition, the handheld model also has a single-point data logging capability. Single-point data collection is used for walk-through industrial hygiene surveys and indoor air quality investigations.

Applications	Desktop	Handheld
Aerosol research studies	▪	▪
Baseline trending and screening	▪	▪
Engineering control evaluations		▪
Engineering studies		▪
Epidemiology studies	▪	▪
Indoor air quality investigations	▪	▪
Industrial/occupational hygiene surveys	▪	▪
Point source monitoring		▪
Outdoor environmental monitoring	▪	
Process monitoring	▪	▪
Remote monitoring	▪	

Battery Performance		
Models 8530 and 8530EP (Typical) 7800 mAH Li-Ion Battery Pack (P/N 801680)	1 Battery	2 Batteries
Battery runtime (hours)	Up to 6	Up to 12
Charge time* (hours) in DustTrak	4	8
Charge time* (hours) in external battery charger (P/N 801685)	4	8

Model 8532 (Typical) 4200 mAH Li-Ion Battery Pack (P/N 801681)	Battery
Battery runtime (hours)	Up to 6
Charge time* (hours) in DustTrak	4
Charge time* (hours) in external battery charger (P/N 801686)	4

* Of a fully depleted battery

DustTrak™ II Aerosol Monitor Features

All Models

- Li-Ion rechargeable batteries
- Internal and external battery charging capabilities
- Outlet port for isokinetic sampling applications
- User serviceable sheath flow and pump filters
- Logged test pause and restart feature
- Logged test programming
  - Color touch screen—either manual mode or program mode
  - TrakPro™ Data Analysis Software via a PC
- User adjustable custom calibration settings
- Instantaneous alarm settings with visual and audible warnings
- Real-time graph display
- View statistical information during and after sampling
- On-screen instrument status indicators: FLOW, LASER and FILTER
- Filter service indicator for user preventative maintenance

Desktop Models (8530 and 8530EP)

- Long life external pump (8530EP)
- Internal pump (8530)
- Hot swappable batteries
- Gravimetric reference sample capability
- STEL alarm setpoint

Optional Accessories

- Auto zeroing module
- Protective environmental enclosure (8535 and 8537)
- Heated inlet sample conditioner (for use with an environmental enclosure)

Handheld Model (8532)

- Long life internal pump
- Single-point data collection for walk through surveys

Easy to Program and Operate

The graphical user interface with color touch-screen puts everything at your fingertips. The easy-to-read display shows real-time mass concentration and graphical data, as well as other statistical information along with instrument pump, laser and flow status, and much more. Perform quick walk-through surveys or program the instrument's advanced logging modes for long-term sampling investigations. Program start times, total sampling times, logging intervals, alarm setpoints and many other parameters. You can even set up the instrument for continuous unattended operation.

TrakPro™ Software Makes Monitoring Easier than Ever

TrakPro™ Data Analysis Software allows you to set up and program directly from a PC. It even features the ability for remote programming and data acquisition from your PC via wireless communication options or over an Ethernet network. As always, you can print graphs, raw data tables, and statistical and comprehensive reports for recordkeeping purposes.

Desktop Monitor with External Pump, Model 8530EP

Specifications

DustTrak™ II Aerosol Monitors

Models 8530, 8530EP and 8532

Sensor Type

90° light scattering

Particle Size Range

0.1 to 10 µm

Aerosol Concentration Range

8530 Desktop	0.001 to 400 mg/m³
8530EP Desktop with External Pump	0.001 to 400 mg/m³
8532 Handheld	0.001 to 150 mg/m³

Resolution

±0.1% of reading or 0.001 mg/m³, whichever is greater

Zero Stability

±0.002 mg/m³ per 24 hours at 10 sec time constant

Flow Rate

3.0 L/min set at factory, 1.40 to 3.0 L/min, user adjustable

Flow Accuracy

±5% of factory set point, internal flow controlled

Temperature Coefficient

+0.001 mg/m³ per °C

Operational Temp

32 to 120°F (0 to 50°C)

Storage Temp

-4 to 140°F (-20 to 60°C)

Operational Humidity

0 to 95% RH, non-condensing

Time Constant

User adjustable, 1 to 60 seconds

Data Logging

5 MB of on-board memory (>60,000 data points)

45 days at 1 minute logging interval

Log Interval

User adjustable, 1 second to 1 hour

Physical Size (H x W x D)

Handheld	4.9 x 4.8 x 12.5 in. (12.5 x 12.1 x 31.6 cm)
Desktop	5.3 x 8.5 x 8.8 in. (13.5 x 21.6 x 22.4 cm)
External Pump	4.0 x 7.0 x 3.5 in. (10.0 x 18.0 x 9.0 cm)

Weight

Handheld	2.9 lb (1.3 kg), 3.3 lb (1.5 kg) with battery
Desktop	3.5 lb (1.6 kg), 4.5 lb (2.0 kg) – 1 battery, 5.5 lb (2.5 kg) – 2 batteries
External Pump	3.0 lb (1.4 kg)

Communications

8530	USB (host and device) and Ethernet. Stored data accessible using flash memory drive
8530EP	USB (host and device) and Ethernet. Stored data accessible using flash memory drive plus, cable assembly for external pump
8532	USB (host and device). Stored data accessible using flash memory drive

Power–AC

Switching AC power adapter with universal line cord included,115–240 VAC

Analog Out

8530/8530EP	User selectable output, 0 to 5 V or 4 to 20 mA. User selectable scaling range

Alarm Out

8530/8530EP	Relay or audible buzzer Relay Non-latching MOSFET switch ▪ User selectable set point ▪ –5% deadband ▪ Connector 4-pin, Mini-DIN connectors
8532	Audible buzzer

Screen

8530	5.7 in. VGA color touchscreen
8532	3.5 in. VGA color touchscreen

Gravimetric Sampling

8530/8530EP	Removable 37 mm cartridge (user supplied)

CE Rating

Immunity	EN61236-1:2006
Emissions	EN61236-1:2006

Specifications are subject to change without notice.

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Appendix B

Calculation of Site-Specific Risk-Based Ambient Air Screening Levels

Table B-1. Site-Specific Risk-Based Ambient Air Screening Levels

Input Value	Name	BaP TEQ	BaP	TCDD TEQ	Naphthalene	Pentachlorophenol
CAS		50-32-8	50-32-8	1746-01-6	91-20-3	87-86-5
Scenario		Child (0-2 yrs)	Child (0-2 yrs)	Child (0-2 yrs)	Child (0-2 yrs)	Child (0-2 yrs)
Target Benchmarks						
Target Risk Level (unitless)	TR	1.0E-06	1.0E-06	1.0E-06	1.0E-06	1.0E-06
Target Hazard Quotient (unitless)	HQ	1.0E+00	1.0E+00	1.0E+00	1.0E+00	1.0E+00
Parameters						
Exposure duration (yrs)	ED	1	1	1	1	1
Exposure frequency (days/year)	EF	60	60	60	60	60
Exposure time (hours/day)	ET	10	10	10	10	10
Averaging time - noncarcinogenic (days)	AT_{nc}	365	365	365	365	365
Averaging time - carcinogenic (days)	AT_c	25,550	25,550	25,550	25,550	25,550
Conversion factor (day/hours)	CF1	0.042	0.042	0.042	0.042	0.042
Conversion factor (mg/µg)	CF2	0.001	0.001	0.001	0.001	0.001
Age dependent adjustment factor (0-2 yrs)	ADAF	10	--	--	--	--
Toxicity Values						
Mutagenic	M	Yes	No	No	No	No
Inhalation Unit Risk $(\mu g/m^3)^{-1}$	IUR	6E-04	NA	4E+01	3E-05	5E-06
Reference Concentration (mg/m^3)	RfC	NA	2E-06	4E-08	3E-03	NA
Cancer Screening Level $(\mu g/m^3)^{a,b}$	SL_{ca}	0.17	NA	0.000027	30	200
Noncancer Screening Level $(\mu g/m^3)^{c}$	SL_{nc}	NA	0.029	0.00058	44	NA
Site-Specific Risk-Based Ambient Air Screening Level $(\mu g/m^3)^{d}$	**SL**	**0.17**	**0.029**	**0.000027**	**30**	**200**

Notes:
-- = not available
BaP = benzo[*a*]pyrene
COC = constituent of concern
NA = not applicable
TEQ = toxic equivalency

Equations:
[a] The cancer screening level for BaP TEQ assumes a mutagenic mode of action and is derived as follows:
$SL_{ca} = (TR \times AT_c) / (IUR \times EF \times ED \times ET \times CF1 \times ADAF)$
[b] All other COC cancer screening levels for ambient air are derived as follows:
$SL_{ca} = (TR \times AT_c) / (IUR \times EF \times ED \times ET \times CF1)$
[c] The noncancer screening level for ambient air is derived as follows:
$SL_{nc} = (HQ \times AT_{nc}) / [(1/RfC) \times CF2 \times EF \times ED \times ET \times CF1]$
[d] The ambient air screening level = minimum (SL_{ca} and SL_{nc}).

Table B-2. Ambient Air Screening Evaluation Summary

Parameter	Unit	BaP TEQ	BaP	TCDD TEQ	Naphthalene	Pentachlorophenol
Maximum surface soil COC concentration at OU2[a]	µg/kg	107,000	65,700	0.275	2,460	1,130
North Carolina dust limit for PM10[b]	$\mu g/m^3$	150	150	150	150	150
Estimated COC concentration in ambient air based on North Carolina dust limit for PM10[c]	$\mu g/m^3$	0.016	0.010	0.000000041	0.00037	0.00017
Site-specific risk-based ambient air screening level for an offsite child (0-2 yrs)[d]	$\mu g/m^3$	0.17	0.029	0.000027	30	200
Does the COC concentration exceed the site-specific ambient air screening level for PM10?		No	No	No	No	No

Notes:
BaP = benzo[*a*]pyrene
COC = constituent of concern
PM10 = particles with an aerodynamic diameter 10 microns or smaller
TEQ = toxic equivalency

[a] See Table 3-1 of Integral (2021).
[b] 15 NCAC 02D.0409 PM10 particulate matter ambient air quality standard.
[c] COC concentration in ambient air ($\mu g/m^3$) = maximum COC concentration in surface soil (µg/kg) x dust limit concentration ($\mu g/m^3$) / conversion factor (µg/kg) (DTSC 2020)

References:
DTSC. 2020. Community air monitoring plan guidance. California Environmental Protection Agency, Department of Toxic Substances Control. January.
Integral. 2021. OU2 soil sampling results and human health risk assessment. Kerr-McGee constituent Corp. - Navassa Superfund Site, Navassa, North Carolina. September.

Appendix C

Air Sample Collection Procedures and Analytical Methods

Table C-1. Air Sampling Summary

Sample Event	Sample Type	Analytes	Method	Analyze/ Archive	No. Samples
Baseline Monitoring	Outdoor Air	Dust, PM10	Modified NIOSH 0500	Analyze	2
		PAHs	EPA TO-13A	Analyze	2
		VOCs	Monitoring Badge (OSHA 1005, NIOSH S292 and 1501)	Analyze	2
Excavation Monitoring	Outdoor Air	Dust, PM10	Modified NIOSH 0500	Analyze	2
		PAHs	EPA TO-13A	Analyze	2
VOC Exceedance [a]	Outdoor Air	VOCs	Monitoring Badge (OSHA 1005, NIOSH S292 and 1501)	Analyze	3

Notes:

PAH = polycyclic aromatic hydrocarbon

PID = photoionization detector

PM10 = particles with an aerodynamic diameter 10 microns or smaller

VOC = volatile organic compound

[a] VOC concentrations in the exclusion zone are sustained above 1 ppm over background for greater than 1 minute

Notes
Samples to be collected from area central to OU2 removal areas and at the Site perimeter downwind of OU2
Samples to be collected downwind of removal area and at the Site perimeter downwind of remedial activities
Samples to be collected within the exclusion zone, upwind of the exclusion zone, and at the Site perimeter downwind of remedial activities

Table C-2. Air Sampling Target Parameters, Analytical Methods, and Reporting Limits

Parameter	CAS Number	Method	Laboratory Reporting Limit and Level of Quantitation Values			
Total Dust					Reporting Limit (µg/m^3)	
			Reporting Limit (µg) [a]	Collected Air Volume (m^3):	1200	960
Total Dust, PM10	--	Modified NIOSH 0500	30		25	31
Volatile Organic Compounds (VOCs) via Monitoring Badges					Level of Quantitation (ppm)	
				Sample Duration:	24 hrs	8 hrs
Benzene	71-43-2	OSHA 1005, NIOSH 1501			0.02	0.05
Naphthalene	91-20-3	NIOSH S292 and 1501			0.02	0.05
Polycyclic Aromatic Hydrocarbons (PAHs) via EPA Method TO-13A					Reporting Limit (µg/m^3)		
			Reporting Limit (µg) [b]	Collected Air Volume (m^3):	300	125	100
Acenaphthene	83-32-9	EPA TO-13A	1		0.0033	0.0080	0.010
Acenaphthylene	208-96-8	EPA TO-13A	1		0.0033	0.0080	0.010
Anthracene	120-12-7	EPA TO-13A	1		0.0033	0.0080	0.010
Benzo[*a*]anthracene	56-55-3	EPA TO-13A	1		0.0033	0.0080	0.010
Benzo[*a*]pyrene	50-32-8	EPA TO-13A	1		0.0033	0.0080	0.010
Benzo[*b*]fluoranthene	205-99-2	EPA TO-13A	1		0.0033	0.0080	0.010
Benzo[*g,h,i*]perylene	191-24-2	EPA TO-13A	1		0.0033	0.0080	0.010
Benzo[*k*]fluoranthene	207-08-9	EPA TO-13A	1		0.0033	0.0080	0.010
Chrysene	218-01-9	EPA TO-13A	1		0.0033	0.0080	0.010
Dibenz[*a,h*]anthracene	53-70-3	EPA TO-13A	1		0.0033	0.0080	0.010
Fluoranthene	206-44-0	EPA TO-13A	1		0.0033	0.0080	0.010
Fluorene	86-73-7	EPA TO-13A	1		0.0033	0.0080	0.010
Indeno[1,2,3-*cd*]pyrene	193-39-5	EPA TO-13A	1		0.0033	0.0080	0.010
Naphthalene	91-20-3	EPA TO-13A	1		0.0033	0.0080	0.010
Phenanthrene	85-01-8	EPA TO-13A	1		0.0033	0.0080	0.010
Pyrene	129-00-0	EPA TO-13A	1		0.0033	0.0080	0.010

Notes:

PM10 = particles with an aerodynamic diameter 10 microns or smaller

[a] Reporting limit provided by SGS Galson, February 2024.

[b] Reporting limit provided by Eurofins, February 2024.

EPA/625/R-96/010b

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air

Second Edition

Compendium Method TO-13A

Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS)

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

January 1999

Method TO-13A
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This method is the result of the efforts of many individuals. Gratitude goes to each person involved in the preparation and review of this methodology.

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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.

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METHOD TO-13A

**Determination of Polycyclic Aromatic Hydrocarbons (PAHs)
in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS)**

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METHOD TO-13A

Determination of Polycyclic Aromatic Hydrocarbons (PAHs)
in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS)

1. Scope

1.1 Polycyclic aromatic hydrocarbons (PAHs) have received increased attention in recent years in air pollution studies because some of these compounds are highly carcinogenic or mutagenic. In particular, benzo[a]pyrene (B[a]P) has been identified as being highly carcinogenic. To understand the extent of human exposure to B[a]P and other PAHs, reliable sampling and analytical methods are necessary. This document describes a sampling and analysis procedure for common PAHs involving the use of a combination of quartz filter and sorbent cartridge with subsequent analysis by gas chromatography with mass spectrometry (GC/MS) detection. The analytical methods are modifications of EPA Test Method 610 and 625, *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, and Methods 8000, 8270, and 8310, *Test Methods for Evaluation of Solid Waste*.

1.2 Fluorescence methods were among the very first methods used for detection of B[a]P and other PAHs as carcinogenic constituents of coal tar (1-7). Fluorescence methods are capable of measuring subnanogram quantities of PAHs, but tend to be fairly non-selective. The normal spectra obtained are often intense and lack resolution. Efforts to overcome this difficulty led to the use of ultraviolet (UV) absorption spectroscopy (8) as the detection method coupled with pre-speciated techniques involving liquid chromatography (LC) and thin layer chromatography (TLC) to isolate specific PAHs, particularly B[a]P. As with fluorescence spectroscopy, the individual spectra for various PAHs are unique, although portions of spectra for different compounds may be the same. As with fluorescence techniques, the possibility of spectral overlap requires complete separation of sample components to ensure accurate measurement of component levels. Hence, the use of UV absorption coupled with pre-speciation involving LC and TLC and fluorescence spectroscopy declined and was replaced with the more sensitive high performance liquid chromatography (HPLC) with UV/fluorescence detection (9) or highly sensitive and specific gas chromatography/mass spectrometry (GC/MS) for detection (10-11).

1.3 The choice of GC/MS as the recommended procedure for analysis of B[a]P and other PAHs was influenced by its sensitivity and selectivity, along with its ability to analyze complex samples.

1.4 The analytical methodology has consequently been defined, but the sampling procedures can reduce the validity of the analytical results. Recent studies (12-17) have indicated that non-volatile PAHs (vapor pressure $<10^{-8}$ mm Hg) may be trapped on the filter, but post-collection volatilization problems may distribute the PAHs downstream of the filter to the back-up sorbent. A wide variety of sorbents such as Tenax®, XAD-2® and polyurethane foam (PUF) have been used to sample common PAHs. All sorbents have demonstrated high collection efficiency for B[a]P in particular. In general, XAD-2® resin has a higher collection efficiency (18-21) for volatile PAHs than PUF, as well as a higher retention efficiency. PUF cartridges, however, are easier to handle in the field and maintain better flow characteristics during sampling. Likewise, PUF has demonstrated (22) its capability in sampling organochlorine pesticides, polychlorinated biphenyls (22), and polychlorinated dibenzo-p-dioxins (23). PUF also has demonstrated a lower recovery efficiency and storage capability for naphthalene than XAD-2®. There have been no significant losses of PAHs up to 30 days of storage at room temperature (23°C) using XAD-2®. It also appears that XAD-2® resin has a higher collection efficiency for volatile PAHs than PUF, as well as a higher retention efficiency for both volatile and reactive PAHs.

Consequently, while the literature cites weaknesses and strengths of using either XAD-2® or PUF, this method includes the utilization of PUF as the primary sorbent.

1.5 This method includes the qualitative and quantitative analysis of the following PAHs (see Figure 1) specifically by utilizing PUF as the sorbent followed by GC/MS analysis:

Acenaphthene (low collection efficiency; see Section 6.1.3)	Coronene
Acenaphthylene (low collection efficiency; see Section 6.1.3)	Dibenz(a,h)anthracene
Anthracene	Fluoranthene
Benz(a)anthracene	Fluorene
Benzo(a)pyrene	Benzo(b)fluoranthene
Benzo(e)pyrene	Indeno(1,2,3-cd)pyrene
Benzo(g,h,i)perylene	Naphthalene (low collection efficiency; see Section 6.1.3)
Benzo(k)fluoranthene	Phenanthrene
Chrysene	Pyrene
	Perylene

The GC/MS method is applicable to the determination of PAHs compounds involving three member rings or higher. Naphthalene, acenaphthylene, and acenaphthene have only ~35 percent recovery when using PUF as the sorbent. Nitro-PAHs have ***not*** been fully evaluated using this procedure; therefore, they are not included in this method.

1.6 With optimization to reagent purity and analytical conditions, the detection limits for the GC/MS method range from 1 ng to 10 pg based on field experience.

2. Summary of Method

2.1 Filters and sorbent cartridges (containing PUF or XAD-2®) are cleaned in solvents and vacuum dried. The filters and sorbent cartridges are stored in screw-capped jars wrapped in aluminum foil (or otherwise protected from light) before careful installation on the sampler.

2.2 Approximately 300 m^3 of air is drawn through the filter and sorbent cartridge using a high-volume flow rate air sampler or equivalent.

2.3 The amount of air sampled through the filter and sorbent cartridge is recorded, and the filter and cartridge are placed in an appropriately labeled container and shipped along with blank filter and sorbent cartridges to the analytical laboratory for analysis.

2.4 The filters and sorbent cartridge are extracted by Soxhlet extraction with appropriate solvent. The extract is concentrated by Kuderna-Danish (K-D) evaporator, followed by silica gel cleanup using column chromatography to remove potential interferences prior to analysis by GC/MS.

2.5 The eluent is further concentrated by K-D evaporation, then analyzed by GC/MS. The analytical system is verified to be operating properly and calibrated with five concentration calibration solutions.

2.6 A preliminary analysis of the sample extract is performed to check the system performance and to ensure that the samples are within the calibration range of the instrument. If the preliminary analysis indicates non-performance, then recalibrate the instrument, adjust the amount of the sample injected, adjust the calibration solution concentration, and adjust the data processing system to reflect observed retention times, etc.

2.7 The samples and the blanks are analyzed and used (along with the amount of air sampled) to calculate the concentration of PAHs in the air sample**.**

3. Significance

3.1 As discussed in Section 1, several documents have been published that describe sampling and analytical approaches for common PAHs. The attractive features of these methods have been combined in this procedure. Although this method has been validated in the laboratory, one must use caution when employing it for specific applications.

3.2 Because of the relatively low levels of common PAHs in the environment, the methodology suggest the use of high volume (0.22 m^3/min) sampling technique to acquire sufficient sample for analysis. However, the volatility of certain PAHs prevents efficient collection on filter media alone. Consequently, this method utilizes both a filter and a backup sorbent cartridge, which provides for efficient collection of most PAHs involving three member rings or higher.

4. Applicable Documents

4.1 ASTM Standards

- **Method D1356** *Definitions of Terms Relating to Atmospheric Sampling and Analysis*.
- **Method 4861-94** *Standard Practice for Sampling and Analysis of Pesticides and Polychlorinated Biphenyl in Air*
- **Method E260** *Recommended Practice for General Gas Chromatography Procedures*.
- **Method E355** *Practice for Gas Chromatography Terms and Relationships*.
- **Method E682** *Practice for Liquid Chromatography Terms and Relationships*.

4.2 EPA Documents

- *Technical Assistance Document for Sampling and Analysis of Toxic Organic Compounds in Ambient Air*, U. S. Environmental Protection Agency, EPA-600/4-83-027, June 1983.
- *Quality Assurance Handbook for Air Pollution Measurement Systems*, U. S. Environmental Protection Agency, EPA-600/R-94-038b, May 1994.
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air: Method TO-13, Second Supplement*, U. S. Environmental Protection Agency, EPA-600/-4-89-018, March 1989.

4.3 Other Documents

- Existing Procedures (24-32).
- Ambient Air Studies (33-50).
- General Metal Works, Inc., "Operating Procedures for Model PS-1 Sampler," Village of Cleves, OH 45002 (800-543-7412).
- Illinois Environmental Protection Agency, Division of Air Quality, "Chicago Air Quality: PCB Air Monitoring Plan (Phase 2)," Chicago, IL, IEAP/APC/86/011, April 1986.
- Thermo Environmental, Inc. (formerly Wedding and Associates), "Operating Procedures for the Thermo Environmental Semi-Volatile Sampler," 8 West Forge Parkway, Franklin, MA 02038 (508-520-0430).
- American Chemical Society (ACS), "Sampling for Organic Chemicals in Air," *ACS Professional Book*, ACS, Washington, D.C., 1996.
- International Organization for Standardization (ISO), "Determination of Gas and Particle-Phase Polynuclear Aromatic Hydrocarbons in Ambient Air - Collected on Sorbent-Backed Filters with Gas Chromatographic/Mass Spectrometric Analysis," ISO/TC 146/SC 3/WG 17N, Case Postale 56, CH-1211, Genève 20, Switzerland.

5. Definitions

[Note: Definitions used in this document and in any user-prepared standard operating procedures (SOPs) should be consistent with ASTM Methods D1356, E260, and E255. All abbreviations and symbols are defined within this document at point of use.]

5.1 Retention time (RT)-time to elute a specific chemical from a chromatographic column. For a specific carrier gas flow rate, RT is measured from the time the chemical is injected into the gas stream until it appears at the detector.

5.2 Sampling efficiency (SE)-ability of the sampler to trap and retain PAHs. The %SE is the percentage of the analyte of interest collected and retained by the sampling medium when it is introduced into the air sampler and the sampler is operated under normal conditions for a period of time equal to or greater than that required for the intended use.

5.3 Dynamic retention efficiency-ability of the sampling medium to retain a given PAH that has been added to the sorbent trap in a spiking solution when air is drawn through the sampler under normal conditions for a period of time equal to or greater than that required for the intended use.

5.4 Polycyclic aromatic hydrocarbons (PAHs)-two or more fused aromatic rings.

5.5 Method detection limit (MDL)-the minimum concentration of a substance that can be measured and reported with confidence and that the value is above zero.

5.6 Kuderna-Danish apparatus-the Kuderna-Danish (K-D) apparatus is a system for concentrating materials dissolved in volatile solvents.

5.7 MS-SCAN-the GC is coupled to a mass spectrometer where the instrument is programmed to acquire all ion data.

5.8 Sublimation-the direct passage of a substance from the solid state to the gaseous state and back into the solid form without at any time appearing in the liquid state. Also applied to the conversion of solid to vapor without the later return to solid state, and to a conversion directly from the vapor phase to the solid state.

5.9 Surrogate standard-a chemically inert compound (not expected to occur in the environmental sample) that is added to each sample, blank, and matrix-spiked sample before extraction and analysis. The recovery of the surrogate standard is used to monitor unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within acceptable limits.

5.10 CAL-calibration standards are defined as five levels of calibration: CAL 1, CAL 2, CAL 3, CAL 4, and CAL 5. CAL 1 is the lowest concentration and CAL 5 is the highest concentration. CAL 3, which is the mid-level standard, is designated as the solution to be used for continuing calibrations.

5.11 Continuing calibration check-a solution of method analytes used to evaluate the mass spectrometer response over a period of time. A continuing calibration check (CCC) is performed once each 12-hour period. The CCC solution (CAL 3) is the standard of the calibration curve.

5.12 GC Response (A_x)-the peak area or height of analyte, x.

5.13 Internal standard (IS)-a compound added to a sample extract in known amounts and used to calibrate concentration measurements of other compounds that are sample components. The internal standard must be a compound that is not a sample component.

6. Limitations and Interferences

6.1 Limitations

6.1.1 PAHs span a broad spectrum of vapor pressures (e.g., from 1.1 x 10^{-2} kPa for naphthalene to 2 x 10^{-13} kPa for coronene at 25°C). PAHs that are frequently found in ambient air are listed in Table 1. Those with vapor pressures above approximately 10^{-8} kPa will be present in the ambient air substantially distributed between the gas and particulate phases. This method will permit the collection of both phases.

6.1.2 Particulate-phase PAHs will tend to be lost from the particle filter during sampling due to volatilization. Therefore, separate analysis of the filter will not reflect the concentrations of the PAHs originally associated with particles, nor will analysis of the sorbent provide an accurate measure of the gas phase. Consequently, this method calls for *extraction of the filter and sorbent together* to permit accurate measurement of total PAH air concentrations.

6.1.3 Naphthalene, acenaphthylene, and acenaphthene possess relatively high vapor pressures and may not be efficiently trapped by this method when using PUF as the sorbent. The sampling efficiency for naphthalene has been determined to be about 35 percent for PUF. The user is encouraged to use XAD-2® as the sorbent if these analytes are part of the target compound list (TCL**).**

6.2 Interferences

6.2.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that result in discrete artifacts and/or elevated baselines in the detector profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

6.2.2 Glassware must be scrupulously cleaned (51). All glassware should be cleaned as soon as possible after use by rinsing with the last solvent used in it and then high-purity acetone and hexane. These rinses should be followed by detergent washing with hot water and rinsing with copious amounts of tap water and several portions of reagent water. The glassware should then be drained dry and heated in a muffle furnace at 400°C for four hours. Volumetric glassware must not be heated in a muffle furnace; rather it should be solvent rinsed with acetone and spectrographic grade hexane. After drying and rinsing, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Glassware should be stored inverted or capped with aluminum foil.

[Note: The glassware may be further cleaned by placing in a muffle furnace at 450°C for 8 hours to remove trace organics.]

6.2.3 The use of high purity water, reagents, and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

6.2.4 Matrix interferences may be caused by contaminants that are coextracted from the sample. Additional clean-up by column chromatography may be required (see Section 12.3).

6.2.5 During sample transport and analysis, heat, ozone, NO_2, and ultraviolet (UV) light may cause sample degradation. Incandescent or UV-shielded fluorescent lighting in the laboratory should be used during analysis.

6.2.6 The extent of interferences that may be encountered using GC/MS techniques has not been fully assessed. Although GC conditions described allow for unique resolution of the specific PAH compounds covered by this method, other PAH compounds may interfere. The use of column chromatography for sample clean-up prior to GC analysis will eliminate most of these interferences. The analytical system must, however, be routinely demonstrated to be free of internal contaminants such as contaminated solvents, glassware, or other reagents which may lead to method interferences. A laboratory reagent blank should be analyzed for each reagent used to determine if reagents are contaminant-free.

6.2.7 Concern about sample degradation during sample transport and analysis was mentioned above. Heat, ozone, NO_2, and ultraviolet (UV) light also may cause sample degradation. These problems should be addressed as part of the user-prepared standard operating procedure (SOP) manual. Where possible, incandescent or UV-shielded fluorescent lighting should be used during analysis. During transport, field samples should be shipped back to the laboratory chilled (~4°C) using blue ice/dry ice.

7. Safety

7.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and are included in the reference list (52-54).

7.2 B[a]P has been tentatively classified as a known or suspected, human or mammalian carcinogen. Many of the other PAHs have been classified as carcinogens. Care must be exercised when working with these substances. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of whomever uses this method to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. The user should be thoroughly familiar with the chemical and physical properties of targeted substances (see Table 1 and Figure 1).

7.3 All PAHs should be treated as carcinogens. Neat compounds should be weighed in a glove box. Spent samples and unused standards are toxic waste and should be disposed according to regulations. Counter tops and equipment should be regularly checked with "black light" for fluorescence as an indicator of contamination.

7.4 The sampling configuration (filter and backup sorbent) and collection efficiency for target PAHs has been demonstrated to be greater than 95 percent (except for naphthalene, acenaphthylene and acenaphthene). Therefore, no field recovery evaluation will be required as part of this procedure.

[Note: Naphthalene, acenaphthylene and acenaphthene have demonstrated significant breakthrough using PUF cartridges, especially at summer ambient temperatures. If naphthalene, acenaphthylene and acenaphthene are target PAHs, the user may want to consider replacing the PUF with XAD-2® in order to minimize breakthrough during sampling.]

8. Apparatus

[Note: This method was developed using the PS-1 semi-volatile sampler provided by General Metal Works, Village of Cleves, OH as a guideline. EPA has experience in the use of this equipment during various field-monitoring programs over the last several years. Other manufacturers' equipment should work as well; however, modifications to these procedures may be necessary if another commercially available sampler is selected.]

8.1 Sampling

8.1.1 High-volume sampler (see Figure 2). Capable of pulling ambient air through the filter/sorbent cartridge at a flow rate of approximately 8 standard cubic feet per minute (scfm) (0.225 std m^3/min) to obtain a total sample volume of greater than 300 m^3 over a 24-hour period. Major manufacturers are:

- Tisch Environmental, Village of Cleves, OH
- Andersen Instruments Inc., 500 Technology Ct., Smyrna, GA
- Thermo Environmental Instruments, Inc., 8 West Forge Parkway, Franklin, MA

Recent EPA studies have concluded that sample volumes *less than* 300 m^3 still collect enough PAHs on the filter/PUF for quantitation. The user is encouraged to investigate appropriate sample volume needed to meet project specific data quality objectives.

8.1.2 Sampling module (see Figure 3). Metal filter holder (Part 2) capable of holding a 102-mm circular particle filter supported by a 16-mesh stainless-steel screen and attaching to a metal cylinder (Part 1) capable of holding a 65-mm O.D. (60-mm I.D.) x 125-mm borosilicate glass sorbent cartridge containing PUF or XAD-2®. The filter holder is equipped with inert sealing gaskets (e.g., polytetrafluorethylene) placed on either side of the

filter. Likewise, inert, pliable gaskets (e.g., silicone rubber) are used to provide an air-tight seal at each end of the glass sorbent cartridge. The glass sorbent cartridge is indented 20 mm from the lower end to provide a support for a 16-mesh stainless-steel screen that holds the sorbent. The glass sorbent cartridge fits into Part 1, which is screwed onto Part 2 until the sorbent cartridge is sealed between the silicone gaskets. Major manufacturers are:

- Tisch Environmental, Village of Cleves, OH
- Andersen Instruments Inc., 500 Technology Ct., Smyrna, GA
- Thermo Environmental Instruments, Inc., 8 West Forge Parkway, Franklin, MA

8.1.3 High-volume sampler calibrator. Capable of providing multipoint resistance for the high-volume sampler. Major manufacturers are**:**

- Tisch Environmental, Village of Cleves, OH
- Andersen Instruments Inc., 500 Technology Ct., Smyrna, GA
- Thermo Environmental Instruments, Inc., 8 West Forge Parkway, Franklin, MA

8.1.4 Ice chest. To hold samples at 4°C or below during shipment to the laboratory after collection.

8.1.5 Data sheets. Used for each sample to record the location and sample time, duration of sample, starting time, and volume of air sampled.

8.2 Sample Clean-Up and Concentration (see Figure 4).

8.2.1 Soxhlet apparatus extractor (see Figure 4a). Capable of extracting filter and sorbent cartridges (5.75-cm x 12.5-cm length), 1,000 mL flask, and condenser, best source.

8.2.2 Pyrex glass tube furnace system. For activating silica gel at 180°C under purified nitrogen gas purge for an hour, with capability of raising temperature gradually, best source.

8.2.3 Glass vial. 40 mL, best source.

8.2.4 Erlenmeyer flask. 50 mL, best source.

[Note: Reuse of glassware should be minimized to avoid the risk of cross contamination. All glassware that is used must be scrupulously cleaned as soon as possible after use. Rinse glassware with the last solvent used in it and then with high-purity acetone and hexane. Wash with hot water containing detergent. Rinse with copious amounts of tap water and several portions of distilled water. Drain, dry, and heat in a muffle furnace at 400°C for 4 hours. Volumetric glassware must not be heated in a muffle furnace; rather, it should be rinsed with high-purity acetone and hexane. After the glassware is dry and cool, rinse it with hexane, and store it inverted or capped with solvent-rinsed aluminum foil in a clean environment.]

8.2.5 White cotton gloves. For handling cartridges and filters, best source.

8.2.6 Minivials. 2 mL, borosilicate glass, with conical reservoir and screw caps lined with Teflon®-faced silicone disks, and a vial holder, best source.

8.2.7 Teflon®-coated stainless steel spatulas and spoons. Best source.

8.2.8 Kuderna-Danish (K-D) apparatus (see Figure 4b). 500 mL evaporation flask (Kontes K-570001-500 or equivalent), 10 mL graduated concentrator tubes (Kontes K570050-1025 or equivalent) with ground-glass stoppers, 1 mL calibrated K-D concentration tubes, and 3-ball macro Snyder Column (Kontes K-570010500, K-50300-0121, and K-569001-219, or equivalent), best source.

8.2.9 Adsorption column for column chromatography (see Figure 4c). 1-cm x 10-cm with stands.

8.2.10 Glove box. For working with extremely toxic standards and reagents with explosion-proof hood for venting fumes from solvents, reagents, etc.

8.2.11 Vacuum oven. Vacuum drying oven system capable of maintaining a vacuum at 240 torr (flushed with nitrogen) overnight.

8.2.12 Concentrator tubes and a nitrogen evaporation apparatus with variable flow rate. Best source.

8.2.13 Laboratory refrigerator. Best source.

8.2.14 Boiling chips. Solvent extracted, 10/40 mesh silicon carbide or equivalent, best source**.**

8.2.15 Water bath. Heated, with concentric ring cover, capable of ±5°C temperature control, best source.

8.2.16 Nitrogen evaporation apparatus. Best source.

8.2.17 Glass wool. High grade, best source**.**

8.3 Sample Analysis

8.3.1 Gas Chromatography with Mass Spectrometry Detection Coupled with Data Processing System (GC/MS/DS). The gas chromatograph must be equipped for temperature programming, and all required accessories must be available, including syringes, gases, and a capillary column. The gas chromatograph injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. On-column injection techniques can be used, but they may severely reduce column lifetime for nonchemically bonded columns. In this protocol, a 2 µL injection volume is used consistently to maximize auto sampler reproducibility. With some gas chromatograph injection ports, however, 1 µL injections may produce some improvement in precision and chromatographic separation. A 1 µL injection volume may be used if adequate sensitivity and precision can be achieved.

[Note: If 1 µL is used as the injection volume, the injection volumes for all extracts, blanks, calibration solutions and performance check samples must be 1 µL.]

All GC carrier gas lines must be constructed from stainless steel or copper tubing. Poly-tetrafluoroethylene (PTFE) thread sealants or flow controllers should only be used.

8.3.2 Gas chromatograph-mass spectrometer interface. The GC is usually coupled directly to the MS source. The interface may include a diverter valve for shunting the column effluent and isolating the mass spectrometer source. All components of the interface should be glass or glass-lined stainless steel. Glass can be deactivated by silanizing with dichorodimethylsilane. The interface components should be compatible with 320°C temperatures. Cold spots and/or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the MS source. Graphite ferrules should be avoided in the gas chromatograph injection area since they may adsorb PAHs. Vespel® or equivalent ferrules are recommended.

8.3.3 Mass spectrometer. The MS should be operated in the full range data acquisition (SCAN) mode with a total cycle time (including voltage reset time) of one second or less (see Section 13.3.2). Operation of the MS in the SCAN mode allows monitoring of all ions, thus assisting with the identification of other PAHs beyond Compendium Method TO-13A target analyte list. In addition, operating in the SCAN mode assists the analyst with identification of possible interferences from non-target analytes due to accessibility of the complete mass spectrum in the investigative process. The MS must be capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact (EI) ionization mode. The mass spectrometer must be capable of producing a mass spectrum for a 50 ng injection of decafluorotriphyenyl phosphine (DFTPP) which meets all of the response criteria (see Section 13.3.3). To ensure sufficient precision of mass spectral data, the MS scan rate must allow acquisition of at least five scans while a sample compound elutes from the GC. The

GC/MS system must be in a room with atmosphere demonstrated to be free of all potential contaminants which will interfere with the analysis. The instrument must be vented outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.

8.3.4 Data system. A dedicated computer data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and multi-ion detector (MID) traces (displays of intensities of each m/z being monitored as a function of time) must be acquired during the analyses. Quantifications may be reported based upon computer generated peak areas or upon measured peak heights (chart recording). The detector zero setting must allow peak-to-peak measurement of the noise on the baseline. The computer should have software that allows searching the GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. This type of plot is defined as Selected Ion Current Profile (SICP). The software used must allow integrating the abundance in any SICP between specified time or scan number limits. The data system should be capable of flagging all data files that have been edited manually by laboratory personnel.

8.3.5 Gas chromatograph column. A fused silica DB-5 column (30 m x 0.32 mm I.D.) crosslinked 5 percent phenyl methylsilicone, 1.0 µm film thickness is utilized to separate individual PAHs. Other columns may be used for determination of PAHs. Minimum acceptance criteria must be determined as per Section 13.3. At the beginning of each 12-hour period (after mass resolution has been demonstrated) during which sample extracts or concentration calibration solutions will be analyzed, column operating conditions must be attained for the required separation on the column to be used for samples.

8.3.6 Balance. Mettler balance or equivalent.

8.3.7 All required syringes, gases, and other pertinent supplies. To operate the GC/MS system.

8.3.8 Pipettes, micropipettes, syringes, burets, etc. Used to make calibration and spiking solutions, dilute samples if necessary, etc., including syringes for accurately measuring volumes such as 25 µL and 100 µL.

9. Equipment and Materials

9.1 Materials for Sample Collection (see Figure 3)

9.1.1 Quartz fiber filter. 102 millimeter binderless quartz microfiber filter, Whatman Inc., 6 Just Road, Fairfield, NJ 07004, Filter Type QMA-4.

9.1.2 Polyurethane foam (PUF) plugs (see Figure 5a). 3-inch thick sheet stock polyurethane type (density .022 g/cm^3). The PUF should be of the polyether type used for furniture upholstery, pillows, and mattresses. The PUF cylinders (plugs) should be slightly larger in diameter than the internal diameter of the cartridge. Sources of equipment are Tisch Environmental, Village of Cleves, OH; University Research Glassware, 116 S. Merritt Mill Road, Chapel Hill, NC; Thermo Environmental Instruments, Inc., 8 West Forge Parkway, Franklin, MA; Supelco, Supelco Park, Bellefonte, PA; and SKC Inc., 334 Valley View Road, Eighty Four, PA.

9.1.3 XAD-2® resin (optional). Supelco, Supelco Park, Bellefonte, PA.

9.1.4 Teflon® end caps (see Figure 5a). For sample cartridge; sources of equipment are Tisch Environmental, Village of Cleves, OH; and University Research Glassware, 116 S. Merritt Mill Road, Chapel Hill, NC.

9.1.5 Sample cartridge aluminum shipping containers (see Figure 5b). For sample cartridge shipping; sources of equipment are Tisch Environmental, Village of Cleves, OH; and University Research Glassware, 116 S. Merritt Mill Road, Chapel Hill, NC.

9.1.6 Glass sample cartridge (see Figure 5a). For sample collection; sources of equipment are Tisch Environmental, Village of Cleves, OH; Thermo Environmental Instruments, Inc., 8 West Forge Parkway, Franklin, MA; and University Research Glassware, 116 S. Merritt Mill Road, Chapel Hill, NC.

9.1.7 Aluminum foil. Best source.

9.1.8 Hexane, reagent grade. Best source.

9.2 Sample Clean-up and Concentration

9.2.1 Methylene chloride (extraction solvent for XAD-2®; optional). Chromatographic grade, glass-distilled, best source.

9.2.2 Sodium sulfate-anhydrous (ACS). Granular (purified by washing with methylene chloride followed by heating at 400°C for 4 hours in a shallow tray).

9.2.3 Boiling chips. Solvent extracted or heated in a muffle furnace at 450°C for 2 hours, approximately 10/40 mesh (silicon carbide or equivalent).

9.2.4 Nitrogen. High purity grade, best source.

9.2.5 Hexane. Chromatographic grade, glass-distilled, best source (extraction solvent for PUF).

9.2.6 Glass wool. Silanized, extracted with methylene chloride and hexane, and dried.

9.2.7 Diethyl ether. High purity, glass distilled (extraction solvent for PUF).

9.2.8 Pentane. High purity, glass distilled.

9.2.9 Silica gel. High purity, type 60, 70-230 mesh.

9.3 GC/MS Sample Analysis

9.3.1 Gas cylinder of helium. Ultra high purity, best source.

9.3.2 Chromatographic-grade stainless steel tubing and stainless steel fitting. For interconnections, Alltech Applied Science, 2051 Waukegan Road, Deerfield, IL 60015, 312-948-8600, or equivalent.

[Note: All such materials in contact with the sample, analyte, or support gases prior to analysis should be stainless steel or other inert metal. Do not use plastic or Teflon® tubing or fittings.]

9.3.3 Native and isotopically labeled PAH isomers for calibration and spiking standards. Cambridge Isotopes, 20 Commerce Way, Woburn, MA 01801 (617-547-1818). Suggested isotopically labeled PAH isomers are: D_{10}-fluoranthene, D_{12}-benzo(a)pyrene, D_{10}-fluorene, D_{10}-pyrene, D_{12}-perylene, D_{10}-acenaphthene, D_{12}-chrysene, D_8-naphthalene and D_{10}-phenanthrene.

9.3.4 Decafluorotriphenylphosphine (DFTPP). Used for tuning GC/MS, best source.

9.3.5 Native stock pure standard PAH analytes. For developing calibration curve for GC/MS analysis, best source.

10. Preparation of PUF Sampling Cartridge

[Note: This method was developed using the PS-1 sample cartridge provider by General Metal Works, Village of Cleves, OH as a guideline. EPA has experience in use of this equipment during various field monitoring program over the last several years. Other manufacturers' equipment should work as well; however, modifications to these procedures may be necessary if another commercially available sampler is selected.]

10.1 Summary of Method

10.1.1 This part of the procedure discusses pertinent information regarding the preparation and cleaning of the filter, sorbent, and filter/sorbent cartridge assembly. The separate batches of filters and sorbents are extracted with the appropriate solvent.

10.1.2 At least one PUF cartridge assembly and one filter from each batch, or 10 percent of the batch, whichever is greater, should be tested and certified before the batch is considered for field use.

10.1.3 Prior to sampling, the cartridges are spiked with field surrogate compounds.

10.2 Preparation of Sampling Cartridge

10.2.1 Bake the Whatman QMA-4 quartz filters at 400°C for 5 hours before use.

10.2.2 Set aside the filters in a clean container for shipment to the field or prior to combining with the PUF glass cartridge assembly for certification prior to field deployment.

10.2.3 The PUF plugs are 6.0-cm diameter cylindrical plugs cut from 3-inch sheet stock and should fit, with slight compression, in the glass cartridge, supported by the wire screen (see Figure 5a). During cutting, rotate the die at high speed (e.g., in a drill press) and continuously lubricate with deionized or distilled water. Pre-cleaned PUF plugs can be obtained from commercial sources (see Section 9.1.2)**.**

10.2.4 For initial cleanup, place the PUF plugs in a Soxhlet apparatus and extract with acetone for 16 hours at approximately 4 cycles per hour. When cartridges are reused, use diethyl ether/hexane (5 to 10 percent volume/volume [v/v]) as the cleanup solvent.

[Note: A modified PUF cleanup procedure can be used to remove unknown interference components of the PUF blank. This method consists of rinsing 50 times with toluene, acetone, and diethyl ether/hexane (5 to 10 percent v/v), followed by Soxhlet extraction. The extracted PUF is placed in a vacuum oven connected to a water aspirator and dried at room temperature for approximately 2 to 4 hours (until no solvent odor is detected). The extract from the Soxhlet extraction procedure from each batch may be analyzed to determine initial cleanliness prior to certification.]

10.2.5 If using XAD-2® in the cartridge, initial cleanup of the resin is performed by placing approximately 50-60 grams in a Soxhlet apparatus and extracting with methylene chloride for 16 hours at approximately 4 cycles per hour. At the end of the initial Soxhlet extraction, the spent methylene chloride is discarded and replaced with a fresh reagent. The XAD-2® resin is once again extracted for 16 hours at approximately 4 cycles per hour. The XAD-2® resin is removed from the Soxhlet apparatus, placed in a vacuum oven connected to an ultra-pure nitrogen gas stream, and dried at room temperature for approximately 2-4 hours (until no solvent odor is detected).

10.2.6 Fit a nickel or stainless steel screen (mesh size 200/200) to the bottom of a hexane-rinsed glass sampling cartridge to retain the PUF or XAD-2® sorbents, as illustrated in Figure 5a. If using XAD-2® alone, then place a small diameter (~1/4") PUF plug on top of the nickel or stainless steel screen to retain the XAD-2® in the glass cartridge. Place the Soxhlet-extracted, vacuum-dried PUF (2.5-cm thick by 6.5-cm diameter) on top of the screen in the glass sampling cartridge using polyester gloves. Place ~200 g of the clean XAD-2® inside the glass sampling cartridge on top of the small diameter PUF plug.

10.2.7 Wrap the sampling cartridge with hexane-rinsed aluminum foil, cap with the Teflon® end caps (optional), place in a cleaned labeled aluminum shipping container, and seal with Teflon® tape. Analyze at least 1 cartridge from each batch of cartridges prepared using the procedure described in Section 10.3, before the batch is considered acceptable for field use.

The acceptance level of the cartridge is for each target PAH analyte to be less than or equal to the detection limit requirements to meet the project data quality objectives. It is generally not possible to eliminate the presence of naphthalene, but the amount detected on the cleaned PUF cartridge should be less than five times the concentration of the lowest calibration standard (~500 ng). This amount is insignificant compared to the amount collected from a typical air sample.

In general, the following guidelines are provided in determining whether a cartridge is clean for field use:

- Naphthalene <500 ng/cartridge
- Other PAHs <200 ng total/cartridge

10.3 Procedure for Certification of PUF Cartridge Assembly

[Note: The following procedure outlines the certification of a filter and PUF cartridge assembly. If using XAD-2® as the sorbent, the procedure remains the same, except the solvent is methylene chloride rather than 10 percent diethyl ether/hexane.]

10.3.1 Extract one filter and PUF sorbent cartridge by Soxhlet extraction and concentrate using a K-D evaporator for each lot of filters and cartridges sent to the field.

10.3.2 Assemble the Soxhlet apparatus. Charge the Soxhlet apparatus (see Figure 4a) with 700 mL of the extraction solvent (10 percent v/v diethyl ether/hexane) and reflux for 2 hours. Let the apparatus cool, disassemble it, and discard the used extraction solvent. Transfer the filter and PUF glass cartridge to the Soxhlet apparatus (the use of an extraction thimble is optional).

[Note: The filter and sorbent assembly are tested together in order to reach detection limits, to minimize cost and to prevent misinterpretation of the data. Separate analyses of the filter and PUF would not yield useful information about the physical state of most of the PAHs at the time of sampling due to evaporative losses from the filter during sampling.]

10.3.3 Add between 300 and 350 mL of diethyl ether/hexane (10 percent v/v) to the Soxhlet apparatus. Reflux the sample for 18 hours at a rate of at least 3 cycles per hour. Allow to cool, then disassemble the apparatus.

10.3.4 Assemble a K-D concentrator (see Figure 4b) by attaching a 10-mL concentrator tube to a 500-mL evaporative flask.

10.3.5 Transfer the extract by pouring it through a drying column containing about 10 cm of anhydrous granular sodium sulfate (see Figure 4c) and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of 10 percent diethyl ether/hexane to complete the quantitative transfer.

10.3.6 Add one or two clean boiling chips and attach a 3-ball Snyder column to the evaporative flask. Pre-wet the Snyder column by adding about 1 mL of the extraction solvent to the top of the column. Place the K-D apparatus on a hot water bath (~50°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 1 hour. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches approximately 5 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 5 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 5 mL of cyclohexane. A 1-mL syringe is recommended for this operation.

10.3.7 Concentrate the extract to 5 mL and analyze using GC/MS.

10.3.8 The acceptance level of the cartridge is for each target PAH analyte to be less than or equal to the detection limit requirements to meet the project data qulity objectives. It is generally not possible to eliminate the presence of naphthalene, but the amount detected on the cleaned PUF cartridge should be less than five times the concentration of the lowest calibration standard (~500 ng). This amount is insignificant compared to the amount collected from a typical air sample.

In general, the following guidelines are provided in determining whether a cartridge is clean for field use:

- Naphthalene <500 ng/cartridge
- Other PAHs <200 ng total/cartridge

Cartridges are considered clean for up to 30 days from date of certification when sealed in their containers.

10.4 Deployment of Cartridges for Field Sampling

10.4.1 Immediately prior to field deployment, add surrogate compounds (i.e., chemically inert compounds not expected to occur in an environmental sample) to the center of the PUF cartridge, using a microsyringe. Spike 20 μL of a 50 μg/mL solution of the surrogates onto the center bed of the PUF trap to yield a final concentration of 1 μg. The surrogate compounds must be added to each cartridge assembly. The following field surrogate compounds should be added to each PUF cartridge prior to field deployment to monitor matrix effects, breakthrough, etc.

Field Surrogate Compound	Total Spiked Amount (µg)
D_{10}-Fluoranthene	1
D_{12}-Benzo(a)pyrene	1

Fill out a "chain-of-custody" indicating cartridge number, surrogate concentration, date of cartridge certification, etc. The chain-of-custody must accompany the cartridge to the field and return to the laboratory.

10.4.2 Use the recoveries of the surrogate compounds to monitor for unusual matrix effects and gross sample processing errors. Evaluate surrogate recovery for acceptance by determining whether the measured concentration falls within the acceptance limits of 60-120 percent.

10.4.3 Cartridges are placed in their shipping containers and shipped to the field. Blank cartridges do not need to be chilled when shipping to the field until after exposure to ambient air.

11. Assembly, Calibration, and Collection Using Sampling System

[Note: This method was developed using the PS-1 semi-volatile sampler provided by General Metal Works, Village of Cleves, OH as a guideline. EPA has experience in the use of this equipment during various field monitoring programs over the last several years. Other manufacturers' equipment should work as well; however, modifications to these procedures may be necessary if another commercially available sampler is selected.]

11.1 Sampling Apparatus

The entire sampling system is diagrammed in Figure 2. This apparatus was developed to operate at a rate of 4 to 10 scfm (0.114 to 0.285 std m^3/min) and is used by EPA for high-volume sampling of ambient air. The method write-up presents the use of this device.

The sampling module (see Figure 3) consists of a filter and a glass sampling cartridge containing the PUF utilized to concentrate PAHs from the air. A field portable unit has been developed by EPA (see Figure 6).

11.2 Calibration of Sampling System

Each sampler should be calibrated (1) when new, (2) after major repairs or maintenance, (3) whenever any audit point deviates from the calibration curve by more than 7 percent, (4) before/after each sampling event, and (5) when a different sample collection medium, other than that which the sampler was originally calibrated to, will be used for sampling.

11.2.1 Calibration of Orifice Transfer Standard. Calibrate the modified high volume air sampler in the field using a calibrated orifice flow rate transfer standard. Certify the orifice transfer standard in the laboratory against a positive displacement rootsmeter (see Figure 7). Once certified, the recertification is performed rather infrequently if the orifice is protected from damage. Recertify the orifice transfer standard performed once per year utilizing a set of five multi-hole resistance plates.

[Note: The set of five multihole resistance plates is used to change the flow through the orifice so that several points can be obtained for the orifice calibration curve. The following procedure outlines the steps to calibrate the orifice transfer standard in the laboratory.]

11.2.1.1 Record the room temperature (T_1 in °C) and barometric pressure (P_b in mm Hg) on the Orifice Calibration Data Sheet (see Figure 8). Calculate the room temperature in K (absolute temperature) and record on Orifice Calibration Data Sheet.

$$T_1 \text{ in K} = 273° + T_1 \text{ in °C}$$

11.2.1.2 Set up laboratory orifice calibration equipment as illustrated in Figure 7. Check the oil level of the rootsmeter prior to starting. There are three oil level indicators, one at the clear plastic end, and two sight glasses, one at each end of the measuring chamber.

11.2.1.3 Check for leaks by clamping both manometer lines, blocking the orifice with cellophane tape, turning on the high-volume motor, and noting any change in the rootsmeter's reading. If the rootsmeter's reading changes, there is a leak in the system. Eliminate the leak before proceeding. If the rootsmeter's reading remains constant, turn off the hi-vol motor, remove the cellophane tape, and unclamp both manometer lines.

11.2.1.4 Install the 5-hole resistance plate between the orifice and the filter adapter.

11.2.1.5 Turn manometer tubing connectors one turn counter-clockwise. Make sure all connectors are open.

11.2.1.6 Adjust both manometer midpoints by sliding their movable scales until the zero point corresponds with the meniscus. Gently shake or tap to remove any air bubbles and/or liquid remaining on tubing connectors. (If additional liquid is required for the water manometer, remove tubing connector and add clean water.)

11.2.1.7 Turn on the high-volume motor and let it run for 5 minutes to set the motor brushes. Turn the motor off. Ensure manometers are set to zero. Turn the high-volume motor on.

11.2.1.8 Record the time in minutes required to pass a known volume of air (approximately 5.6 to 8.4 m^3 of air for each resistance plate) through the rootsmeter by using the rootsmeter's digital volume dial and a stopwatch.

11.2.1.9 Record both manometer readings [orifice water manometer ($\triangle$H) and rootsmeter mercury manometer ($\triangle$P)] on Orifice Calibration Data Sheet (see Figure 8).

[Note: $\triangle H$ is the sum of the difference from zero (0) of the two column heights.]

11.2.1.10 Turn off the high-volume motor.

11.2.1.11 Replace the 5-hole resistance plate with the 7-hole resistance plate.

11.2.1.12 Repeat Sections 11.2.1.3 through 11.2.1.11.

11.2.1.13 Repeat for each resistance plate. Note results on Orifice Calibration Data Sheet (see Figure 8). Only a minute is needed for warm-up of the motor. Be sure to tighten the orifice enough to eliminate any leaks. Also check the gaskets for cracks.

[Note: The placement of the orifice prior to the rootsmeter causes the pressure at the inlet of the rootsmeter to be reduced below atmospheric conditions, thus causing the measured volume to be incorrect. The volume measured by the rootsmeter must be corrected.]

11.2.1.14 Correct the measured volumes on the Orifice Calibration Data Sheet:

$$V_{std} = V_m \left(\frac{P_a - \triangle P}{P_{std}}\right)\left(\frac{T_{std}}{T_a}\right)$$

where:

V_{std} = standard volume, std m^3

V_m = actual volume measured by the rootsmeter, m^3

P_a = barometric pressure during calibration, mm Hg

$\triangle P$ = differential pressure at inlet to volume meter, mm Hg

P_{std} = 760 mm Hg

T_{std} = 298 K

T_a = ambient temperature during calibration, K.

11.2.1.15 Record standard volume on Orifice Calibration Data Sheet.

11.2.1.16 The standard flow rate as measured by the rootsmeter can now be calculated using the following formula:

$$Q_{std} = \frac{V_{std}}{\theta}$$

where:

Q_{std} = standard volumetric flow rate, std m^3/min

θ = elapsed time, min

11.2.1.17 Record the standard flow rates to the nearest 0.01 std m^3/min.

11.2.1.18 Calculate and record $\sqrt{\triangle H\ (P_1/P_{std})(298/T_1)}$ value for each standard flow rate.

11.2.1.19 Plot each $\sqrt{\triangle H\ (P_1/P_{std})(298/T_1)}$ value (y-axis) versus its associated standard flow rate (x-axis) on arithmetic graph paper and draw a line of best fit between the individual plotted points.

[Note: This graph will be used in the field to determine standard flow rate.]

11.2.2 Calibration of the High-Volume Sampling System Utilizing Calibrated Orifice Transfer Standard

For this calibration procedure, the following conditions are assumed in the field:

- The sampler is equipped with an valve to control sample flow rate.
- The sample flow rate is determined by measuring the orifice pressure differential using a Magnehelic gauge.
- The sampler is designed to operate at a standardized volumetric flow rate of 8 ft^3/min (0.225 m^3/min), with an acceptable flow rate range within 10 percent of this value.
- The transfer standard for the flow rate calibration is an orifice device. The flow rate through the orifice is determined by the pressure drop caused by the orifice and is measured using a "U" tube water manometer or equivalent.
- The sampler and the orifice transfer standard are calibrated to standard volumetric flow rate units (scfm or scmm).
- An orifice transfer standard with calibration traceable to NIST is used.
- A "U" tube water manometer or equivalent, with a 0- to 16-inch range and a maximum scale division of 0.1 inch, will be used to measure the pressure in the orifice transfer standard.
- A Magnehelic gauge or equivalent with a 9- to 100-inch range and a minimum scale division of 2 inches for measurements of the differential pressure across the sampler's orifice is used.
- A thermometer capable of measuring temperature over the range of 32° to 122°F (0° to 50°C) to ±2°F (±1°C) and referenced annually to a calibrated mercury thermometer is used.
- A portable aneroid barometer (or equivalent) capable of measuring ambient barometric pressure between 500 and 800 mm Hg (19.5 and 31.5 in. Hg) to the nearest mm Hg and referenced annually to a barometer of known accuracy is used.
- Miscellaneous handtools, calibration data sheets or station log book, and wide duct tape are available.

11.2.2.1 Set up the calibration system as illustrated in Figure 9. Monitor the airflow through the sampling system with a venturi/Magnehelic assembly, as illustrated in Figure 9. Audit the field sampling system once per quarter using a flow rate transfer standard, as described in the EPA *High-Volume Sampling Method, 40 CVR 50, Appendix B.* Perform a single-point calibration before and after each sample collection, using the procedures described in Section 11.2.3.

11.2.2.2 Prior to initial multi-point calibration, place an empty glass cartridge in the sampling head and activate the sampling motor. Fully open the flow control valve and adjust the voltage variator so that a sample flow rate corresponding to 110 percent of the desired flow rate (typically 0.20 to 0.28 m^3/min) is indicated on the Magnehelic gauge (based on the previously obtained multipoint calibration curve). Allow the motor to warm up for 10 min and then adjust the flow control valve to achieve the desire flow rate. Turn off the sampler. Record the ambient temperature and barometric pressure on the Field Calibration Data Sheet (see Figure 10).

11.2.2.3 Place the orifice transfer standard on the sampling head and attach a manometer to the tap on the transfer standard, as illustrated in Figure 9. Properly align the retaining rings with the filter holder and secure by tightening the three screw clamps. Connect the orifice transfer standard by way of the pressure tap to a

manometer using a length of tubing. Set the zero level of the manometer or Magnehelic. Attach the Magnehelic gauge to the sampler venturi quick release connections. Adjust the zero (if needed) using the zero adjust screw on face of the gauge.

11.2.2.4 To leak test, block the orifice with a rubber stopper, wide duct tape, or other suitable means. Seal the pressure port with a rubber cap or similar device. Turn on the sampler.

Caution: *Avoid running the sampler for too long a time with the orifice blocked. This precaution will reduce the chance that the motor will be overheated due to the lack of cooling air. Such overheating can shorten the life of the motor*.

11.2.2.5 Gently rock the orifice transfer standard and listen for a whistling sound that would indicate a leak in the system. A leak-free system will not produce an upscale response on the sampler's magnehelic. Leaks are usually caused either by damaged or missing gaskets, by cross-threading, and/or not screwing sample cartridge together tightly. All leaks must be eliminated before proceeding with the calibration. When the sample is determined to be leak-free, turn off the sampler and unblock the orifice. Now remove the rubber stopper or plug from the calibrator orifice.

11.2.2.6 Turn the flow control valve to the fully open position and turn the sampler on. Adjust the flow control valve until a Magnehelic reading of approximately 70 in. is obtained. Allow the Magnehelic and manometer readings to stabilize and record these values on the orifice transfer Field Calibration Data Sheet (see Figure 10).

11.2.2.7 Record the manometer reading under Y1 and the Magnehelic reading under Y2 on the Field Calibration Data Sheet. For the first reading, the Magnehelic should still be at 70 inches as set above.

11.2.2.8 Set the Magnehelic to 60 inches by using the sampler's flow control valve. Record the manometer (Y1) and Magnehelic (Y2) readings on the Field Calibration Data Sheet (see Figure 10).

11.2.2.9 Repeat the above steps using Magnehelic settings of 50, 40, 30, 20, and 10 inches.

11.2.2.10 Turn the voltage variator to maximum power, open the flow control valve, and confirm that the Magnehelic reads at least 100 inches. Turn off the sampler and confirm that the Magnehelic reads zero.

11.2.2.11 Read and record the following parameters on the Field Calibration Data Sheet. Record the following on the calibration data sheet:

- Data, job number, and operator's signature.
- Sampler serial number.
- Ambient barometric pressure.
- Ambient temperature.

11.2.2.12 Remove the "dummy" cartridge and replace with a sample cartridge.

11.2.2.13 Obtain the manufacturer high volume orifice calibration certificate.

11.2.2.14 If not performed by the manufacturer, calculate values for each calibrator orifice static pressure (Column 6, inches of water) on the manufacturer's calibration certificate using the following equation:

$$\sqrt{\triangle H(P_a/760)[298/(T_a + 273)]}$$

where:

P_a = the barometric pressure (mm Hg) at time of manufacturer calibration, mm Hg
T_a = temperature at time of calibration, °C

11.2.2.15 Perform a linear regression analysis using the values in Column 7 of the manufacturer's High Volume Orifice Calibration Certificate for flow rate (Q_{std}) as the "X" values and the calculated values as the Y

values. From this relationship, determine the correlation (CC1), intercept (B1), and slope (M1) for the Orifice Transfer Standard.

11.2.2.16 Record these values on the Field Calibration Data Sheet (see Figure 10).

11.2.2.17 Using the Field Calibration Data Sheet values (see Figure 10), calculate the Orifice Manometer Calculated Values (Y3) for each orifice manometer reading using the following equation:

Y3 Calculation

$$Y3 = \{Y1(P_a/760)[298/(T_a + 273)]\}^{½}$$

11.2.2.18 Record the values obtained in Column Y3 on the Field Calibration Data Sheet (see Figure 10).

11.2.2.19 Calculate the Sampler Magnehelic Calculated Value (Y4) using the following equation:

Y4 Calculation

$$Y4 = \{Y2(P_a/760)[298/(T_a + 273)]\}^{½}$$

11.2.2.20 Record the value obtained in Column Y4 on the Field Calibration Data Sheet (see Figure 10).

11.2.2.21 Calculate the Orifice Flow Rate (X1) in scm using the following equation:

X1 Calculation

$$X1 = \frac{Y3 - B1}{M1}$$

11.2.2.22 Record the values obtained in Column X1 on the Field Calibration Data Sheet (see Figure 10).

11.2.2.23 Perform a linear regression of the values in Column X1 (as X) and the values in Column Y4 (as Y). Record the relationship for correlation (CC2), intercept (B2), and slope (M2) on the Field Calibration Data Sheet. The correlation coefficient must be 0.990 or greater.

11.2.2.24 Using the following equation, calculate a set point (SP) for the manometer to represent a desired flow rate:

Set Point

$$\text{Set point (SP)} = [(\text{Expected } P_a)/(\text{Expected } T_a)(T_{std}/P_{std})][M2\ (\text{Desired flow rate}) + B2]^2$$

where:

P_a = Expected atmospheric pressure (P_a), mm Hg
T_a = Expected atmospheric temperature (T_a), 273 + °C
M2 = Slope of developed relationship
B2 = Intercept of developed relationship
T_{std} = Temperature standard, 273 + 25°C
P_{std} = Pressure standard, 760 mm Hg

11.2.2.25 During monitoring, calculate a flow rate from the observed Magnehelic reading using the following equations:

Flow Rate

$$Y5 = [\text{Average Magnehelic Reading } (\triangle H)\ (P_a/T_a)(T_{std}/P_{std})]^{½}$$

$$X2 = \frac{Y5 - B2}{M2}$$

where:

Y5 = Corrected average magnehelic reading
X2 = Instant calculated flow rate, scm

11.2.2.26 The relationship in calibration of a sampling system between Orifice Transfer Standard and flow rate through the sampler is illustrated in Figure 11.

11.2.3 Single-Point Audit of the High Volume Sampling System Utilizing Calibrated Orifice Transfer Standard

Single point calibration checks are required as follows:

- Prior to the start of each 24-hour test period.
- After each 24-hour test period. The post-test calibration check may serve as the pre-test calibration check for the next sampling period if the sampler is not moved.
- Prior to sampling after a sample is moved.

For samplers, perform a calibration check for the operational flow rate before each 24-hour sampling event and when required as outlined in the user quality assurance program. The purpose of this check is to track the sampler's calibration stability. Maintain a control chart presenting the percentage difference between a sampler's indicated and measured flow rates. This chart provides a quick reference of sampler flow-rate drift problems and is useful for tracking the performance of the sampler. Either the sampler log book or a data sheet will be used to document flow-check information. This information includes, but is not limited to, sampler and orifice transfer standard serial number, ambient temperature, pressure conditions, and collected flow-check data.

In this subsection, the following is assumed:

- The flow rate through a sampler is indicated by the orifice differential pressure;
- Samplers are designed to operate at an actual flow rate of 8 scfm, with a maximum acceptable flow-rate fluctuation range of ±10 percent of this value;
- The transfer standard will be an orifice device equipped with a pressure tap. The pressure is measured using a manometer; and
- The orifice transfer standard's calibration relationship is in terms of standard volumetric flow rate (Q_{std}).

11.2.3.1 Perform a single point flow audit check before and after each sampling period utilizing the Calibrated Orifice Transfer Standard (see Section 11.2.1).

11.2.3.2 Prior to single point audit, place a "dummy" glass cartridge in the sampling head and activate the sampling motor. Fully open the flow control valve and adjust the voltage variator so that a sample flow rate corresponding to 110 percent of the desired flow rate (typically 0.19 to 0.28 m^3/min) is indicated on the Magnehelic gauge (based on the previously obtained multipoint calibration curve). Allow the motor to warm up for 10 minutes and then adjust the flow control valve to achieve the desired flow rate. Turn off the sampler. Record the ambient temperature and barometric pressure on the Field Test Data Sheet (see Figure 12).

11.2.3.3 Place the flow rate transfer standard on the sampling head.

11.2.3.4 Properly align the retaining rings with the filter holder and secure by tightening the three screw clamps. Connect the flow rate transfer standard to the manometer using a length of tubing.

11.2.3.5 Using tubing, attach one manometer connector to the pressure tap of the transfer standard. Leave the other connector open to the atmosphere.

11.2.3.6 Adjust the manometer midpoint by sliding the movable scale until the zero point corresponds with the water meniscus. Gently shake or tap to remove any air bubbles and/or liquid remaining on tubing connectors. (If additional liquid is required, remove tubing connector and add clean water.)

11.2.3.7 Turn on the high-volume motor and let run for 5 minutes.

11.2.3.8 Record the pressure differential indicated, $\triangle H$, in inches of water, on the Field Test Data Sheet. Be sure a stable $\triangle H$ has been established.

11.2.3.9 Record the observed Magnehelic gauge reading in inches of water on the Field Test Data Sheet. Be sure stable $\triangle M$ has been established.

11.2.3.10 Using previous established Orifice Transfer Standard curve, calculate Q_{xs} (see Section 11.2.2.23).

11.2.3.11 This flow should be within ±10 percent of the sampler set point, normally, 0.224 m^3. If not, perform a new multipoint calibration of the sampler.

11.2.3.12 Remove flow rate transfer standard and dummy sorbent cartridge.

11.3 Sample Collection

11.3.1 General Requirements

11.3.1.1 The sampler should be located in an unobstructed area, at least 2 meters from any obstacle to air flow. The exhaust hose should be stretched out in the downwind direction to prevent recycling of air into the sample head.

11.3.1.2 All cleaning and sample module loading and unloading should be conducted in a controlled environment, to minimize any chance of potential contamination.

11.3.1.3 When new or when using the sampler at a different location, all sample contact areas need to be cleaned. Use triple rinses of reagent grade hexane or methylene chloride contained in Teflon® rinse bottles. Allow the solvents to evaporate before loading the PUF modules.

11.3.2 Preparing Cartridge for Sampling

11.3.2.1 Detach the lower chamber of the cleaned sample head. While wearing disposable, clean, lint-free nylon, or cotton gloves, remove a clean glass sorbent module from its shipping container. Remove the Teflon® end caps (if applicable). Replace the end caps in the sample container to be reused after the sample has been collected.

11.3.2.2 Insert the glass module into the lower chamber and tightly reattach the lower chambers to the module.

11.3.2.3 Using clean rinsed (with hexane) Teflon®-tipped forceps, carefully place a clean conditioned fiber filter atop the filter holder and secure in place by clamping the filter holder ring over the filter. Place the

aluminum protective cover on top of the cartridge head. Tighten the 3 screw clamps. Ensure that all module connections are tightly assembled. Place a small piece of aluminum foil on the ball-joint of the sample cartridge to protect from back-diffusion of semi-volatiles into the cartridge during transporting to the site.

[Note: Failure to do so could expose the cartridge to contamination during transport.]

11.3.2.4 Place the cartridge in a carrying bag to take to the sampler.

11.3.3 Collection

11.3.3.1 After the sampling system has been assembled, perform a single point flow check as described in Sections 11.2.3.

11.3.3.2 With the empty sample module removed from the sampler, rinse all sample contact areas using reagent grade hexane in a Teflon® squeeze bottle. Allow the hexane to evaporate from the module before loading the samples.

11.3.3.3 With the sample cartridge removed from the sampler and the flow control valve fully open, turn the pump on and allow it to warm-up for approximately 5 minutes.

11.3.3.4 Attach a "dummy" sampling cartridge loaded with the exact same type of filter and PUF media to be used for sample collection.

11.3.3.5 Turn the sampler on and adjust the flow control valve to the desired flow as indicated by the Magnehelic gauge reading determined in Section 11.2.2.24. Once the flow is properly adjusted, take extreme care not to inadvertently alter its setting.

11.3.3.6 Turn the sampler off and remove the "dummy" module. The sampler is now ready for field use.

11.3.3.7 Check the zero reading of the sampler Magnehelic. Record the ambient temperature, barometric pressure, elapsed time meter setting, sampler serial number, filter number, and PUF cartridge number on the Field Test Data Sheet (see Figure 12). Attach the loaded sampler cartridge assembly to the sampler.

11.3.3.8 Place the voltage variator and flow control valve at the settings used in Section 11.3.2, and the power switch. Activate the elapsed time meter and record the start time. Adjust the flow (Magnehelic setting), if necessary, using the flow control valve.

11.3.3.9 Record the Magnehelic reading every 6 hours during the sampling period. Use the calibration factors (see Section 11.2.2.24) to calculate the desired flow rate. Record the ambient temperature, barometric pressure, and Magnehelic reading at the beginning and during sampling period.

11.3.4 Sample Recovery

11.3.4.1 At the end of the desired sampling period, turn the power off. Carefully remove the sampling head containing the filter and sorbent cartridge. Place the protective "plate" over the filter to protect the cartridge during transport to a clean recovery area. Also, place a piece of aluminum foil around the bottom of the sampler cartridge assembly.

11.3.4.2 Perform a final calculated sampler flow check using the calibration orifice, assembly, as described in Section 11.3.2. If calibration deviates by more than 10 percent from initial reading, mark the flow data for that sample as suspect and inspect and/or remove from service, record results on Field Test Data Sheet, Figure 12.

11.3.4.3 Transport the sampler cartridge assembly to a clean recovery area.

11.3.4.4 While wearing white cotton gloves, remove the PUF glass cartridge from the lower module chamber and lay it on the retained aluminum foil in which the sample was originally wrapped.

11.3.4.5 Carefully remove the quartz fiber filter from the upper chamber using clean Teflon®-tipped forceps.

11.3.4.6 Fold the filter in half twice (sample side inward) and place it in the glass cartridge atop the PUF.

11.3.4.7 Wrap the combined samples in the original hexane-rinsed aluminum foil, attach Teflon® end caps (if applicable) and place them in their *original* aluminum shipping container. Complete a sample label and affix it to the aluminum shipping container.

11.3.4.8 Chain-of-custody should be maintained for all samples. Store the containers under blue ice or dry ice and protect from UV light to prevent possibly photo-decomposition of collected analytes. If the time span between sample collection and laboratory analysis is to exceed 24 hours, refrigerate sample at 4°C.

11.3.4.9 Return at least one field blank filter/PUF cartridge to the laboratory with each group of samples. Treat a field blank exactly as the sample except that air is not drawn through the filter/sorbent cartridge assembly.

11.3.4.10 Ship and store field samples chilled (<4°C) using blue ice until receipt at the analytical laboratory, after which samples should be refrigerated at less than or equal to 4°C for up to 7 days prior to extraction; extracts should be analyzed within 40 days of extraction.

12. Sample Extraction, Concentration, and Cleanup

[Note: The following sample extraction, concentration, solvent exchange and analysis procedures are outlined for user convenience in Figure 13.]

12.1 Sample Identification

12.1.1 The chilled (<4°C) samples are returned in the aluminum shipping container (containing the filter and sorbents) to the laboratory for analysis. The "chain-of-custody" should be completed.

12.1.2 The samples are logged in the laboratory logbook according to sample location, filter and sorbent cartridge number identification, and total air volume sampled (uncorrected).

12.1.3 If the time span between sample registration and analysis is greater than 24-hours, then the sample must be kept refrigerated at <4°C. Minimize exposure of samples to fluorescent light. All samples should be extracted within one week (7 days) after sampling.

12.2 Soxhlet Extraction and Concentration

[Note: If PUF is the sorbent, the extraction solvent is 10 percent diethyl ether in hexane. If XAD-2® resin is the sorbent, the extraction solvent is methylene chloride.]

12.2.1 Assemble the Soxhlet apparatus (see Figure 4a). Immediately before use, charge the Soxhlet apparatus with 700 to 750 mL of 10 percent diethyl ether in hexane and reflux for 2 hours. Let the apparatus cool, disassemble it, transfer the diethyl ether in hexane to a clean glass container, and retain it as a blank for later analysis, if required. Place the sorbent and filter together in the Soxhlet apparatus (the use of an extraction thimble is optional).

[Note: The filter and sorbent are analyzed together in order to reach detection limits, avoid questionable interpretation of the data, and minimize cost.]

12.2.1.1 Prior to extraction, add appropriate laboratory surrogate standards to the Soxhlet solvent. A surrogate standard (i.e., a chemically compound not expected to occur in an environmental sample) should be added to each sample, blank, and matrix spike sample just prior to extraction or processing. The recovery of the laboratory surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measure concentration falls within the acceptance limits. Spike 20 μL of a 50 μg/mL solution of the surrogates onto the PUF cartridge, prior to Soxhlet extraction, to yield a final concentration of 1 μg. The following laboratory surrogate standards have been

successfully utilized in determining Soxhlet extraction effects, sample process errors, etc., for GC/MS/DS analysis.

Laboratory Surrogate Standard	Total Spiked Amount (µg)
D_{10}-Fluorene	1
D_{10}-Pyrene	1

Section 13.2 outlines preparation of the laboratory surrogates. Add the laboratory surrogate compounds to the PUF cartridge. Add 700 mL of 10 percent diethyl ether in hexane to the apparatus and reflux for 18 hours at a rate of at least 3 cycles per hour. Allow to cool, then disassemble the apparatus.

12.2.1.2 Dry the extract from the Soxhlet extraction by passing it though a drying column containing about 10 grams of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator assembly. Wash the extractor flask and sodium sulfate column with 100-125 mL of 10 percent diethyl ether/hexane to complete the quantitative transfer.

12.2.2 Assemble a K-D concentrator (see Figure 4b) by attaching a 10 mL concentrator tube to a 500 mL evaporative flask.

[Note: Other concentration devices (vortex evaporator) or techniques may be used in place of the K-D as long as qualitative and quantitative recovery can be demonstrated.]

12.2.2.1 Add two boiling chips, attach a three-ball macro-Snyder column to the K-D flask, and concentrate the extract using a water bath at 60 to 65°C. Place the K-D apparatus in the water bath so that the concentrator tube is about half immersed in the water and the entire rounded surface of the flask is bathed with water vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in one hour. At the proper rate of distillation, the balls of the column actively chatter but the chambers do not flood. When the liquid has reached an approximate volume of 5 mL, remove the K-D apparatus from the water bath and allow the solvent to drain for at least 5 minutes while cooling.

12.2.2.2 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 5 mL of cyclohexane. A 5 mL syringe is recommended for this operation. The extract is now ready for further concentration to 1.0 mL by nitrogen blowdown.

12.2.2.3 Place the 1 mL calibrated K-D concentrator tube with an open micro-Snyder attachment in a warm water bath (30 to 3 5°C) and evaporate the solvent volume to just below 1 mL by blowing a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon) above the extract.

12.2.2.4 The internal wall of the concentrator tube must be rinsed down several times with hexane during the operation.

12.2.2.5 During evaporation, the tube solvent level must be kept below the water level of the bath. the extract must never be allowed to become dry.

12.2.2.6 Bring the final volume back to 1.0 mL with hexane. Transfer the extract to a Teflon®-sealed screw-cap amber vial, label the vial, and store at 4°C (±2°C).

[Note: It is not necessary to bring the volume to exactly 1.0 mL if the extract will be cleaned up by solid phase extraction cleanup methods. Final volume is brought to 1.0 mL after cleanup.]

12.3 Sample Cleanup

12.3.1 If the extract is cloudy, impurities may be removed from the extract by solid phase extraction using activated silica gel. Clean-up procedures may not be needed for relatively clean matrix samples.

12.3.2 Approximately 10 grams of silica gel, type 60 (70-230 mesh), are extracted in a Soxhlet extractor with 10 percent diethyl ether for 6 hours (minimum rate, 3 cycles/hr) and then activated by heating in a foil-covered glass container for 16 hours at 150°C.

12.3.3 Using a disposable Pasteur pipette (7.5-mm x 14.6-cm), place a small piece of glass wool in the neck of the pipette. Prepare a slurry of activated silica gel in 10 percent diethyl ether. Place 10 grams of the activated silica gel slurry into the column using additional 10 percent diethyl ether. Finally, 1 gram of anhydrous sodium sulfate is added to the top of the silica gel. Prior to use, the column is rinsed with 10 percent diethyl ether at 1 mL/min for 1 hour to remove any trace of contaminants. It is then pre-eluted with 40 mL of pentane and the eluate discarded.

12.3.4 While the pentane pre-elutant covers the top of the column, 1 mL of the sample extract is transferred to the column, and washed on with 2 mL of *n*-hexane to complete the transfer. Allow to elute through the column. Immediately prior to exposure of the sodium sulfate layer the air, add 25 mL of pentane and continue the elution process. The pentane eluate is discarded.

12.3.5 The column is finally eluted at 2 mL/min with 25 mL of 10 percent diethyl ether in pentane (4:6 v/v) and collected in a 50 mL K-D flask equipped with a 5 mL concentrator tube for concentration to less than 5 mL. The concentrate is further concentrated to 1.0 mL under a gentle stream of nitrogen as previously described.

12.3.6 The extract is now ready for GC/MS analysis. Spike the extract with internal standards (ISs) before analysis. The following internal standards (ISs) have been successfully used in PAH analysis by GC/MS.

Internal Standard (IS)	**Total Spiked Amount (µg)**
D_8-Naphthalene	0.5
D_{10}-Acenaphthene	0.5
D_{10}-Phenanthrene	0.5
D_{12}-Chrysene	0.5
D_{12}-Perylene	0.5

Section 13.2 outlines preparation of the ISs.

13. Gas Chromatography with Mass Spectrometry Detection

13.1 General

13.1.1 The analysis of the extracted sample for benzo[a]pyrene and other PAHs is accomplished by an electron ionization gas chromatograph/mass spectrometer (EI GC/MS) in the mode with a total cycle time (including voltage reset time) of 1 second or less. The GC is equipped with an DB-5 fused silica capillary column (30-m x 0.32-mm I.D.) with the helium carrier gas for analyte separation. The GC column is temperature controlled and interfaced directly to the MS ion source.

13.1.2 The laboratory must document that the EI GC/MS system is properly maintained through periodic calibration checks. The GC/MS system should be operated in accordance with specifications outlined in Table 2.

13.1.3 The GC/MS is tuned using a 50 ng/µL solution of decafluorotriphenylphosphine (DFTPP). The DFTPP permits the user to tune the mass spectrometer on a daily basis. If properly tuned, the DFTPP key ions and ion abundance criteria should be met as outlined in Table 3.

13.1.4 The GC/MS operating conditions are outlined in Table 2. The GC/MS system should be calibrated using the internal standard technique. Figure 14 outlines the following sequence involving the GC/MS calibration.

13.2 Calibration of GC/MS/DS

13.2.1 Standard Preparation

Stock PAH Standards Including Surrogate Compounds

13.2.1.1 Prepare stock standards of B[a]P and other PAHs. The stock standard solution of B[a]P (2.0 µg/µL) and other PAHs can be user prepared from pure standard materials or can be purchased commercially.

13.2.1.2 Place 0.2000 grams of native B[a]P and other PAHs on a tared aluminum weighing disk and weigh on a Mettler balance.

13.2.1.3 Quantitatively transfer the material to a 100 mL volumetric flask. Rinse the weighing disk with several small portions of 10 percent diethyl ether/hexane. Ensure all material has been transferred.

13.2.1.4 Dilute to mark with 10 percent diethyl ether/hexane.

13.2.1.5 The concentration of the stock standard solution of B[a]P or other PAHs in the flask is 2.0 µg/µL.

[Note: Commercially prepared stock PAH standards may be used at any concentration if they are certified by the manufacturer or by an independent source.]

13.2.1.6 Transfer the stock standard solutions into Teflon®-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

13.2.1.7 Stock PAH standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

Mix Internal Standard (IS) Solution

13.2.1.8 For PAH analysis, deuterated internal standards are selected that are similar in analytical behavior to the compound of interest. The following internal standards are suggested for PAH analysis:

D_{12}-Perylene
Benzo(e)pyrene
Benzo(a)pyrene
Benzo(k)fluoranthene

D_{10}-Acenaphthene
Acenaphthene (if using XAD-2® as the sorbent)
Acenaphthylene (if using XAD-2® as the sorbent)
Fluorene
Benzo(g,h,i)perylene
Dibenz(a,h)anthracene
Indeno(1,2,3-cd)pyrene
Perylene
Benzo(b)fluoranthene
Coronene

D_{12}-Chrysene
Benz(a)anthracene
Chrysene
Pyrene

D_8-Naphthalene
Naphthalene (if using XAD-2® as the sorbent)

D_{10}-Phenanthrene
Anthracene
Fluoranthene
Phenanthrene

13.2.1.9 Purchase a mix IS solution containing specific IS needed for quantitation at a concentration of 2,000 ng/µL.

Mixed Stock PAH Standard Including Surrogate Compounds

13.2.1.10 Prepare a mixed stock PAH standard by taking 125 µL of the stock PAH standard(s) and diluting to mark with hexane in a 10-mL volumetric flask. The concentration of the mixed stock PAH standard(s) is 25 ng/µL.

Calibration PAH Standards Including Surrogate Compounds

13.2.1.11 Calibration PAH standards can be generated from the stock PAH standard using serial dilution utilizing the following equation:

$$C_1V_1 = C_2V_2$$

where:

C_1 = Concentration of stock PAH standards, ng/µL
V_1 = Volume of stock PAH standard solution taken to make calibration PAH standards, µL
V_2 = Final volume diluted to generate calibration PAH standards, µL
C_2 = Final concentration of calibration PAH standards, ng/µL

13.2.1.12 Using the above equation, prepare a series of calibration PAH standards which include the surrogate compounds (i.e., 2.50 ng/µL, 1.25 ng/µL, 0.50 ng/µL, 0.25 ng/µL, and 0.10 ng/µL) according to the scheme illustrated in Table 4 and described below.

- For CAL 5, transfer 1.00 mL of the mixed PAH stock standard in a 10-mL volumetric flask and dilute to 10.0 mL with hexane. The resulting concentration is 2.5 ng/µL for the PAH analytes.
- To prepare CAL 4, transfer 500 µL of the mixed PAH stock standard solution to a 10-mL volumetric flask and dilute to 10.0 mL with hexane. The resulting concentration is 1.25 ng/µL for PAH analytes.
- To prepare CAL 3, transfer 200 µL of the mixed PAH stock solution to a 10-mL volumetric flask and dilute to 10-mL with hexane. The resulting concentration is 0.50 ng/µL for PAH analytes.
- To prepare CAL 2, transfer 100 μL of the mixed PAH stock solution to a 10-mL volumetric flask and dilute to 10-mL with hexane. The resulting concentration is 0.25 ng/μL for PAH analytes.
- To prepare CAL 1, transfer 40 µL of the mixed PAH stock solution to a 10-mL volumetric flask and dilute to 10-mL with hexane. The resulting concentration is 0.10 ng/µL for PAH analytes.

13.2.2 Internal Standard Spiking

13.2.2.1 Prior to GC/MS analysis, each 1 mL aliquot of the five calibration standards is spiked with internal standard to a final concentration of 0.5 ng/µL. To do this, first prepare a 1:40 dilution of the 2,000 ng/µL mixed internal standard solution by diluting 250 µL to a volume of 10 mL to yield a concentration of 50 ng/µL.

13.2.2.2 Each 1.0-mL portion of calibration standard and sample extract is then spiked with 10 µL of the internal standard solution prior to analysis by GC/MS/DS operated in the SCAN mode.

13.2.3 Storage, Handling, and Retention of Standards

13.2.3.1 Store the stock and mixed standard solutions at 4°C (±2°C) in Teflon®-lined screw-cap amber bottles. Store the working standard solutions at 4°C (±2°C) in Teflon®-lined screw-cap amber bottles.

13.2.3.2 Protect all standards from light. Samples, sample extracts, and standards must be stored separately.

13.2.3.3 Stock standard solutions must be replaced every 12 months, or sooner, if comparison with quality control check samples indicates a problem. Diluted working standards are usable for 6 months. Analysis difficulties, which warrant investigation, may require preparation of new standards. All standards are securely stored at ~4°C (±2°C) but above freezing. The concentration, preparation and expiration date, and solvent are identified on standard vial labels. Each standard is uniquely identified with its laboratory notebook number and a prefix. This procedure helps provide traceability to standard preparation.

13.2.3.4 Take care to maintain the integrity of each standard. The solvent, hexane, is volatile and can easily evaporate. Make sure each vial is sealed after use, and mark the solvent level on the side of the vial. When retrieving a vial for use, if the solvent level does not match the mark, dispose of the standard and obtain a new one.

13.3 GC/MS Instrument Operating Conditions

13.3.1 Gas Chromatograph (GC). The following are the recommended GC analytical conditions, as also outlined in Table 3, to optimize conditions for compound separation and sensitivity.

Carrier Gas:	Helium
Linear Velocity:	28-29 cm^3/sec
Injector Temperature:	250-300°C
Injector:	Grob-type, splitless, 2 µL
Temperature Program:	Initial Temperature: 70°C
Initial Hold Time:	4.0 ± 0.1 min.
Ramp Rate:	10°C/min to 300°C, hold for 10 min
Final Temperature:	300°C
Final Hold Time:	10 min (or until all compounds of interest have eluted).
Analytical Time:	Approximately 50 min.

13.3.2 Mass Spectrometer. Following are the required mass spectrometer conditions for scan data acquisition:

Transfer Line Temperature:	290°C
Source Temperature:	According to manufacturer's specifications
Electron Energy:	70 volts (nominal)
Ionization Mode:	EI
Mass Range:	35 to 500 amu, SCAN data acquisition
Scan Time:	At least 5 scans per peak, not to exceed 1 second per scan

13.3.3 Instrument Performance Check for GC/MS.

13.3.3.1 Summary. It is necessary to establish that the GC/MS meet tuning and standard mass spectral abundance criteria prior to initiating any on-going data collection, as illustrated in Figure 14. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP).

13.3.3.2 Frequency. The instrument performance check solution of DFTPP will be analyzed initially and once per 12-hour time period of operation. Also, whenever the laboratory takes corrective action which may change or affect the mass spectral criteria (e.g., ion source cleaning or repair, column replacement, etc.), the instrument performance check must be verified irrespective of the 12-hour laboratory requirement. The 12-hour

time period for GC/MS analysis begins at the injection of the DFTPP, which the laboratory submits as documentation of a compliance tune. The time period ends after 12 hours have elapsed. To meet instrument performance check requirements, samples, blanks, and standards must be injected within 12 hours of the DFTPP injection.

13.3.3.3 Procedure. Inject 50 ng of DFTPP into the GC/MS system. DFTPP may be analyzed separately or as part of the calibration standard.

13.3.3.4 Technical Acceptance Criteria. The following criteria have been established in order to generate accurate data:

- Prior to the analysis of any samples, blanks, or calibration standards, the laboratory must establish that the GC/MS system meets the mass spectral ion abundance criteria for the instrument performance check solution containing DFTPP.
- The GC/MS system must be tuned to meet the manufacturer's specifications, using a suitable calibrant. The mass calibration and resolution of the GC/MS system are verified by the analysis of the instrument performance check solution.
- The abundance criteria listed in Table 3 must be met for a 50 ng injection of DFTPP. The mass spectrum of DFTPP must be acquired by averaging three scans (the peak apex scan and the scans immediately preceding and following the apex). Background subtraction is required, and must be accomplished using a single scan prior to the elution of DFTPP.

[Note: All ion abundance MUST be normalized to m/z 198, the nominal base peak, even though the ion abundances of m/z 442 may be up to 110 percent of m/z 198.]

- The above criteria are based on adherence to the acquisition specifications identified in Table 4 and were developed for the specific target compound list associated with this document. The criteria are based on performance characteristics of instruments currently utilized in routine support of ambient air program activities. These specifications, in conjunction with relative response factor criteria for target analytes, are designed to control and monitor instrument performance associated with the requirements if this document. As they are performance-based criteria for these specific analytical requirements, they may not be optimal for additional target compounds.
- If the mass spectrometer has the ability for autotuning, then the user may utilize this function following manufacturer's specifications. Autotune automatically adjusts ion source parameters within the detector using FC-43 (Heptacos). Mass peaks at m/z 69, 219, and 502 are used for tuning. After the tuning is completed, the FC-43 abundances at m/z 50, 69, 131, 219, 414, 502, and 614 are further adjusted such that their relative intensities match the selected masses of DFTPP.

13.3.3.5 Corrective Action. If the DFTPP acceptance criteria are not met, the MS must be retuned. It may be necessary to clean the ion source, or quadrupoles, or take other actions to achieve the acceptance criteria. DFTPP acceptance criteria MUST be met before any standards, or required blanks, are analyzed. Any standards, field samples, or required blanks analyzed when tuning criteria have not been met will require reanalysis.

13.3.4 Initial Calibration for GC/MS.

13.3.4.1 Summary. Prior to the analysis of samples and required blanks, and after tuning criteria (instrument performance check) have been met, each GC/MS system will be initially calibrated at a minimum of five concentrations to determine instrument sensitivity and the linearity of GC/MS response for the analyte compounds and the surrogates.

13.3.4.2 Frequency. Each GC/MS system must be initially calibrated whenever the laboratory takes corrective action, which may change or affect the initial calibration criteria (e.g., ion source cleaning or repair,

column replacement, etc.), or if the continuing calibration acceptance criteria have not been met. If time still remains in the 12-hour time period after meeting the technical acceptance criteria for the initial calibration, samples may be analyzed. It is not necessary to analyze a continuing calibration standard within the 12-hour time period if the initial calibration standard (CAL 3) is the same concentration as the continuing calibration standard and both meet the continuing calibration technical acceptance criteria. Quantify all sample results using the mean of the relative response factors ($\overline{\text{RRFs}}$) from the initial calibration.

13.3.4.3 Procedure. Perform the following activities to generate quantitative data:

- Set up the GC/MS system.
- Warm all standard/spiking solutions, sample extracts, and blanks to ambient temperature (~1 hour) before analysis.
- Tune the GC/MS system to meet the technical acceptance criteria (see Section 13.3.3).
- Prepare five calibration standards containing the target compounds, internal standards, and surrogate compounds at the concentrations outlined in Table 4.
- Calibrate the GC/MS by injecting 2.0 µL of each standard. If a compound saturates when the CAL 5 standard is injected, and the system is calibrated to achieve a detection sensitivity of no less than the MDL for each compound, the laboratory must document it and attach a quantitation report and chromatogram. In this instance, the laboratory must calculate the results based on a four-point initial calibration for the *specific compound* that saturates. Secondary ion quantitation is only allowed when there are sample interferences with the primary quantitation ion. If secondary ion quantitation is used, calculate a relative response factor using the area response from the most intense secondary ion which is free of interferences and document the reasons for the use of the secondary ion.
- Record a mass spectrum of each target compound. Figure 15(a) through 15(q) documents the mass spectrum for each of the 16 target PAHs discussed in Compendium Method TO-13A. Judge the acceptability of recorded spectra by comparing them to spectra in libraries. If an acceptable spectrum of a calibration standard component is not acquired, take necessary actions to correct GC/MS performance. If performance cannot be corrected, report sample extract data for the particular compound(s), but document the affected compound(s) and the nature of the problem.

13.3.4.4 Calculations. Perform the following calculations to generate quantitative data:

[Note: In the following calculations, the area response is that of the primary quantitation ion unless otherwise stated.]

- **Relative Response Factors (RRFs)**. Calculate RRFs for each analyte target compound and surrogate using the following equation with the appropriate internal standard. Table 5 outlines characteristic ions for the surrogate compounds and internal standards. Table 6 outlines primary quantitation ions for each PAH. Use the following equation for RRF calculation.

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where:

A_x = area of the primary quantitation ion for the compound to be measured, counts
A_{is} = area of the primary quantitation ion for the internal standard, counts
C_{is} = concentration or amount of the internal standard, ng/µL

C_x = concentration or amount of the compound to be measured, ng/µL

- **Percent Relative Standard Deviation (%RSD)**. Using the RRFs from the initial calibration, calculate the %RSD for all target compounds and surrogates using the following equations:

$$\%RSD = \frac{SD_{RRF}}{\bar{x}} \times 100$$

and

$$SD_{RRF} = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N - 1}}$$

where:

SD_{RRF} = standard deviation of initial response factors (per compound)
x = mean of initial relative response factors (per compound)
X_i = ith RRF
N = number of determinations

- **Relative Retention Times (RRT)**. Calculate the RRTs for each target compound and surrogate over the initial calibration range using the following equation:

$$RRT = \frac{RT_c}{RT_{is}}$$

where:

RT_c = retention time of the target compound, minutes
RT_{is} = retention time of the internal standard, minutes

- **Mean of the Relative Retention Times ($\overline{RRT}$)**. Calculate the mean of the relative retention times ($\overline{RRT}$) for each analyte target compound and surrogate over the initial calibration range using the following equation:

$$\overline{RRT} = \sum_{i=1}^{n} \frac{RRT_i}{n}$$

where:

$\overline{RRT}$ = mean relative retention time for the target compound or surrogate for each initial calibration standard, minutes
RRT = relative retention time for the target compound or surrogate for each initial calibration standard, minutes

- **Mean Area Response ($\overline{Y}$) for Internal Standard**. Calculate the area response (Y) mean for primary quantitation ion each internal standard compound over the initial calibration range using the following equation:

$$\overline{Y} = \sum_{i=1}^{n} \frac{Y_i}{n}$$

where:

$\overline{Y}$ = mean area response, counts
Y_i = area response for the primary quantitation ion for the internal standard for each calibration standard, counts

- **Mean of the Retention Time ($\overline{RT}$) For Internal Standard**. Calculate the mean of the retention times ($\overline{RT}$) for each internal standard over the initial calibration range using the following equation:

$$\overline{RT} = \sum_{i=1}^{n} \frac{RT_i}{n}$$

where:
$\overline{RT}$ = mean retention time, minutes
RT = retention time for the internal standard for each initial calibration standard, minutes

13.3.4.5 Technical Acceptance Criteria. All initial calibration standards must be analyzed at the concentration levels at the frequency described in Section 13.3.3 on a GC/MS system meeting the DFTPP instrument performance check criteria.

- The relative response factor (RRF) at each calibration concentration for each target compound and surrogate that has a required minimum response factor value must be greater than or equal to the minimum acceptable relative response factor (see Table 7) of the compound.
- The percent relative standard deviation (%RSD) over the initial calibration range for each target compound and surrogate that has a required maximum %RSD must be less than or equal to the required maximum value (see Table 7). For all the other target compounds, the value for %RSD must be less than or equal to 30 percent. When the value for %RSD exceeds 30 percent, analyze additional aliquots of appropriate CALs to obtain an acceptable %RSD of RRFs over the entire concentration range, or take action to improve GC/MS performance.
- The relative retention time for each of the target compounds and surrogates at each calibration level must be within ±0.06 relative retention time units of the mean relative retention time for the compound.
- The retention time shift for each of the internal standards at each calibration level must be within ±20.0 seconds compared to the mean retention time ($\overline{RT}$) over the initial calibration range for each internal standard.
- The compounds must meet the minimum RRF and maximum %RSD criteria for the initial calibration.

13.3.4.6 Corrective Action. If the technical acceptance criteria for initial calibration are not met, the system should be inspected for problems. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the acceptance criteria. Initial calibration technical acceptance criteria MUST

be met before any samples or required blanks are analyzed in a 12-hour time period for an initial calibration analytical sequence.

13.3.5 Continuing Calibration.

13.3.5.1 Summary. Prior to the analysis of samples and required blanks and after tuning criteria have been met, the initial calibration of each GC/MS system must be routinely checked by analyzing a continuing calibration standard (see Table 4, CAL 3) to ensure that the instrument continues to meet the instrument sensitivity and linearity requirements of the method. The continuing calibration standard (CAL 3) shall contain the appropriate target compounds, surrogates, and internal standards.

13.3.5.2 Frequency. Each GC/MS used for analysis must be calibrated once every time period of operation. The 12-hour time period begins with injection of DFTPP. If time still remains in the 12-hour time period after meeting the technical acceptance criteria for the initial calibration, samples may be analyzed. It is not necessary to analyze a continuing calibration standard within this 12-hour time period, if the initial calibration standard that is the same concentration as the continuing calibration standard meets the continuing calibration technical acceptance criteria.

13.3.5.3 Procedure. The following activities should be performed for continuing calibration:

- Set up the GC/MS system as specified by the manufacturer.
- Tune the GC/MS system to meet the technical acceptance criteria (see Section 13.3.3).
- Analyze the CAL 3 standard solution containing all the target analytes, surrogate compounds, and internal standards using the procedure listed for the initial calibration.
- Allow all standard/spiking solutions and blanks to warm to ambient temperature (approximately 1 hour) before preparation or analysis.
- Start the analysis of the continuing calibration by injecting 2.0 µL of the CAL 3 standard solution.

13.3.5.4 Calculations. The following calculations should be performed:

- **Relative Response Factor (RRF)**. Calculate a relative response factor (RRF) for each target compound and surrogate.
- **Percent Difference (%D)**. Calculate the percent difference between the mean relative response factor ($\overline{RRF}$) from the most recent initial calibration and the continuing calibration RRF for each analyte target compound and surrogate using the following equation:

$$\%D_{RRF} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

where:

$\%D_{RRF}$ = percent difference between relative response factors

$\overline{RRF_i}$ = average relative response factor from the most recent initial calibration

RRF_c = relative response factor from the continuing calibration standard

13.3.5.5 Technical Acceptance Criteria. The continuing calibration standard must be analyzed for the compounds listed in concentration levels at the frequency described and on a GC/MS system meeting the DFTPP instrument performance check and the initial calibration technical acceptance criteria. The relative response factor for each target analyte and surrogate that has a required minimum relative response factor value must be greater than or equal to the compound's minimum acceptable relative response factor. For an acceptable

continuing calibration, the %D between the measured RRF for each target/surrogate compound of the CAL 3 standard and the mean value calculated during initial calibration must be within ±30 percent. If the criteria for %D are not met for the target or surrogate compounds, remedial action must be taken and recalibration may be necessary.

13.3.5.6 Corrective Action. If the continuing calibration technical acceptance criteria are not met, recalibrate the GC/MS instrument. It may be necessary to clean the ion source, change the column, or take other corrective actions to achieve the acceptance criteria. Continuing calibration technical acceptance criteria ***MUST*** be met before any samples or required blanks are analyzed in a 12-hour continuing calibration analytical sequence. Any samples or required blanks analyzed when continuing calibration criteria were not met will require reanalysis. Remedial actions, which include but are not limited to the following, must be taken if criteria are not met:

- Check and adjust GC and/or MS operating conditions.
- Clean or replace injector liner.
- Flush column with solvent according to manufacturers instructions.
- Break off a short portion (approximately 0.33 cm) of the column.
- Replace the GC column (performance of all initial calibration procedures are then required).
- Adjust MS for greater or lesser resolution.
- Calibrate MS mass scale.
- Prepare and analyze new continuing calibration.
- Prepare a new initial calibration curve.

13.3.6 Laboratory Method Blank (LMB).

13.3.6.1 Summary. The purpose of the LMB is to monitor for possible laboratory contamination. Perform all steps in the analytical procedure using all reagents, standards, surrogate compounds, equipment, apparatus, glassware, and solvents that would be used for a sample analysis. An LMB is an unused, certified filter/cartridge assembly which is carried though the same extraction procedure as a field sample. The LMB extract must contain the same amount of surrogate compounds and internal standards that is added to each sample. All field samples must be extracted and analyzed with an associated LMB.

13.3.6.2 Frequency. Analyze an LMB along with each batch of ≤20 samples through the entire extraction, concentration, and analysis process. The laboratory may also analyze a laboratory reagent blanks which is the same as an LMB except that no surrogate compounds or internal standards are added. This demonstrates that reagents contain no impurities producing an ion current above the level of background noise for quantitation ions for those compounds.

13.3.6.3 Procedure. Extract and analyze a clean, unused filter and glass cartridge assembly.

13.3.6.4 Technical Acceptance Criteria. Following are the technical criteria for the LMB:

- All blanks must be analyzed on a GC/MS system meeting the DFTPP instrument performance check and initial calibration or continuing calibration technical acceptance criteria.
- The percent recovery for each of the surrogates in the blank must be within the acceptance windows.
- The area response change for each of the internal standards for the blank must be within -50 percent and +100 percent compared to the internal standards in the most recent continuing calibration analysis.
- The retention time for each of the internal standards must be within ±20.0 seconds between the blank and the most recent CAL 3 analysis.
- The LMB must not contain any target analyte at a concentration greater than the MDL and must not contain additional compounds with elution characteristics and mass spectral features that would interfere

with identification and measurement of a method analyte at its MDL. If the LMB that was extracted along with a batch of samples is contaminated, the entire batch of samples must be flagged.

13.3.6.5 Corrective Action. Perform the following if the LCBs exceed criteria:

- If the blanks do not meet the technical acceptance criteria, the analyst must consider the analytical system to be out of control. It is the analyst's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measure *MUST* be taken and documented before further sample analysis proceeds.
- All samples processed with a method blank that is out of control (i.e., contaminated) will require data qualifiers to be attached to the analytical results.

13.3.7 Laboratory Control Spike (LCS).

13.3.7.1 Summary. The purpose of the LCS is to monitor the extraction efficiency of Compendium Method TO-13A target analytes from a clean, uncontaminated PUF cartridge. An LCS is an unused, certified PUF that is spiked with the target analytes (1 μg) and carried through the same extraction procedures as the field samples. The LCS must contain the same amount of surrogate compounds and internal standards that is added to each sample. All field samples must be extracted and analyzed with an associated LCS. All steps in the analytical procedure must use the same reagents, standards, surrogate compounds, equipment, apparatus, glassware, and solvents that would be used for a sample analysis.

13.3.7.2 Frequency. Analyze an LCS along with each of $\leq$20 samples through the entire extraction, concentration, and analysis. (The laboratory may also analyze a laboratory reagent blank which is the same as an LMB except that no surrogate compounds or internal standards are added. This demonstrates that reagents contain no impurities producing an ion current above the level of background noise for quantitation ions of those compounds.)

13.3.7.3 Procedure. Extract and analyze a clean, unused certified PUF cartridge assembly.

13.3.7.4 Technical Acceptance Criteria. Technical criteria for the LCS are:

- All LCSs must be analyzed on a GC/MS system meeting the DFTPP instrument performance check and initial calibration or continuing calibration technical acceptance criteria.
- The percent recovery for each of the surrogates in the LCS must be within the acceptance windows.
- The area response change for each of the internal standards for the LCS must be within -50 percent and +100 percent compared to the internal standards in the most recent continuing calibration analysis.
- The retention time for each of the internal standards must be within ±20.0 seconds between the LCS and the most recent CAL 3 analysis.
- All target analytes spiked on the certified PUF cartridge must meet a percent recovery between 60-120 to be acceptable.

13.3.7.5 Corrective Action. Perform the following if the LCS exceed criteria:

- If the LCS do not meet the technical acceptance criteria, the analyst must consider the analytical system to be out of control. It is the analyst's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms be eliminated. If contamination is a problem, the source of the contamination must be investigated and appropriate corrective measure *MUST* be taken and documented before further sample analysis proceeds.

- All samples processed with a LCS that is out of control (i.e., contaminated) will require re-analysis or data qualifiers to be attached to the analytical results.

13.4 Sample Analysis by GC/MS

13.4.1 Summary. The sample extract is analyzed by GC/MS and quantitated by the internal standard method.

13.4.2 Frequency. Before samples can be analyzed, the instrument must meet the GC/MS tuning and initial calibration or continuing calibration technical acceptance criteria. If there is time remaining in the 12-hour time period with a valid initial calibration or continuing calibration, samples may be analyzed in the GC/MS system that meet the instrument performance check criteria.

13.4.3 Procedure. For sample analysis, perform the following:

- Set up the GC/MS system.
- All sample extracts must be allowed to warm to ambient temperature (~1 hour) before analysis. All sample extracts must be analyzed under the same instrumental conditions as the calibration standards.
- Add the internal standard spiking solution to the 1.0 mL extract. For sample dilutions, add an appropriate amount of the internal standard spiking solution to maintain the concentration of the internal standards at 2 ng/µL in the diluted extract.
- Inject 2.0 µL of sample extract into the GC/MS, and start data acquisition.
- When all semi-volatile target compounds have eluted from the GC, terminate the MS data acquisition and store data files on the data system storage device. Use appropriate data output software to display full range mass spectra and SICPs. The sample analysis using the GC/MS is based on a combination of retention times and relative abundances of selected ions (see Table 6). These qualifiers should be stored on the hard disk of the GC/MS data computer and are applied for identification of each chromatographic peak. The retention time qualifier is determined to be +0.10 minute of the library retention time of the compound. The acceptance level for relative abundance is determined to be ±15% of the expected abundance. Three ions are measured for most of the PAH compounds. When compound identification is made by the computer, any peak that fails any of the qualifying tests is flagged (e.g., with an *). The data should be manually examined by the analyst to determine the reason for the flag and whether the compound should be reported as found. Although this step adds some subjective judgment to the analysis, computer-generated identification problems can be clarified by an experienced operator. Manual inspection of the quantitative results should also be performed to verify concentrations outside the expected range.

13.4.4 Dilutions. The following section provides guidance when an analyte exceeds the calibration curve.

- When a sample extract is analyzed that has an analyte target compound concentration greater than the upper limit of the initial calibration range or saturated ions from a compound excluding the compound peaks in the solvent front), the extract must be diluted and reanalyzed. Secondary ion quantitation is *only* allowed when there are sample interferences with the primary quantitation ion. If secondary ion quantitation is used, calculate a relative response factor using the area response for the most intense secondary ion which is free of sample interferences, and document the reasons for the use of the secondary ion.
- Calculate the sample dilution necessary to keep the semi-volatile target compounds that required dilution within the upper half of the initial calibration range so that no compound has saturated ions (excluding the compound peaks in the solvent front). Dilute the sample in hexane in a volumetric flask. Analyze the sample dilution.

- The dilution factor chosen should keep the response of the largest peak for a *target compound* in the upper half of the initial calibration range of the instrument.
- If the on-column concentration of any target compound in any sample exceeds the initial calibration range, that sample must be diluted, the internal standard concentration readjusted, and the sample extract reanalyzed.
- Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.

13.4.5 Quantitation. This section provides guidance for quantitating PAH analytes.

- Target components identified shall be quantified by the internal standard method. The internal standards used for the target compounds are the ones nearest the retention time of a given analyte.
- The relative response factor (RRF) from the daily continuing calibration standard analysis (or RRF of CAL 3) if the sample is analyzed in the same 12-hour sequence as the initial calibration) is used to calculate the concentration in the sample. Secondary ion quantitation is allowed *only* when there are sample interferences with the primary ion. If secondary ion quantitation is performed, document the reasons. The area of a secondary ion cannot be substituted for the area of a primary ion unless a relative response factor is calculated using the secondary ion.
- A retention time window is calculated for each single component analyte and surrogate. Windows are established as ±0.01 RRT units of the retention time for the analyte in CAL 3 of the initial calibration or the continuing calibration.

13.4.6 Calculations. Perform the following calculations:

13.4.6.1 Calculation of Concentration. Calculate target compound concentrations using the following equation:

$$\text{Concentration, (ng/std m}^3\text{)} = \frac{A_x I_s V_t D_f}{A_{is} V_i \overline{RRF}}$$

where:

A_x = area response for the compound to be measured, counts
A_{is} = area response for the internal standard, counts
I_s = amount of internal standard, ng/µL
$\overline{RRF}$ = the mean RRF from the most recent initial calibration, dimensionless
V_i = volume of air sampled, std m^3
V_t = volume of final extract, µL
D_f = dilution factor for the extract. If there was no dilution, D_f equals 1. If the sample was diluted, the D_f is greater than 1.

The concentrations calculated can be converted to ppb_v for general reference. The analyte concentration can be converted to ppb_v using the following equation:

$$C_A(ppb_v) = C_A(ng/m^3) \times 24.4/MW_A$$

where:

C_A = concentration of analyte calculated, ng/std. m^3
MW_A = molecular weight of analyte, g/g-mole
24.4 = molar volume occupied by ideal gas at standard temperature and pressure (25°C and 760 mm Hg), L/mole.

13.4.6.2 Estimated Concentration. The equation in Section 13.4.6.1 is also used for calculating the concentrations of the non-target compounds. Total area counts (or peak heights) from the total ion chromatogram generated by the mass spectrometer for Compendium Method TO-13A PAHs (see Figure 16) are to be used for both the non-target compound to be measured (A_x) and the internal standard (A_{is}). Associate the nearest internal standard free of interferences with the non-target compound to be measured. A relative response factor (RRF) of one (1) is to be assumed. The value from this quantitation shall be qualified as estimated ("J") (estimated, due to lack of a compound-specific response factor) and "N" (presumptive evidence of presence), indicating the quantitative and qualitative uncertainties associated with this non-target component. An estimated concentration should be calculated for all tentatively identified compounds (TICs) as well as those identified as unknowns.

13.4.6.3 Surrogate Percent Recovery (%R). Calculate the surrogate percent recovery using the following equation:

$$\%R = \frac{Q_d}{Q_a} \times 100$$

where:
Q_d = Quantity determined by analysis, ng
Q_a = Quantity added to sample/blank, ng

The surrogate percent recovery must fall between 60-120% to be acceptable.

13.4.6.4 Percent Area Response Change (%ARC). Calculate the percent area response change (%ARC) for the sample/blank analysis compared to the most recent CAL 3 analysis for each of the internal standard compounds using the following equation:

$$\%ARC = \frac{A_s - A_x}{A_x} \times 100$$

where:
%ARC = percent area response change, %
A_s = area response of the internal standard in the sample/blank analysis, counts
A_x = area response of the internal standard in the most recent CAL 3 analysis, counts

The area change for the internal standard must not exceed -50 to +100 percent.

13.4.6.5 Internal Standard Retention Time Shift (RTS). Calculate the retention time shift (RTS) between the sample/blank analysis and the most recent CAL 3 analysis for each of the internal standards using the following equation:

$$RTS = RT_s - RT_x$$

where:

RT_s = retention time of the IS in the sample
RT_x = retention time of the IS in the most recent CAL 3 analysis.

13.4.7 Technical Acceptance Criteria. The following guideline is provided as technical acceptance criteria.

13.4.7.1 All target compound concentrations must not exceed the upper limit of the initial calibration range and no compound ion (excluding the compound peaks in the solvent front) may saturate the detector.

13.4.7.2 Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 20 seconds from the latest continuing calibration standard or CAL 3 if samples are analyzed in the same 12-hour sequence as the initial calibration, the chromatographic system must be inspected for malfunctions, and corrections made as required. The SICP of the internal standards must be monitored and evaluated for each field and QC sample. If the SICP area for any internal standard changes by more than a factor of -50 to +100 percent, the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. If the analysis of a subsequent sample or standard indicates that the system is functioning properly, then corrections may not be required.

13.4.7.3 When target compounds are below the low standard, but the spectrum meets the identification criteria, report the concentration/amount with a "J." For example, if the low standard corresponds to 0.1μg and an amount of 0.05 μg is calculated, report as "0.05J."

13.4.8 Corrective Action. The following section provides guidance if analyte exceeds the technical criteria.

- If the sample technical acceptance criteria for the surrogates and internal standards are not met, check calculations, surrogate and internal standard solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the surrogate and internal standard technical acceptance criteria.
- Sample analysis technical acceptance criteria *must* be met before data are reported. Samples contaminated from laboratory sources, or associated with a contaminated method blank, or any samples analyzed that are not meet the technical acceptance criteria will require reanalysis.
- The samples or standards with SICP areas outside the limits must be reanalyzed. If corrections are made, then the laboratory must demonstrate that the mass spectrometric system is functioning properly. This must be accomplished by the analysis of a standard or sample that meets the SICP criteria. After corrections are made, the reanalysis of samples analyzed while the system was malfunctioning is required.
- If after reanalysis, the SICP areas for all internal standards are inside the technical acceptance limits (-50 to +100 percent), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, submit *only* data from the analysis with SICPs within the technical acceptance limits. This is considered the *initial* analysis and must be reported as such on all data deliverables.
- If the reanalysis of the sample does not solve the problem (i.e., the SICP areas are outside the technical acceptance limits for both analyses) then the laboratory must submit the SICP data and sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables, using the sample suffixes specified.
- Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window.
- If sample peaks are not detected, or all are less than full-scale deflection, the undiluted extract is acceptable for GC/MS analysis. If any sample ions are greater than the 120 percent of the initial calibration curve range, calculate the dilution necessary to reduce the major ion to between half- and full-range response.

14. Quality Assurance/Quality Control (QA/QC)

14.1 General System QA/QC

14.1.1 Each laboratory that uses Compendium Method TO-13A must operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality data. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate a typical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation**.**

14.1.2 Before processing any samples, the analyst should demonstrate, through the analysis of a reagent solvent blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent solvent blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement steps.

14.1.3 For each analytical batch (up to 20 samples), a reagent blank, matrix spike, and deuterated/surrogate samples must be analyzed (the frequency of the spikes may be different for different monitoring programs). The blank and spiked samples must be carried through all stages of the sample preparation and measurement steps.

14.1.4 The experience of the analyst performing GC/MS is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration sample should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Are the response windows obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g., column changed), recalibration of the system must take place.

14.2 Process, Field, and Solvent Blanks

14.2.1 One PUF cartridge and filter from each batch of approximately 20 should be analyzed without shipment to the field for the compounds of interest to serve as a process blank. A blank level specified in Section 10.2 for each cartridge/filter assembly is considered to be acceptable.

14.2.2 During each sampling episode, at least one cartridge and filter should be shipped to the field and returned, without drawing air through the sampler, to serve as a field blank.

14.2.3 During the analysis of each batch of samples at least one solvent process blank (all steps conducted but no cartridge or filter included) should be carried through the procedure and analyzed. Blank levels should be those specified in Section 10.2 for single components to be acceptable.

14.2.4 Because the sampling configuration (filter and backup sorbent) has been tested for targeted PAHs in the laboratory in relationship to collection efficiency and has been demonstrated to be greater than 95 percent for targeted PAHs (except naphthalene, acenaphthylene, and acenaphthene), no field recovery evaluation is required as part of the QA/QC program outlined in this section.

15. References

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TABLE 1. FORMULAE AND PHYSICAL PROPERTIES OF SELECTED PAHs

Compound	Formula	Molecular Weight	Melting Point, °C	Boiling Point, °C	Vapor Pressure, kPa	CAS RN #
Naphthalene	$C_{10}H_8$	128.18	80,2	218	1.1x10	91-20-3
Acenaphthylene	$C_{12}H_8$	152.20	92-93	265-280	3.9x10	208-96-8
Acenaphthene	$C_{12}H_{10}$	154.20	90-96	278-279	2.1x10	83-32-9
Fluorene	$C_{13}H_{10}$	166.23	116-118	293-295	8.7x10	86-73-7
Anthracene	$C_{14}H_{10}$	178.24	216-219	340	36x10	120-12-7
Phenanthrene	$C_{14}H_{10}$	178.24	96-101	339-340	2.3x10	85-01-8
Fluoranthene	$C_{16}H_{10}$	202.26	107-111	375-393	6.5x10	206-44-0
Pyrene	$C_{16}H_{10}$	202.26	150-156	360-404	3.1x10	129-00-0
Benz(a)anthracene	$C_{18}H_{12}$	228.30	157-167	435	1.5x10	56-55-3
Chrysene	$C_{18}H_{12}$	228.30	252-256	441-448	5.7x10	218-01-9
Benzo(b)fluoranthene	$C_{20}H_{12}$	252.32	167-168	481	6.7x10	205-99-2
Benzo(k)fluoranthene	$C_{20}H_{12}$	252.32	198-217	480-471	2.1x10	207-08-9
Perylene	$C_{20}H_{12}$	252.32	273-278	500-503	7.0x10	198-55-8
Benzo(a)pyrene	$C_{20}H_{12}$	252.32	177-179	493-496	7.3x10	50-32-8
Benzo(e)pyrene	$C_{20}H_{12}$	252.32	178-179	493	7.4x10	192-92-2
Benzo(g,h,i)perylene	$C_{22}H_{12}$	276.34	275-278	525	1.3x10	191-24-2
Indeno(1,2,3-cd)pyrene	$C_{22}H_{12}$	276.34	162-163	--	ca.10	193-39-5
Dibenz(a,h)anthracene	$C_{22}H_{14}$	278.35	266-270	524	1.3x10	53-70-3
Coronene	$C_{24}H_{12}$	300.36	438-440	525	2.0x10	191-07-1

Many of these compounds sublime.

TABLE 2. GC-MS OPERATING CONDITIONS

Activity	Conditions
Gas Chromatography	
Column	J&W Scientific, DB-5 crosslinked 5% phenylmethyl silicone (30 m x 0.32 mm, 1.0 µm film thickness) or equivalent
Carrier Gas	Helium, velocity between 28-30 cm^3/sec at 250°C
Injection Volume	2 µL, Grob-type, splitless
Injector Temperature	290°C
Temperature Program	
Initial Column Temperature	70°C
Initial Hold Time	4 ± 0.1 min.
Program	10°C/min to 300°C and hold 10 min.
Final Temperature	300°C
Final Hold Time	10 min. or until all compounds of interest have eluted
Mass Spectrometer	
Transfer Line Temperature	290°C or According to Manufacturer's Specification
Source Temperature	According to Manufacturer's Specifications
Electron Energy	70 volts (nominal)
Ionization Mode	EI
Mass Range	35 to 500 amu, full range data acquisition (SCAN) mode
Scan Time	At least 5 scans per peak, not to exceed 1 second per scan.

TABLE 3. DFTPP KEY IONS & ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30 to 60% of mass 198
68 70	Less than 2% of mass 69 Less than 2% of mass 69
127	40 to 60% of mass 198
197 198 199	Less than 2% of mass 198 Base peak, 100% relative abundance 5 to 9% of mass 198
275	10 to 30% of mass 198
365	Greater than 1.0% of mass 198
441 442 443	Present but less than mass 443 40% of mass 198 17 to 23% of mass 442

TABLE 4. COMPOSITION AND APPROXIMATE CONCENTRATION OF CALIBRATION SOLUTIONS

Target Compound	Concentration, ng/µL				
	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5
PAHs	0.10	0.25	0.50	1.25	2.50
Acenaphthene	0.10	0.25	0.50	1.25	2.50
Acenaphthylene	0.10	0.25	0.50	1.25	2.50
Anthracene	0.10	0.25	0.50	1.25	2.50
Benz(a)anthracene	0.10	0.25	0.50	1.25	2.50
Benzo(a)pyrene	0.10	0.25	0.50	1.25	2.50
Benzo(b)fluoranthene	0.10	0.25	0.50	1.25	2.50
Benzo(e)pyrene	0.10	0.25	0.50	1.25	2.50
Benzo(g,h,i)perylene	0.10	0.25	0.50	1.25	2.50
Benzo(k)fluoranthene	0.10	0.25	0.50	1.25	2.50
Chrysene	0.10	0.25	0.50	1.25	2.50
Perylene	0.10	0.25	0.50	1.25	2.50
Dibenz(a,h)anthracene	0.10	0.25	0.50	1.25	2.50
Fluoranthene	0.10	0.25	0.50	1.25	2.50
Fluorene	0.10	0.25	0.50	1.25	2.50
Indeno(1,2,3-c,d)pyrene	0.10	0.25	0.50	1.25	2.50
Naphthalene	0.10	0.25	0.50	1.25	2.50
Coronene	0.10	0.25	0.50	1.25	2.50
Phenanthrene	0.10	0.25	0.50	1.25	2.50
Pyrene	0.10	0.25	0.50	1.25	2.50

TABLE 4. (Continued)

Target Compound	Concentration, ng/µL				
	CAL 1	CAL 2	CAL 3	CAL 4	CAL 5
SUGGESTED INTERNAL STANDARDS					
D_8-Naphthalene	0.5	0.5	0.5	0.5	0.5
D_{10}-Acenaphthene	0.5	0.5	0.5	0.5	0.5
D_{10}-Phenanthrene	0.5	0.5	0.5	0.5	0.5
D_{12}-Chrysene	0.5	0.5	0.5	0.5	0.5
D_{12}-Perylene	0.5	0.5	0.5	0.5	0.5
SUGGESTED SURROGATE COMPOUNDS					
D_{10}-Fluoranthene (field)	0.10	0.25	0.50	1.25	2.50
D_{12}-Benzo[a]pyrene (field)	0.10	0.25	0.50	1.25	2.50
D_{10}-Fluorene (lab)	0.10	0.25	0.50	1.25	2.50
D_{10}-Pyrene (lab)	0.10	0.25	0.50	1.25	2.50

TABLE 5. CHARACTERISTIC IONS FOR SURROGATE SUGGESTED STANDARDS

Classification	Primary Ion	Secondary Ion
Internal Standards		
D_8-Naphthalene	136	68,137
D_{10}-Acenaphthene	164	162,165
D_{10}-Phenanthrene	188	94,189
D_{12}-Chrysene	240	120,241
D_{12}-Perylene	264	260,265
Laboratory Surrogates		
D_{10}-Fluorene	176	88,177
D_{10}-Pyrene	212	106,213
Field Surrogates		
D_{10}-Fluoranthene	212	106,213
D_{12}-Benzo(a)pyrene	264	132,265

TABLE 6. EXAMPLE OF CHARACTERISTIC IONS FOR COMMON PAHs

Analyte	Primary Ion	Secondary Ion(s)
Pyrene	202	101,203
Benz(a)anthracene	228	229,226
Chrysene	228	226,229
Benzo(a)pyrene	252	253,126
Benzo(b)fluoranthene	252	253,126
Benzo(k)fluoranthene	252	253,126
Benzo(g,h,i)perylene	276	138,277
Dibenz(a,h)anthracene	278	139,279
Anthracene	178	179,176
Phenanthrene	178	179,176
Acenaphthene	154	153,152
Acenaphthylene	152	151,153
Benzo(e)pyrene	252	253,126
Fluoranthene	202	101,203
Fluorene	166	165,167
Ideno(1,2,3-cd)pyrene	276	138,227
Naphthalene	128	129,127
Perylene	252	253,126
Coronene	300	150,301

TABLE 7. EXAMPLE OF RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION OF COMMON SEMI-VOLATILE COMPOUNDS

Semi-volatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Difference
Naphthalene	0.700	30	30
Acenaphthylene	1.300	30	30
Acenaphthene	0.800	30	30
Fluorene	0.900	30	30
Phenanthrene	0.700	30	30
Anthracene	0.700	30	30
Fluoranthene	0.600	30	30
Pyrene	0.600	30	30
Benz(a)anthracene	0.800	30	30
Chrysene	0.700	30	30
Benzo(b)fluoranthene	0.700	30	30
Benzo(k)fluoranthene	0.700	30	30
Benzo(a)pyrene	0.700	30	30
Indeno(1,2,3-cd)pyrene	0.500	30	30
Dibenz(a,h)anthracene	0.400	30	30
Benzo(g,h,i)perylene	0.500	30	30
Perylene	0.500	30	30
Coronene	0.700	30	30

TABLE 8. MINIMUM SAMPLING EQUIPMENT CALIBRATION AND ACCURACY REQUIREMENTS

Equipment	Acceptance limits	Frequency and method of measurement	Action if require-ments are not met
Sampler	Indicated flow rate = true flow rate, ±10%.	Calibrate with certified transfer standard on receipt, after maintenance on sampler, and any time audits or flow checks deviate more than ±10% from the indicated flow rate or ±10% from the design flow rate.	Recalibrate
Associated equipment			
Sampler on/off timer	±30 min/24 hour	Check at purchase and routinely on sample-recovery days	Adjust or replace
Elapsed-time meter	±30 min/24 hour	Compare with a standard time-piece of known accuracy at receipt and at 6-month intervals	Adjust or replace
Flowrate transfer standard (orifice device)	Check at receipt for visual damage	Recalibrate annually against positive displacement standard volume meter	Adopt new calibration curve

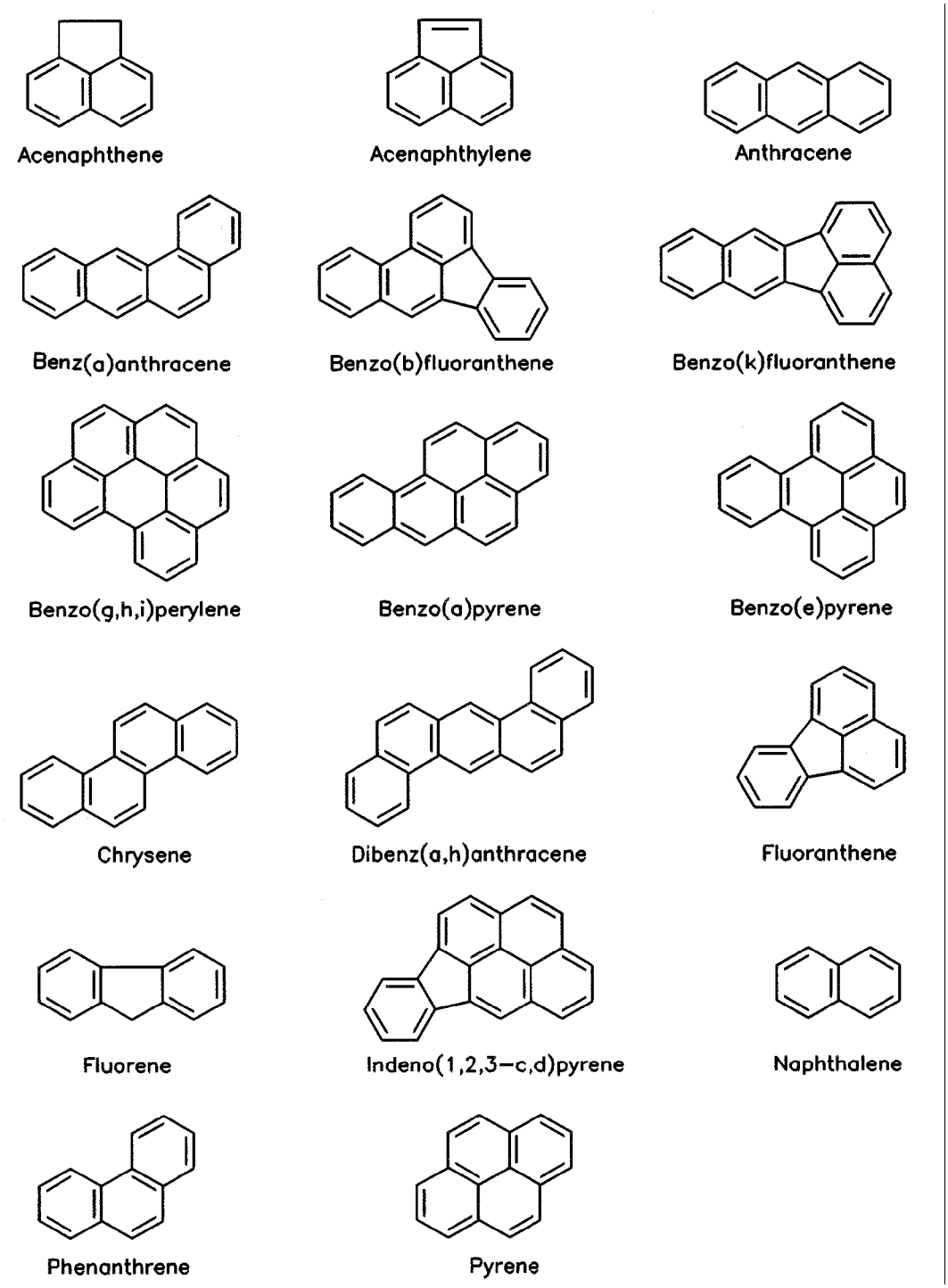


Figure 1. Ring structure of common PAHs.

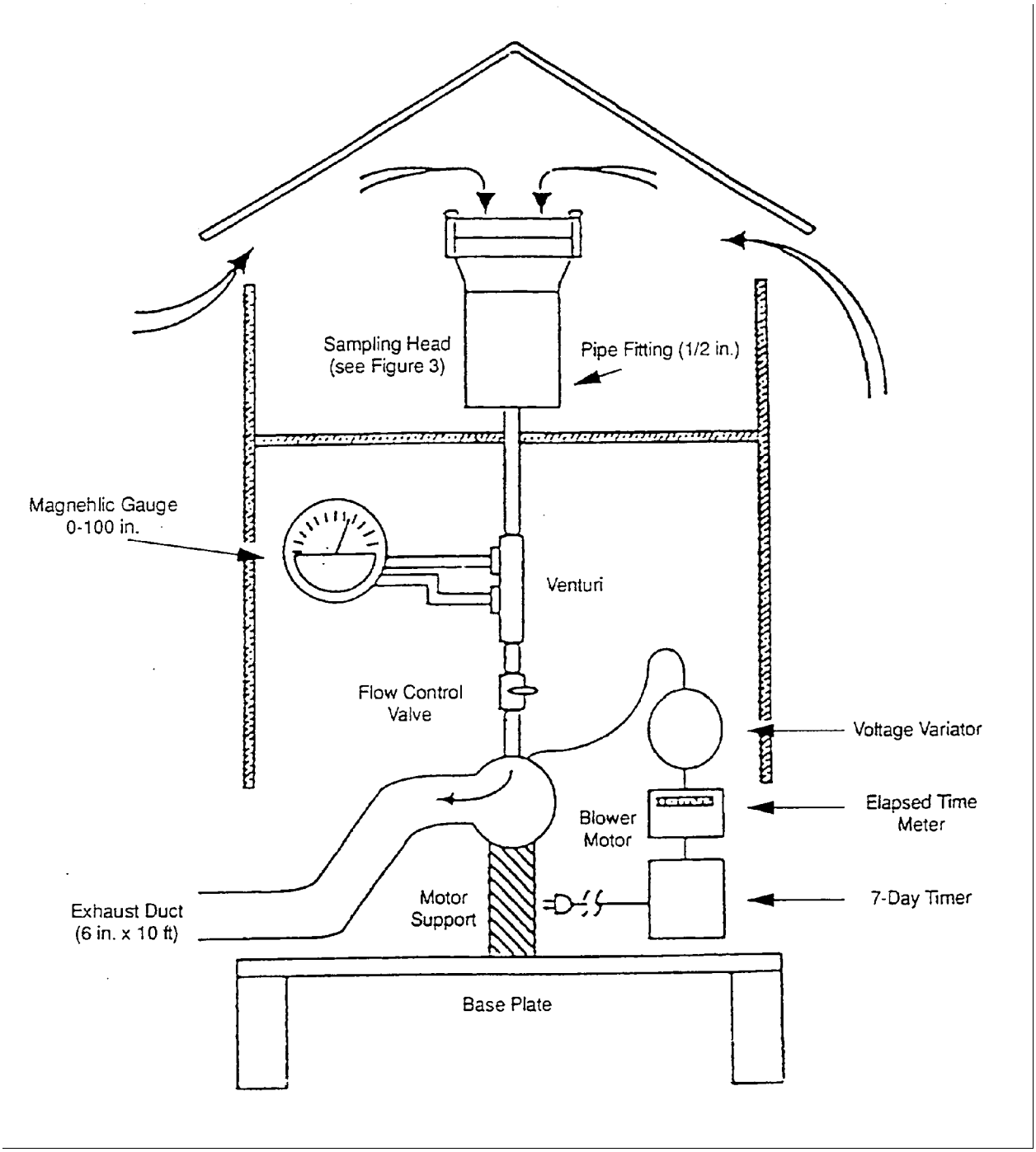


Figure 2. Typical high volume air sampler for PAHs.

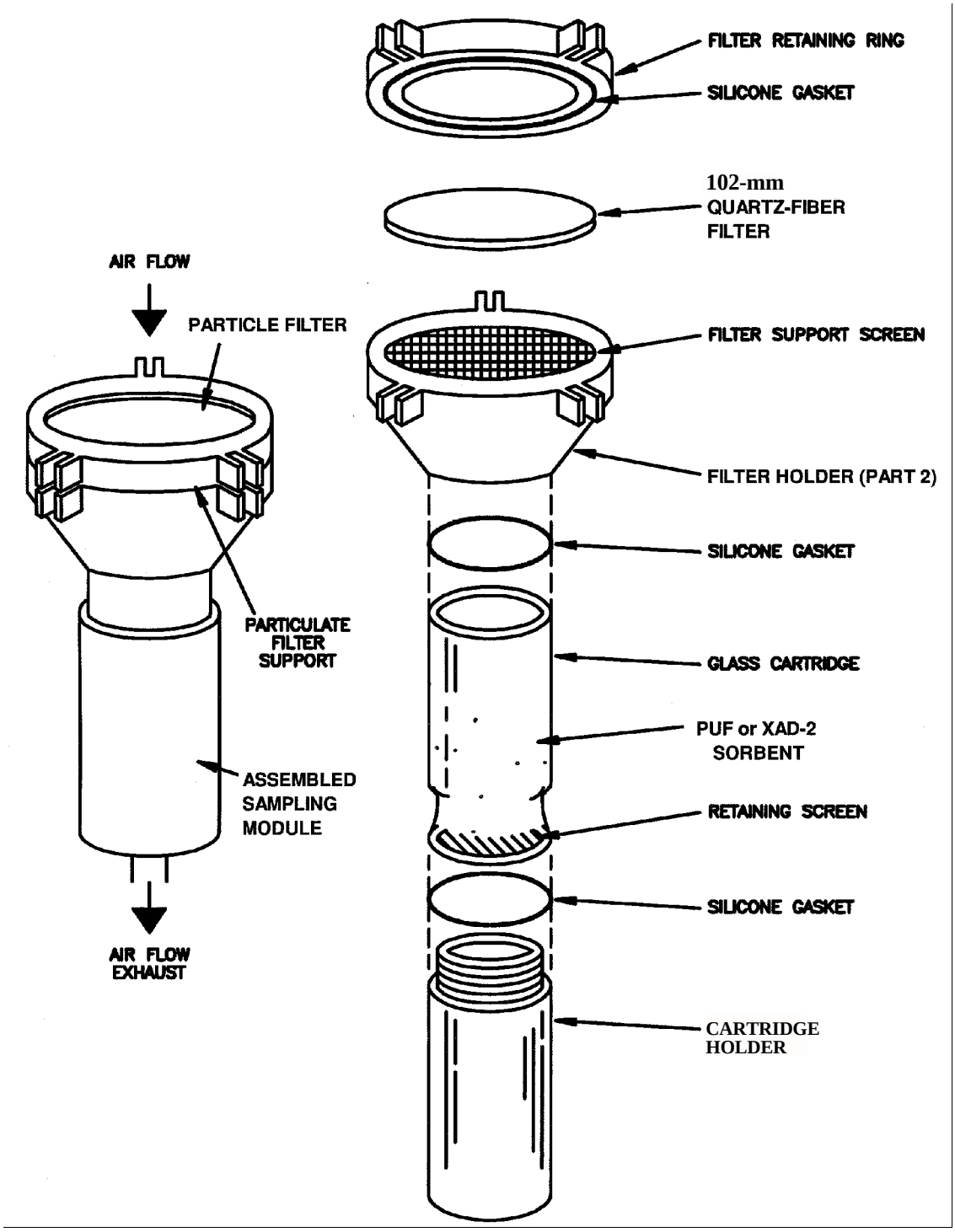


Figure 3. Typical absorbent cartridge assembly for sampling PAHs.

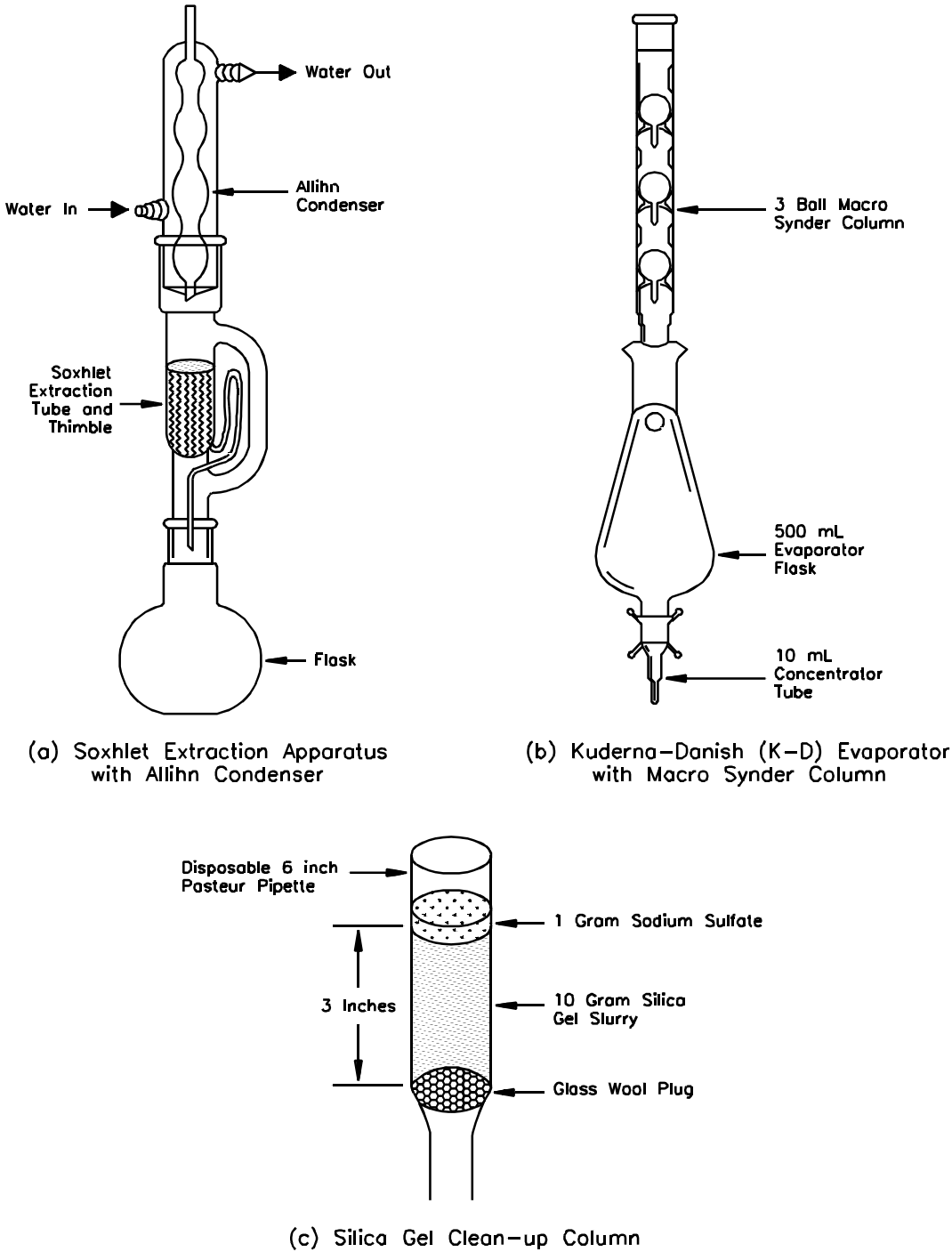
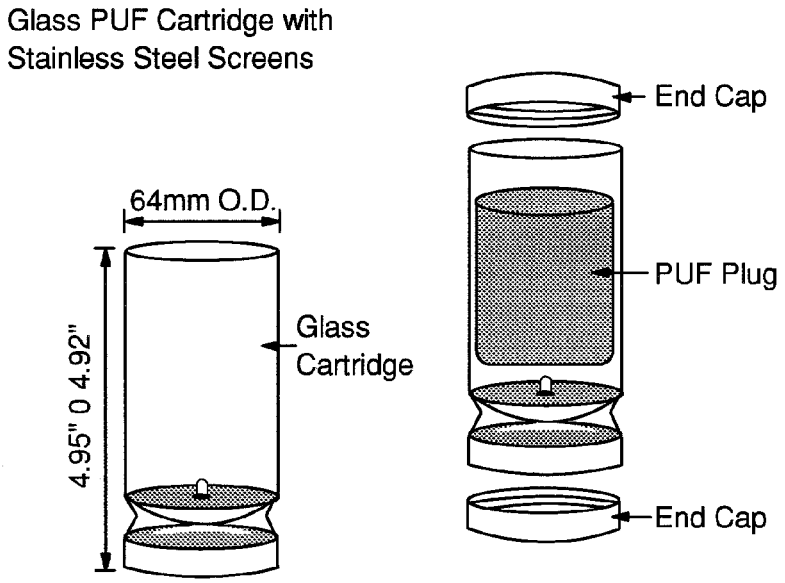
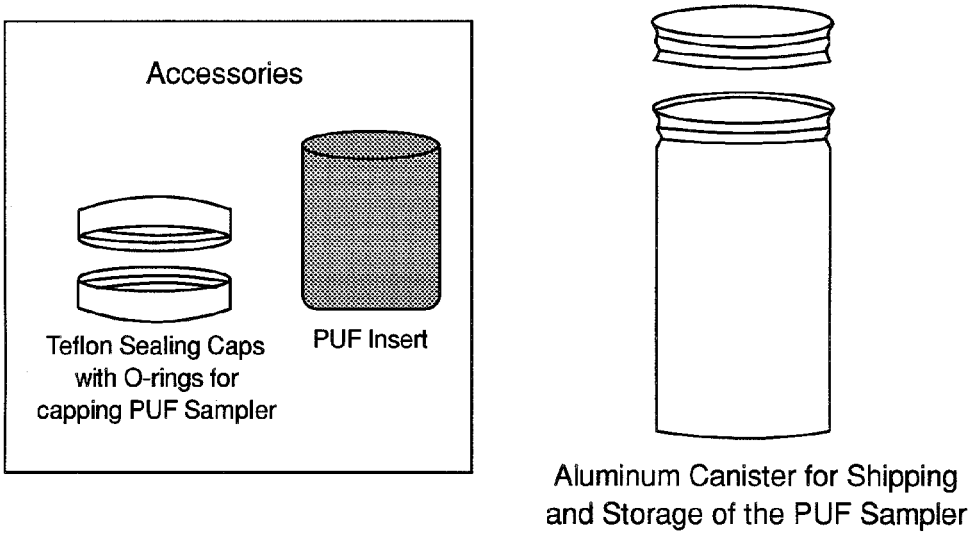


Figure 4. Apparatus used for sample clean-up and extraction.



5a. Glass PUF cartridge, plug, and end caps.



5b. PUF shipping container.

Figure 5. Glass PUF cartridge (5a) and shipping container (5b) for use with Compendium Method TO-13A.

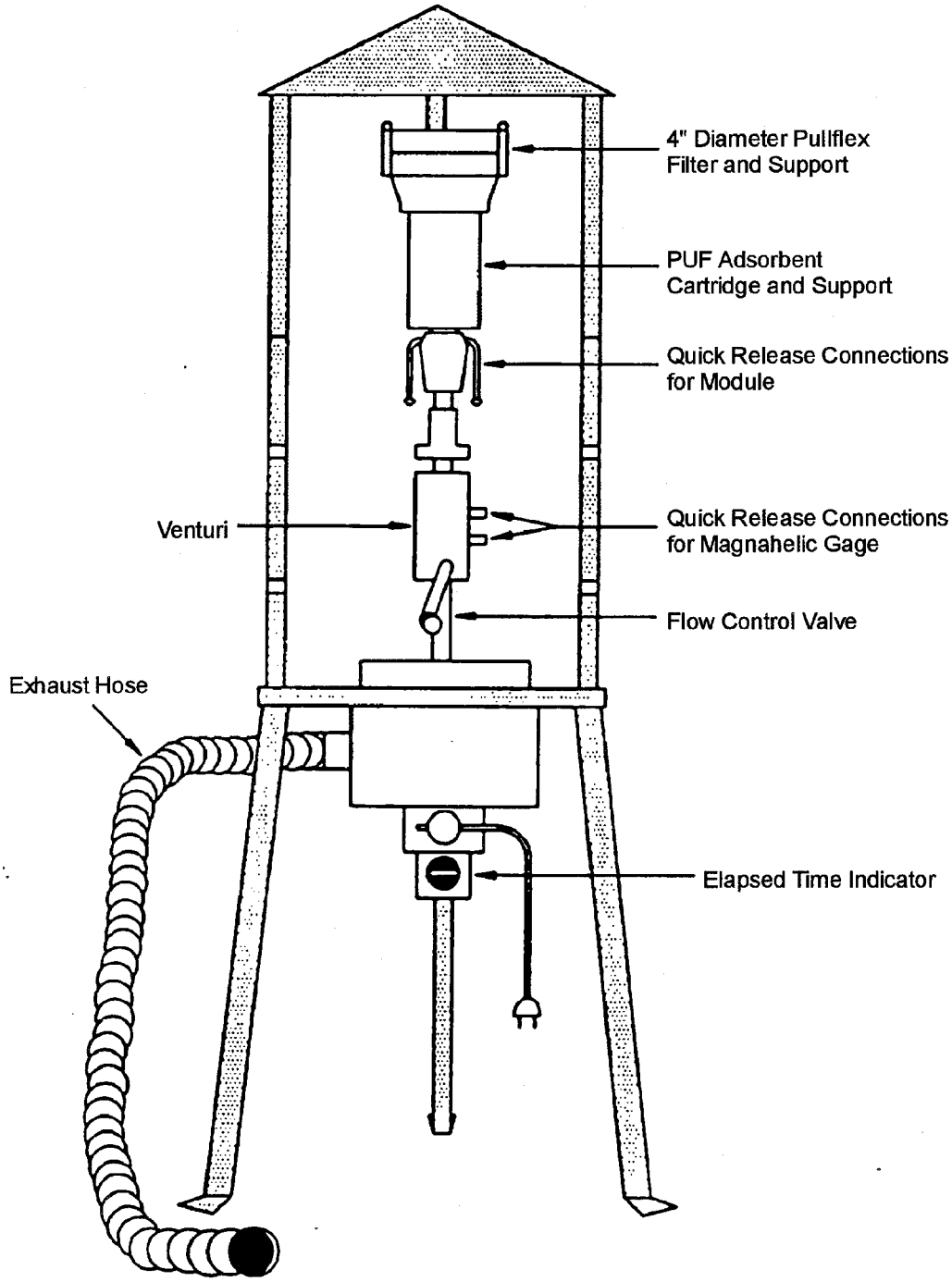


Figure 6. Example of a field portable high volume air sampler for sampling PAHs developed by EPA.

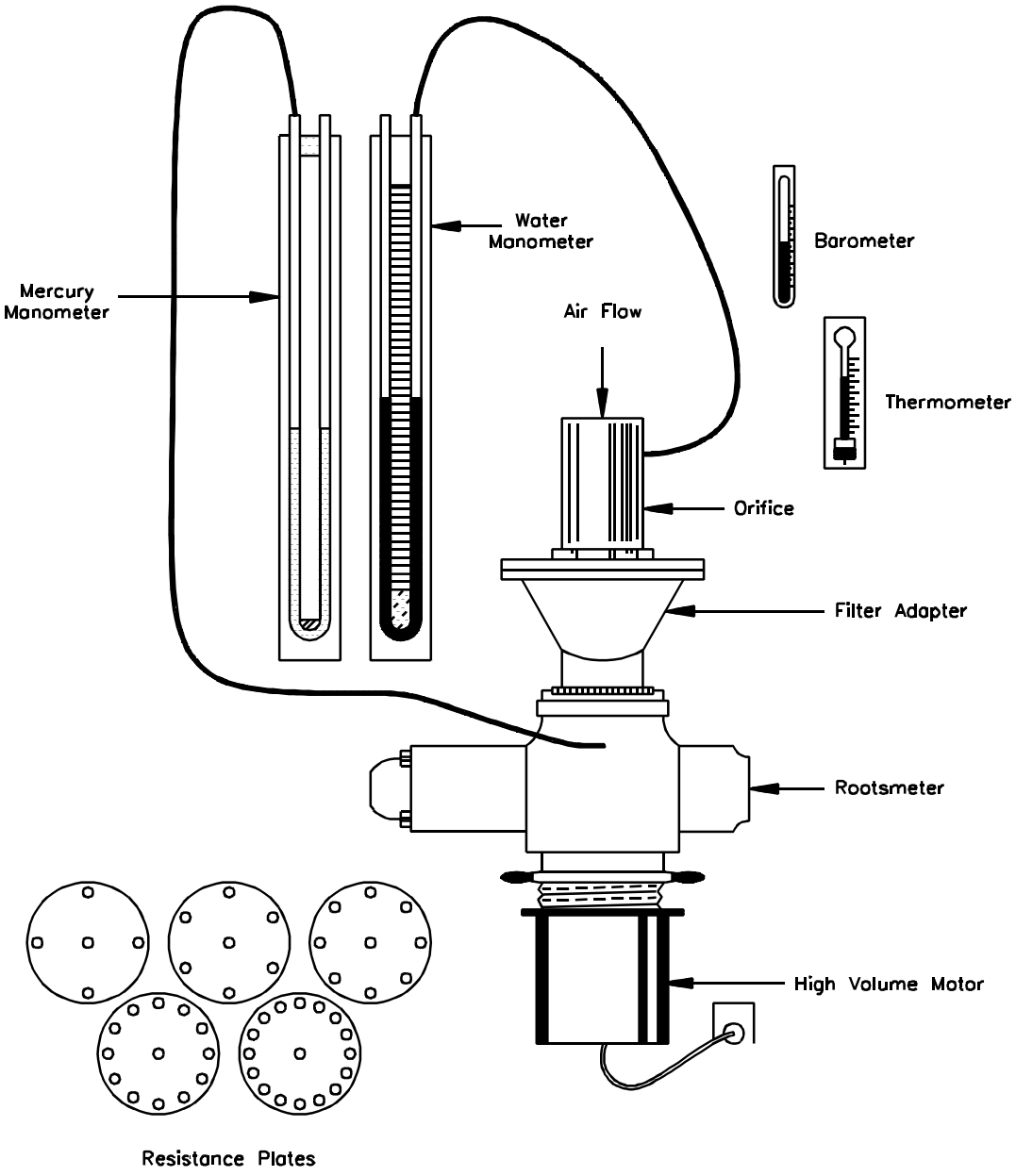


Figure 7. Positive displacement rootsmeter used to calibrate orifice transfer standard used in Compendium Method TO-13A.

COMPENDIUM METHOD TO-13A
ORIFICE CALIBRATION DATA SHEET

T_1 _____ Name _____

P_1 _____ mmHg Date _____

Orifice No. _____

Rootsmeter No. _____

Resistance Plants (No. of holes)	Air Volume Measured by Rootsmeter V_m		Standard Volume, V_{std} (std m^3)	Time for Air Volume to Pass Through Rootsmeter, θ (min)	Rootsmeter Pressure Differential, ΔP (mm Hg)	Pressure Drop Across Orifice, ΔH (in. H_2O)	x-Axis Standard Flowrate, Q_{std} (std m^3/min)	
	(R^3)	(m^3)						
5	200	5.66						
7	200	5.66						
10	300	8.50						
13	300	8.50						
18	300	8.50						

Factors: $(R^3)(0.02832\ \frac{m^3}{R^3}) = m^3$ and (in. Hg) $25.4\ (\frac{mm\ Hg}{in.\ Hg}) = mm\ Hg$

Calculation Equations:

1. $V_{std} = V_m\ (\frac{P_1 - \Delta P}{P_{std}})(\frac{T_{std}}{T_1})$

where:

$T_{std} = 296°K$

$P_{std} = 760.0$ mm Hg

2. $Q_{std} = \frac{V_{std}}{\theta}$

Figure 8. Example of a high-volume orifice calibration data sheet for Compendium Method TO-13A.

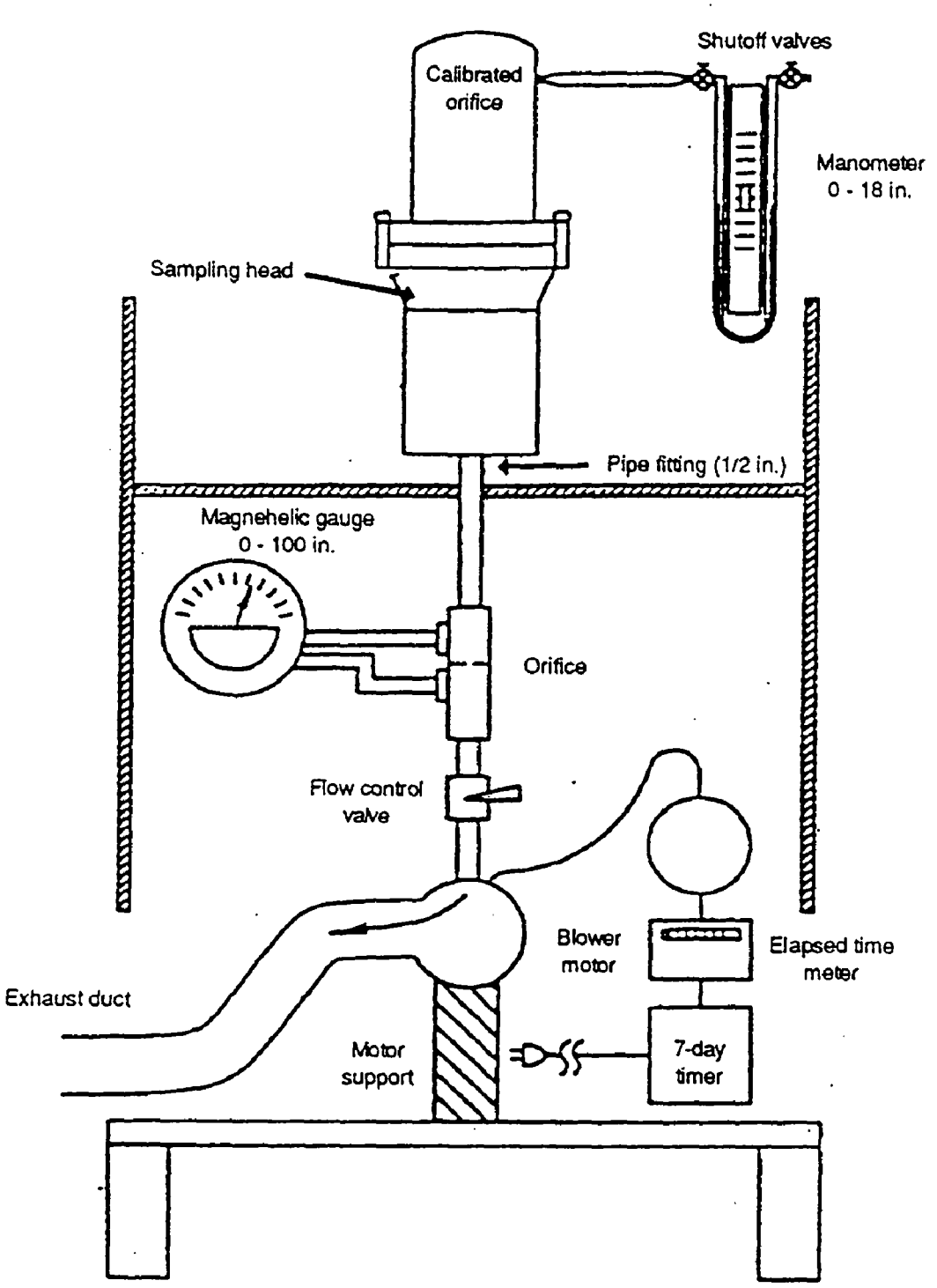


Figure 9. Typical field calibration configuration for Compendium Method TO-13A sampler.

FIELD CALIBRATION DATA SHEET FOR COMPENDIUM METHOD TO-13A PAH
SAMPLER CALIBRATION

Sampler ID: _____

Sampler Location: _____

Calibration Orifice ID: _____

Job No.: _____

High Volume Transfer Orifice Data:

Correlation Coefficient (CC1): _____ Slope (M1): _____

(CC2): _____ (M2): _____

Intercept (B1): _____

(B2): _____

Calibration Date: ___ Time: _____

Calibration Ambient Temperature: ___°F ___°C CALIBRATOR'S SIGNATURE

Calibration Ambient Barometric Pressure: ___ "Hg ___ mm Hg _____

Calibration set point (SP): _____

SAMPLER CALIBRATION

Actual values from calibration		Calibrated values		
Orifice manometer, inches (Y1)	Monitor magnehelic, inches (Y2)	Orifice manometer (Y3)	Monitor magnehelic (Y4)	Calculated value orifice flow, scm (X1)
	70			
	60			
	50			
	40			
	30			
	20			
	10			

Definitions

Y1 = Calibration orifice reading, in. H_2O

Y2 = Monitor magnehelic reading, in. H_2O

P_a = Barometric pressure actual, mm Hg

B1 = Manufacturer's Calibration orifice Intercept

M1 = Manufacturer's Calibration orifice manometer slope

Y3 = Calculated value for orifice manometer
= $\{Y1(Pa/760)[298/(Ta + 273)]\}^{½}$

Y4 = Calculated value for magnehelic
= $\{Y2(Pa/760)[298/(Ta + 273)]\}^{½}$

X1 = Calculated value orifice flow, scm
= (Y3 - B1)/M1

P_{std} = Barometric pressure standard, 760 mm Hg

T_a = Temperature actual, °C

T_{std} = Temperature standard, 25°C

Figure 10. Typical orifice transfer field calibration data sheet for Compendium Method TO-13A.

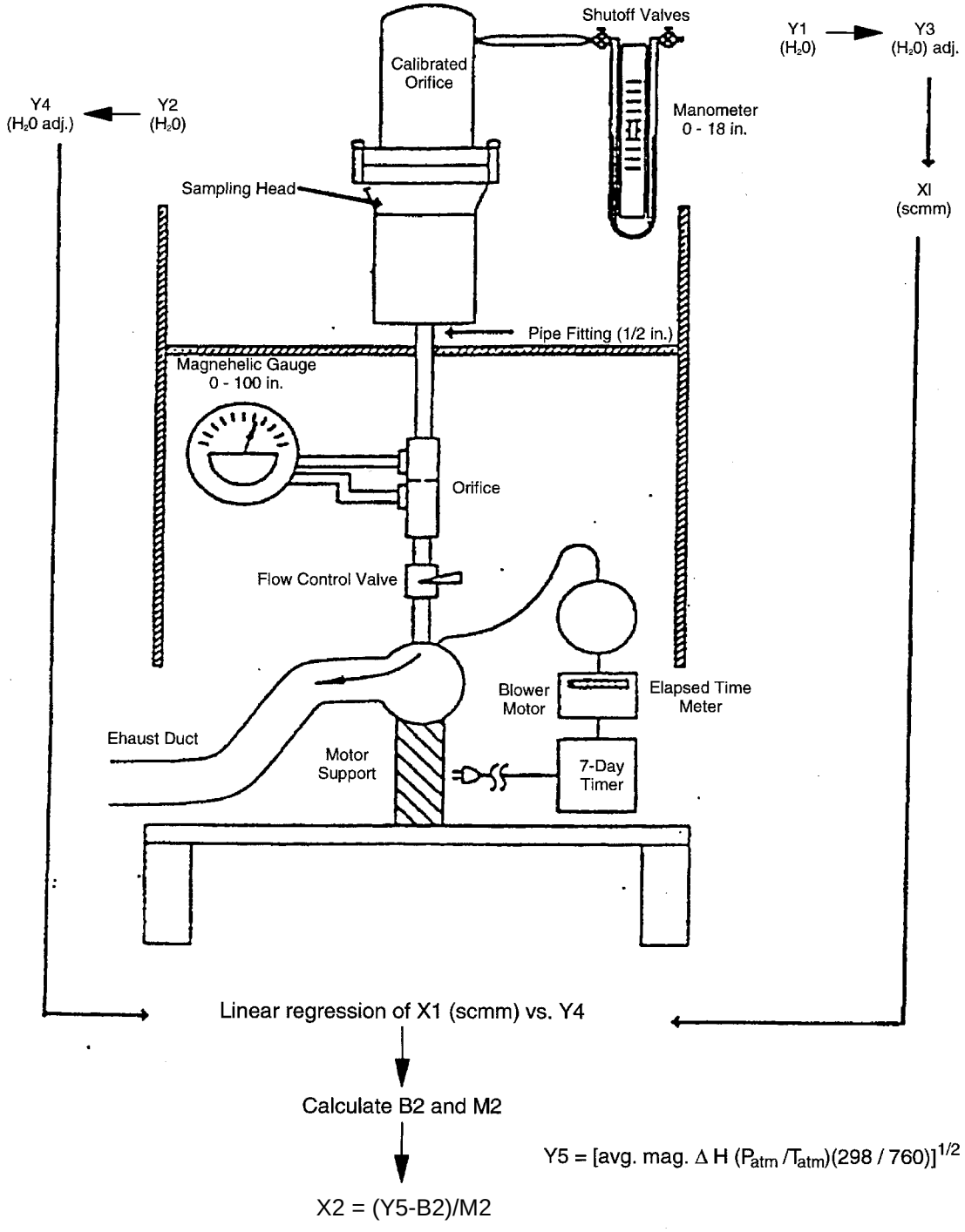


Figure 11. Example of relationship between orifice transfer standard and flow rate through Compendium Method TO-13A sampler.

COMPENDIUM METHOD TO-13A
FIELD TEST DATA SHEET
GENERAL INFORMATION

Sampler I.D. No.: _____
Lab PUF Sample No.: _____
Sample location: _____
_____
_____

Operator: _____
Other: _____
_____
_____

PUF Cartridge Certification Date: _____
Date/Time PUF Cartridge Installed: _____
Elapsed Timer: _____
Start _____
Stop _____
Diff. _____

Sampling

M1 _____ **B1** _____
M2 _____ **B2** _____

	Start	Stop
Barometric pressure ("Hg)	_____	_____
Ambient Temperature (°F)	_____	_____
Rain	**Yes** _____	**Yes** _____
	No _____	**No** _____

Sampling time
Start _____
Stop _____
Diff. _____

Audit flow check within ±10 of set point
_____ **Yes**
_____ **No**

TIME	TEMP	BAROMETRIC PRESSURE	MAGNEHELIC READING	CALCULATED FLOW RATE (std. m^3)	READ BY
Avg.					

Comments

_____
_____
_____

Figure 12. Example of typical Compendium Method TO-13A field test data sheet (FTDS).

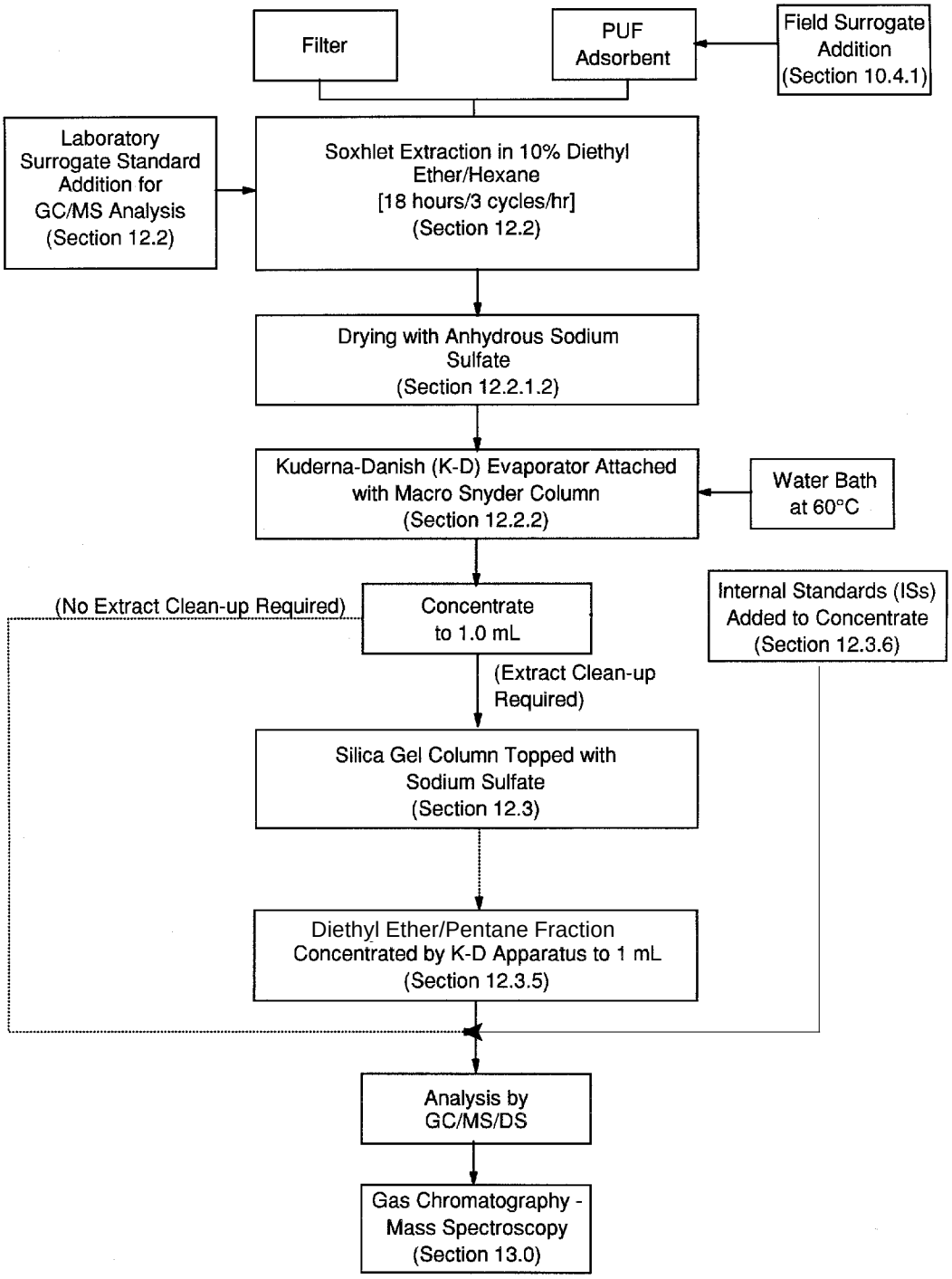


Figure 13. Sample clean-up, concentration, separation and analysis sequence for common PAHs.
[Note: XAD-2 sequence is similar to PUF except methylene chloride is the solvent.]

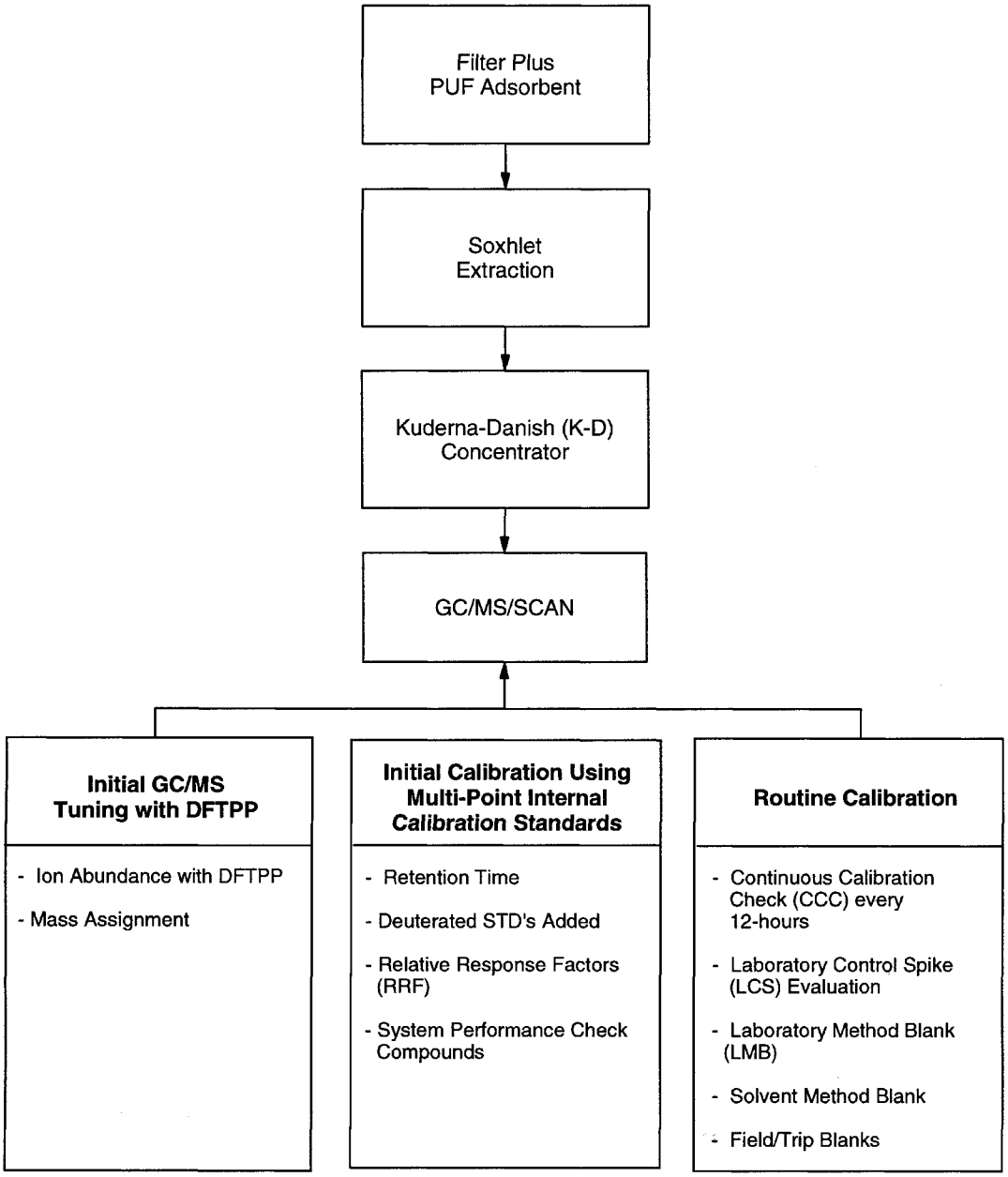


Figure 14. Typical quality assurance specifications for GC/MS/DS operation.

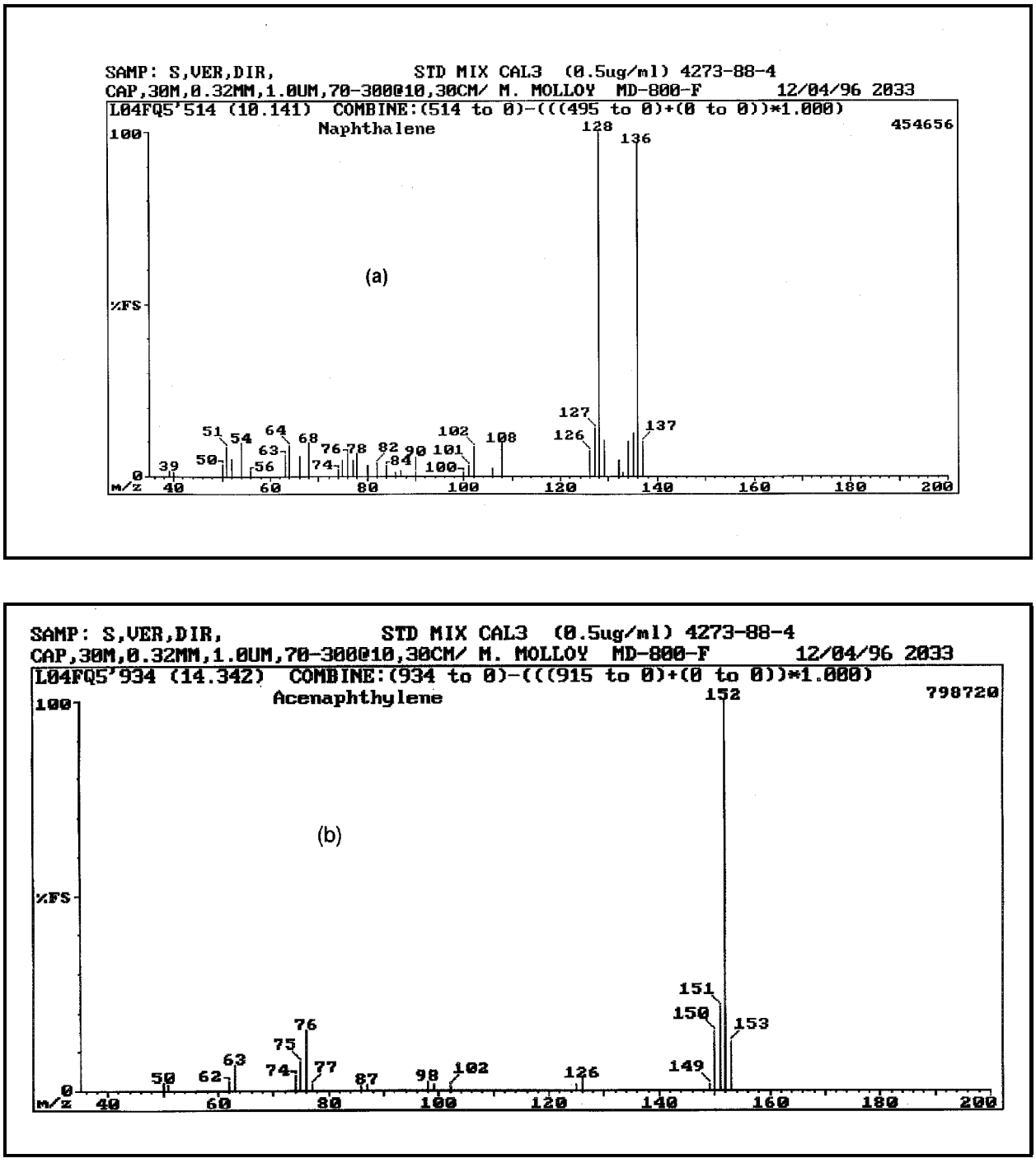
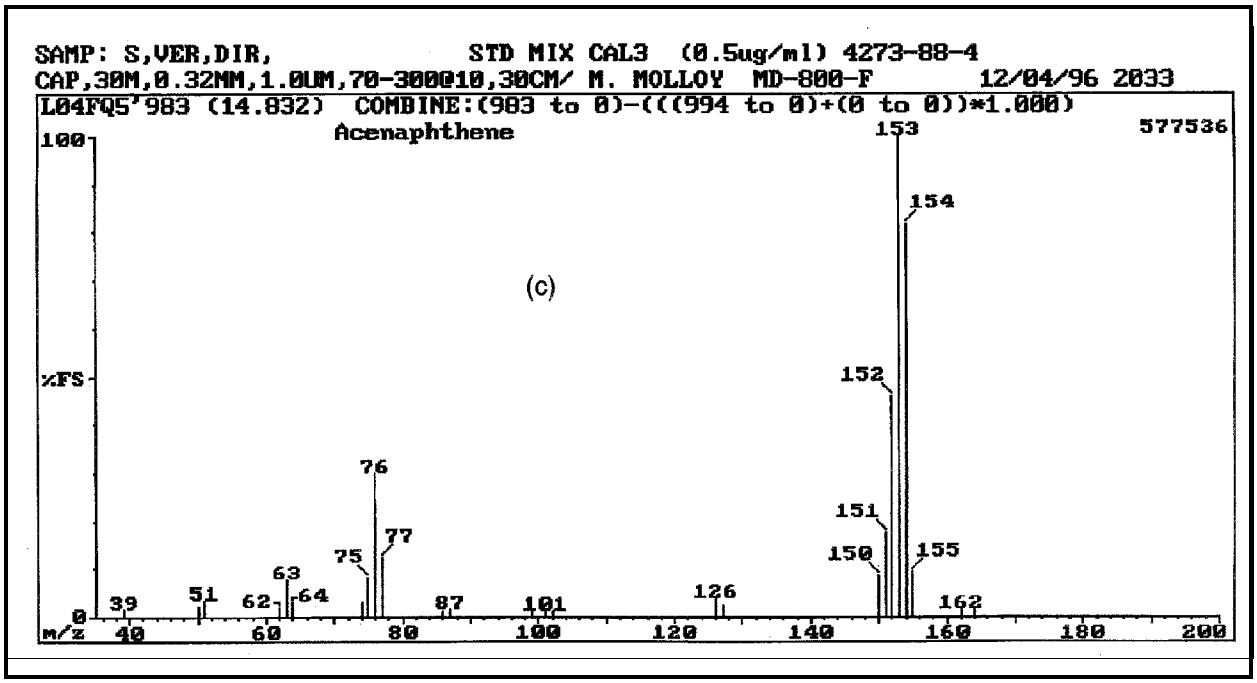


Figure 15. Mass spectra of Compendium Method TO-13A compounds for (a) naphthalene and (b) acenaphthylene.



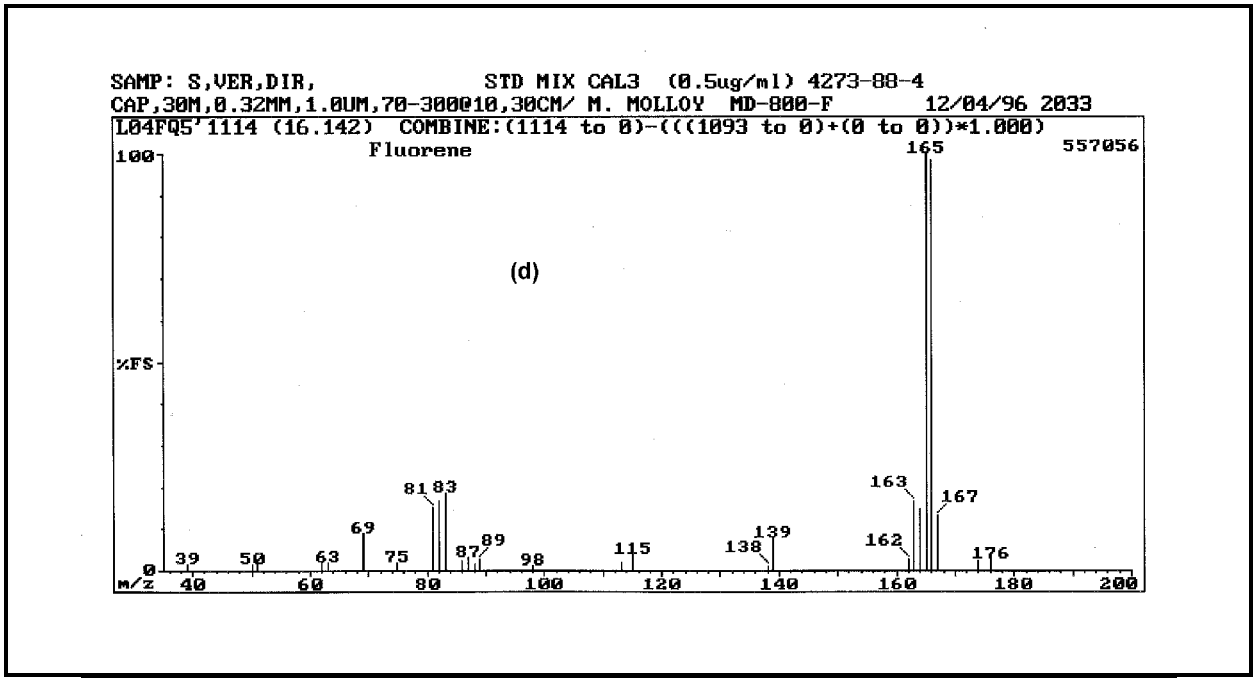
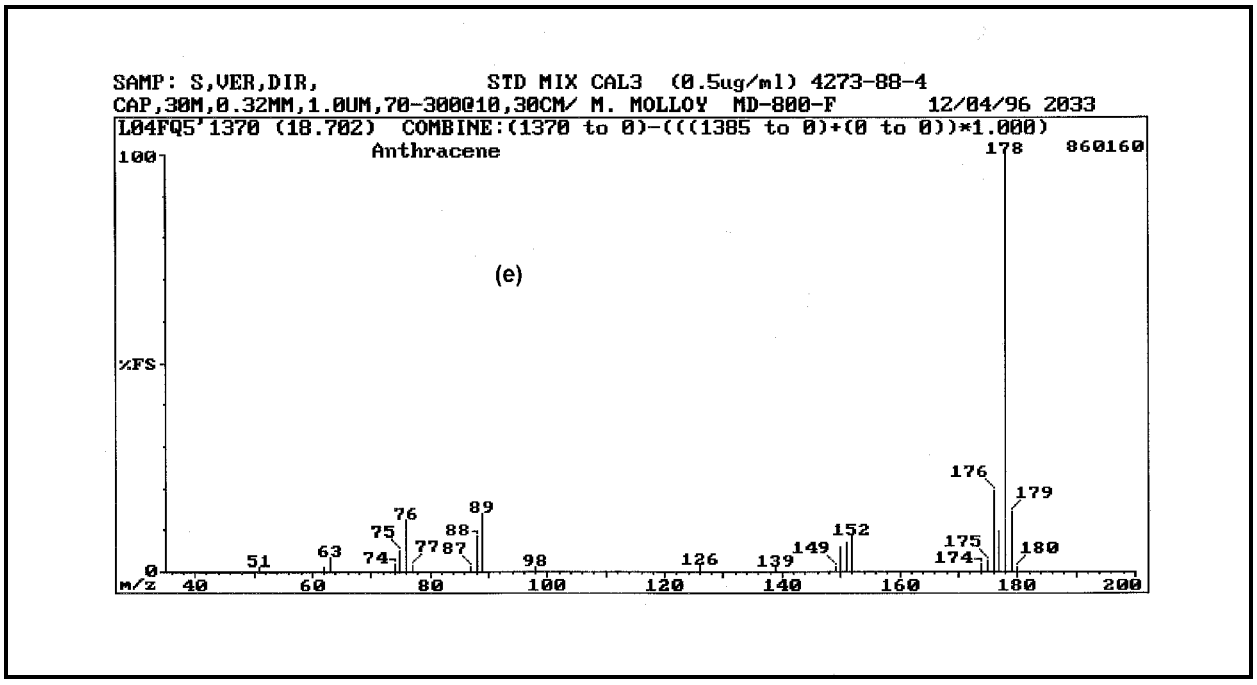


Figure 15 (Cont). Mass spectra of Compendium Method TO-13A compounds for (c) acenaphthene and (d) fluorene.



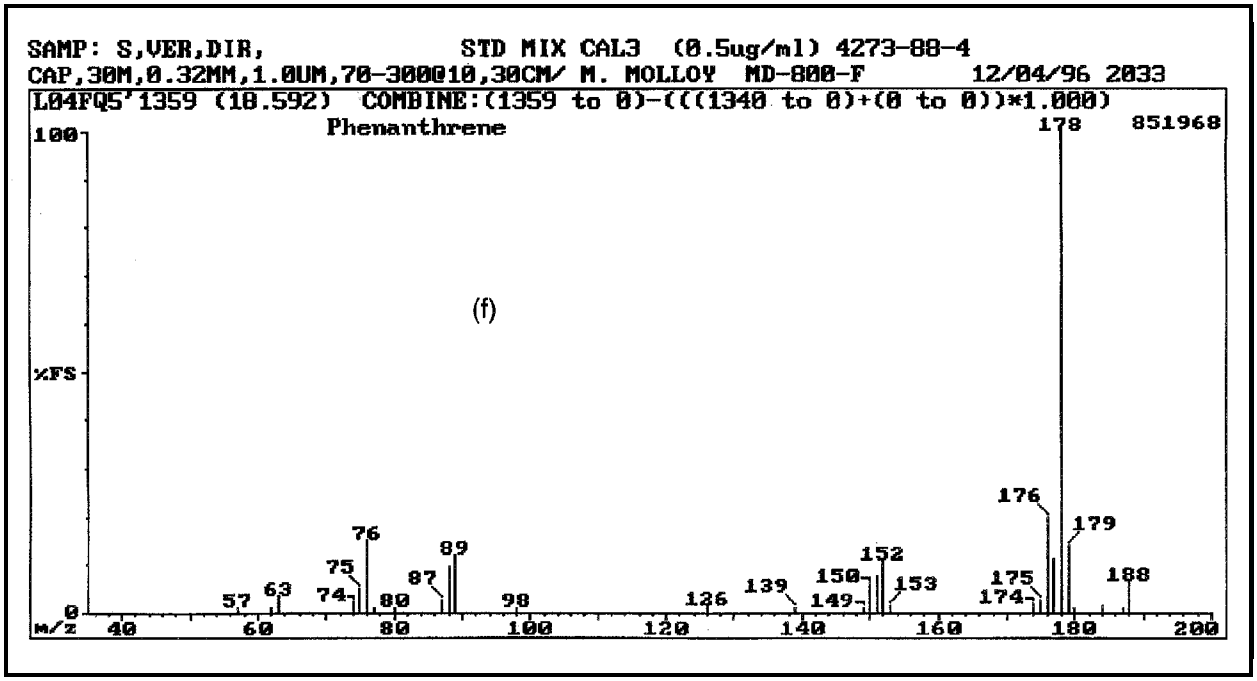
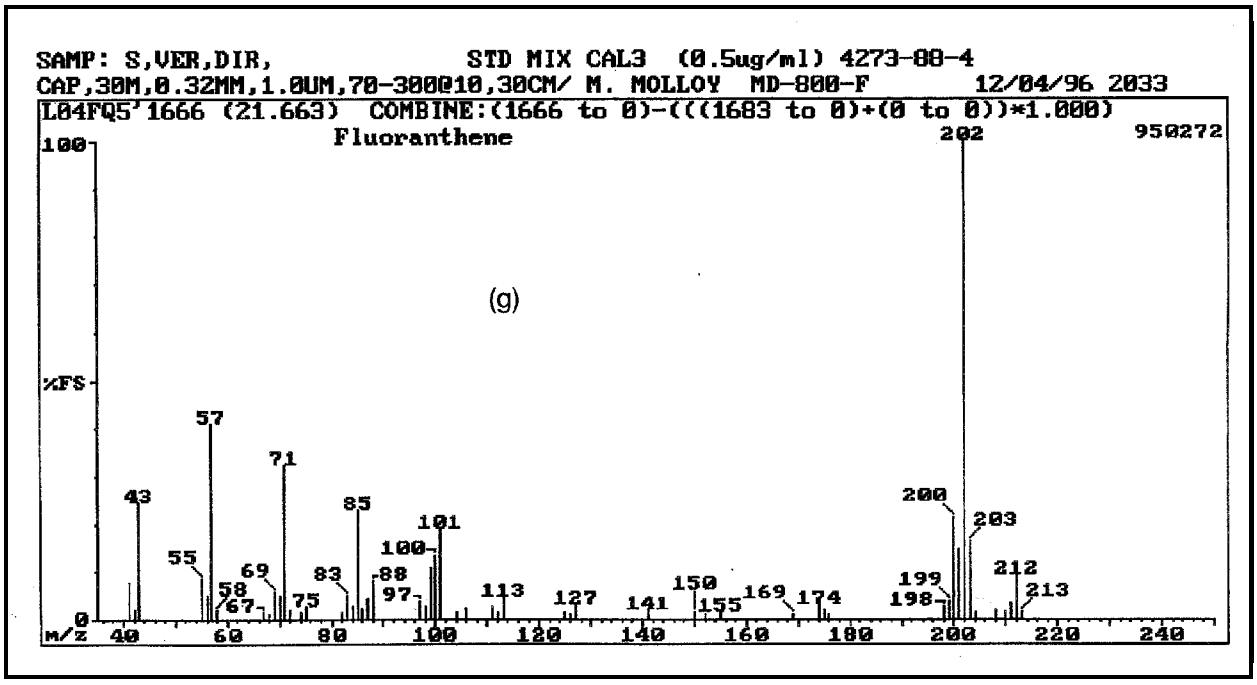


Figure 15 (Cont). Mass spectra of Compendium Method TO-13A compounds for (e) anthracene and (f) phenanthrene.



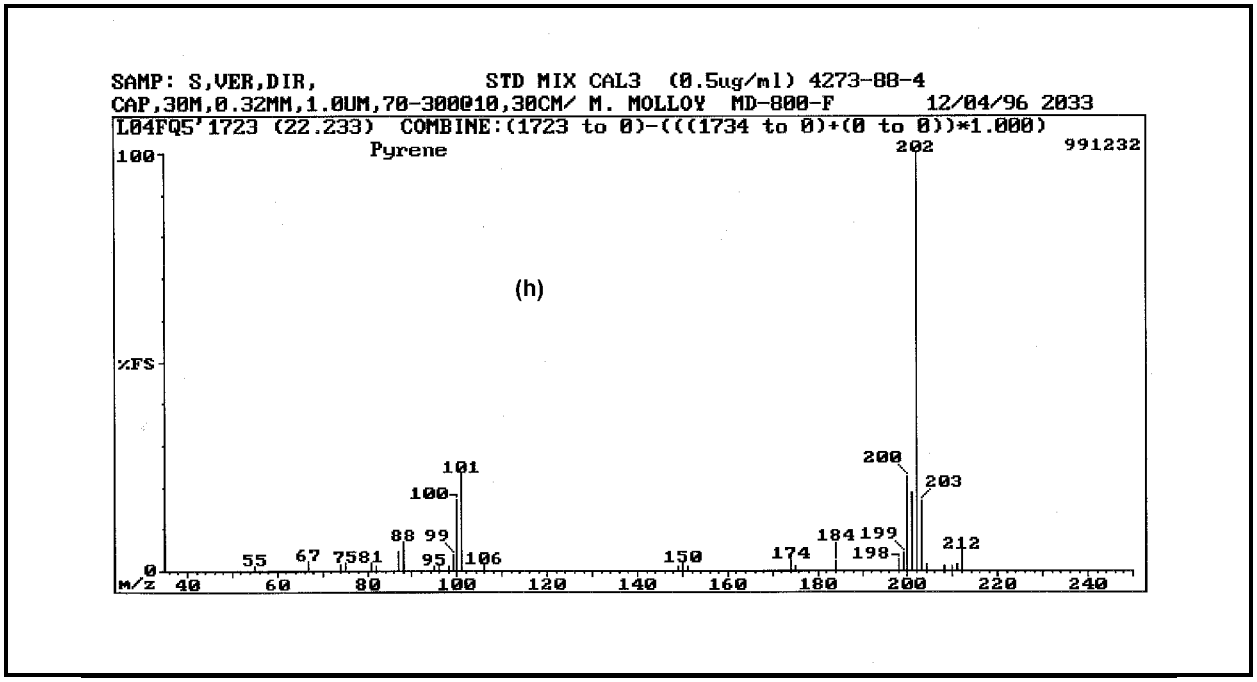


Figure 15 (Cont). Mass spectra of Compendium Method TO-13A compounds for (g) fluoranthene and (h) pyrene.

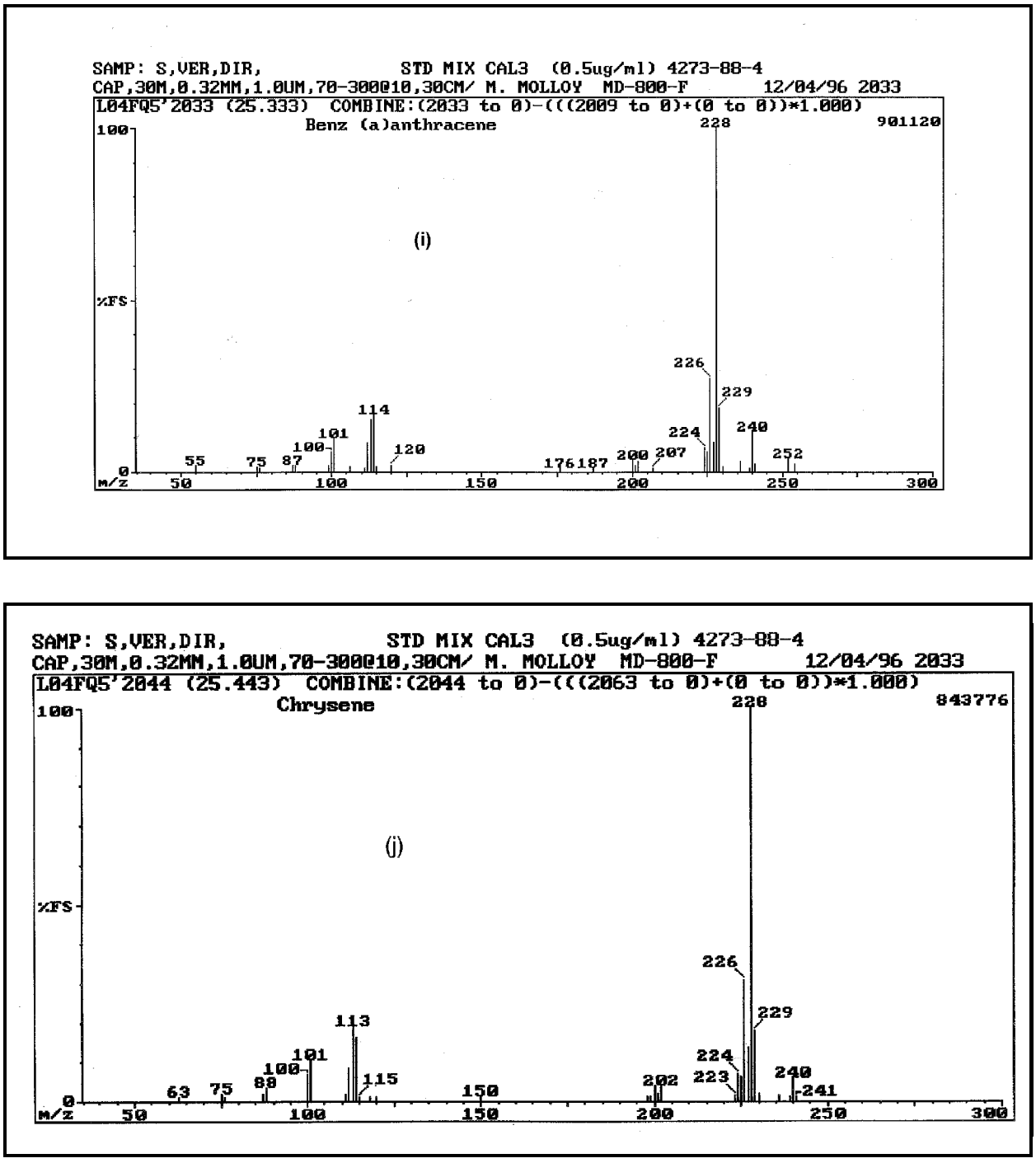
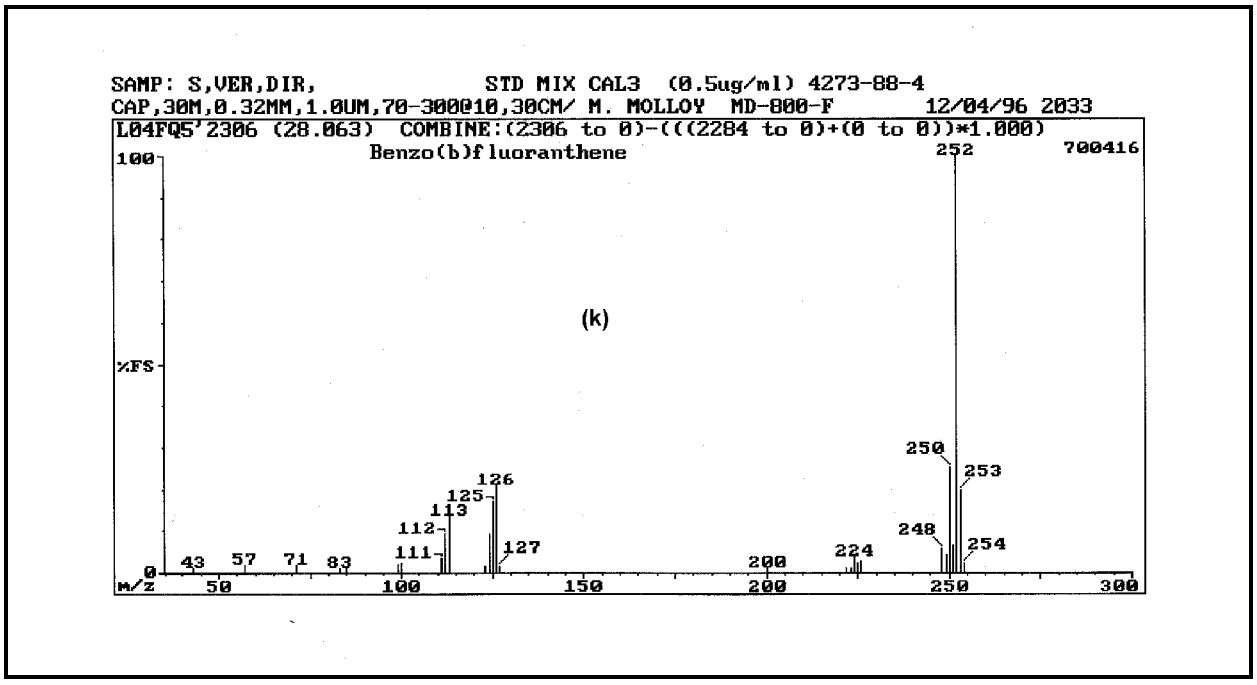


Figure 15 (Cont). Mass spectra of Compendium Method TO-13A compounds for (i) benz(a)anthracene and (j) chrysene.



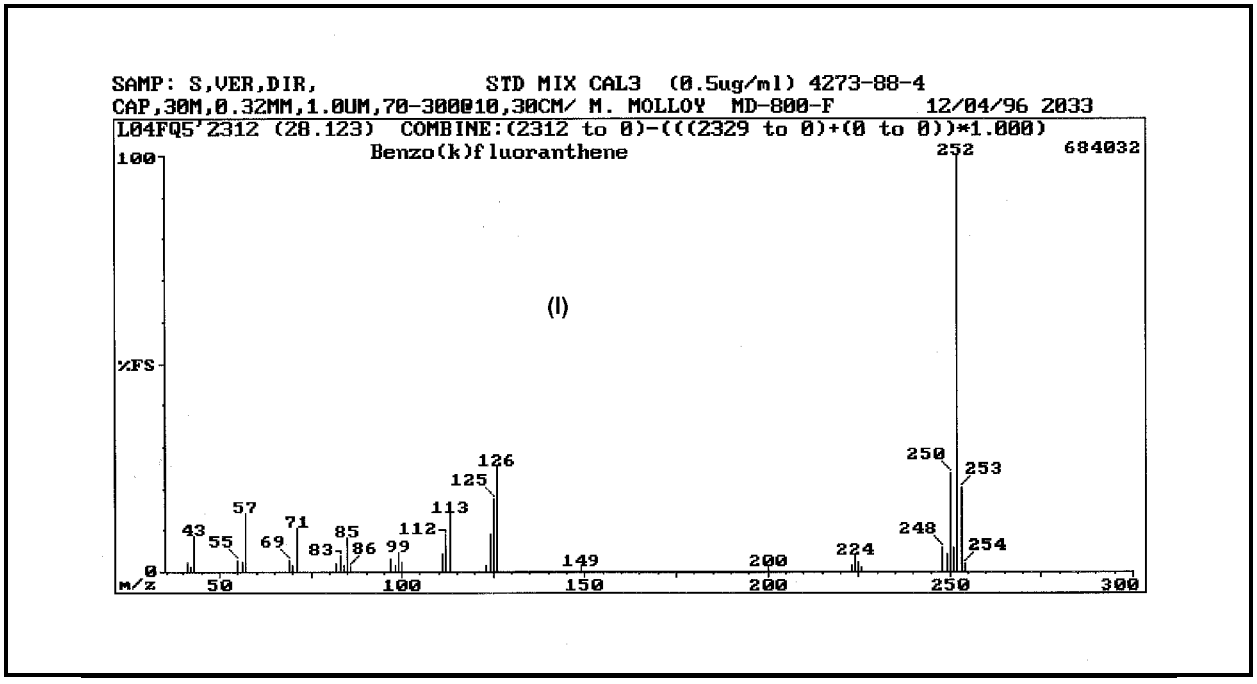
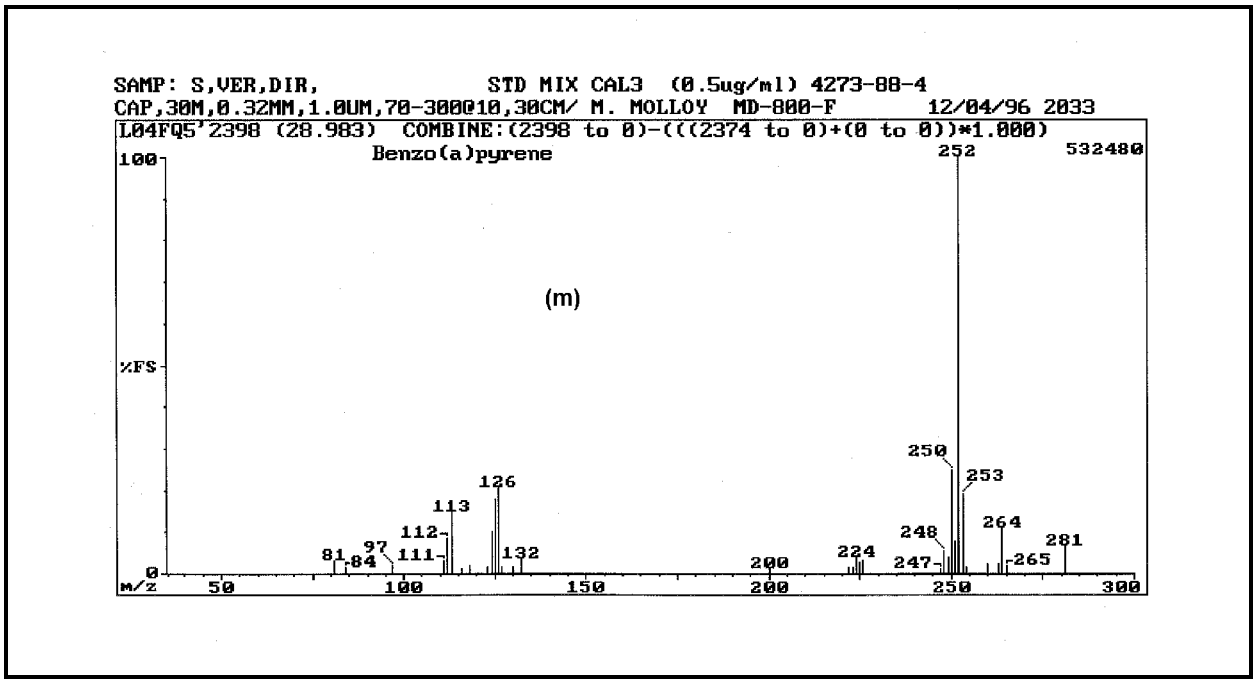


Figure 15 (Cont). Mass spectra of Compendium Method TO-13A compounds for (k) benzo(b)fluoranthene and (l) benzo(k)fluoranthene.



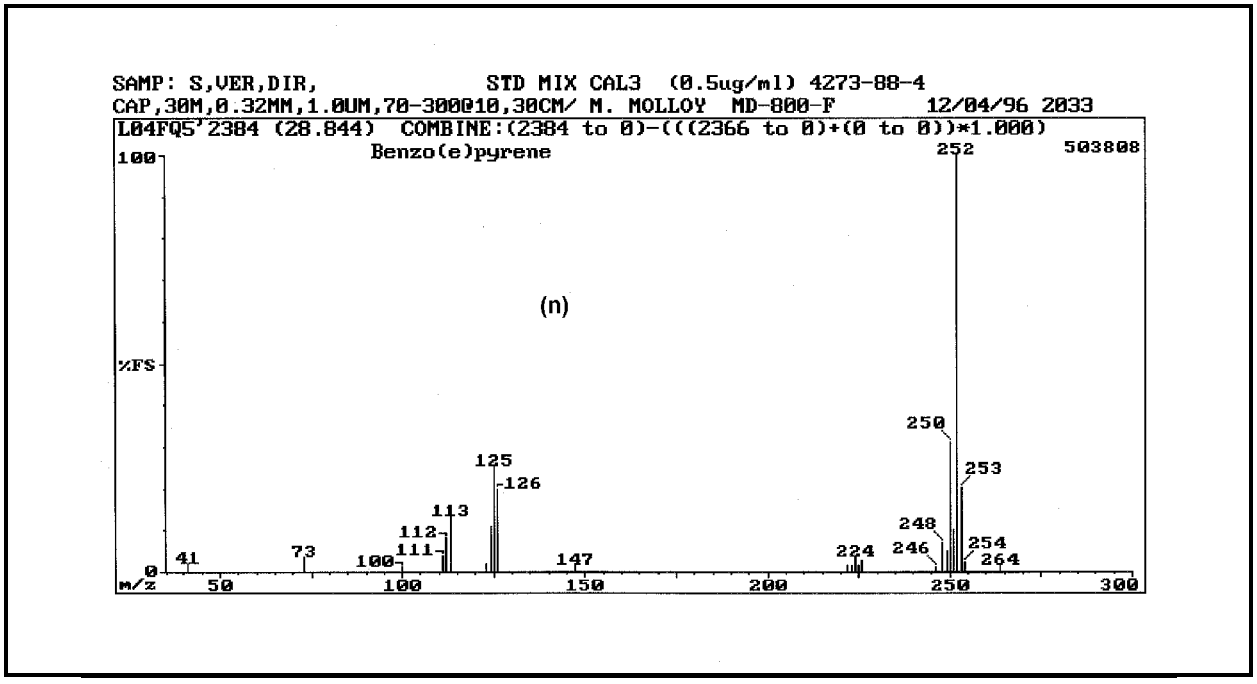
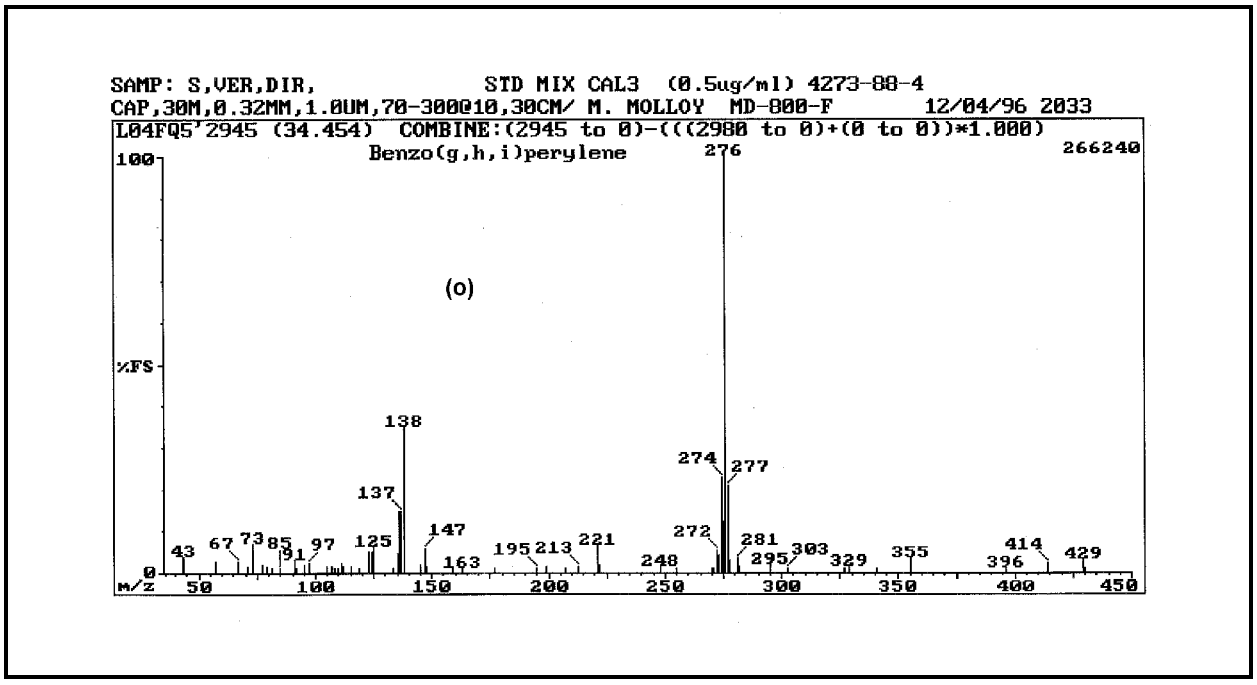


Figure 15 (Cont). Mass spectra of Compendium Method TO-13A compounds for (m) benzo(a)pyrene and (n) benzo(e)pyrene.



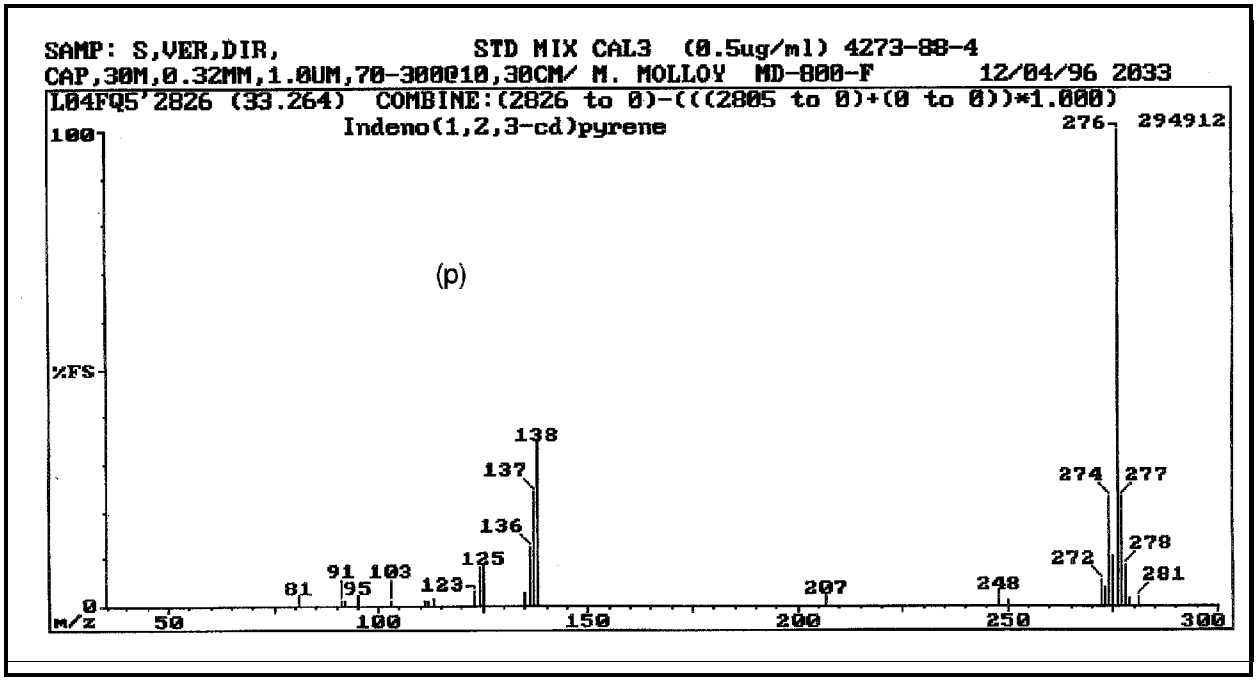


Figure 15 (Cont). Mass spectra of Compendium Method TO-13A compounds for (o) benzo(g,h,i)perylene and (p) indeno(1,2,3-cd)pyrene.

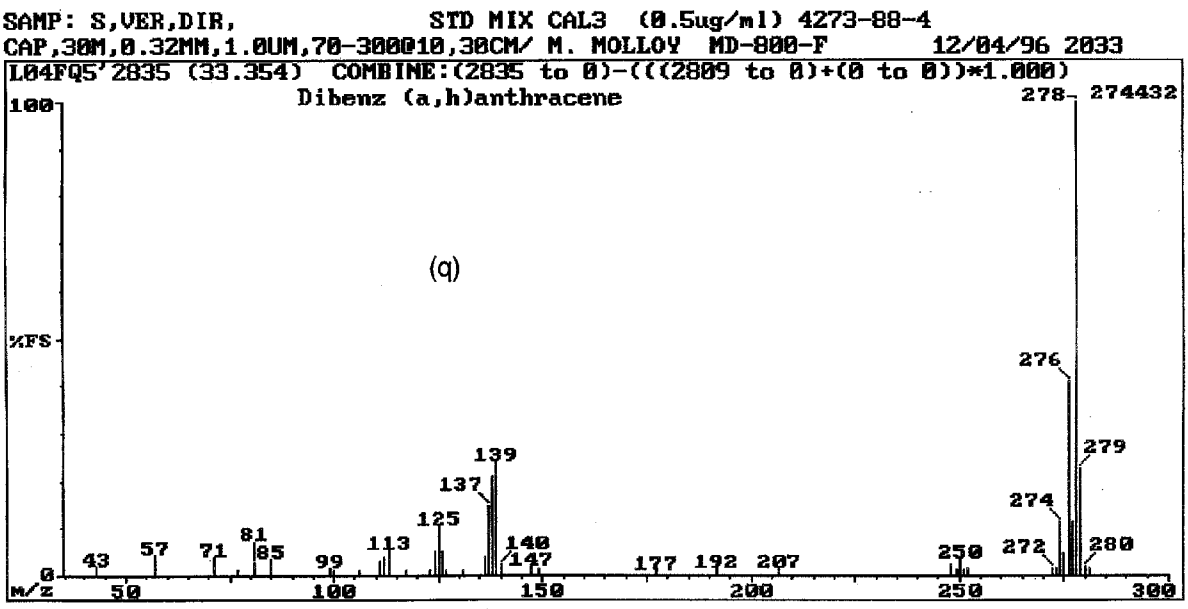


Figure 15 (Cont). Mass spectra of Compendium Method TO-13A compounds for (q) dibenz(a,h)anthracene.

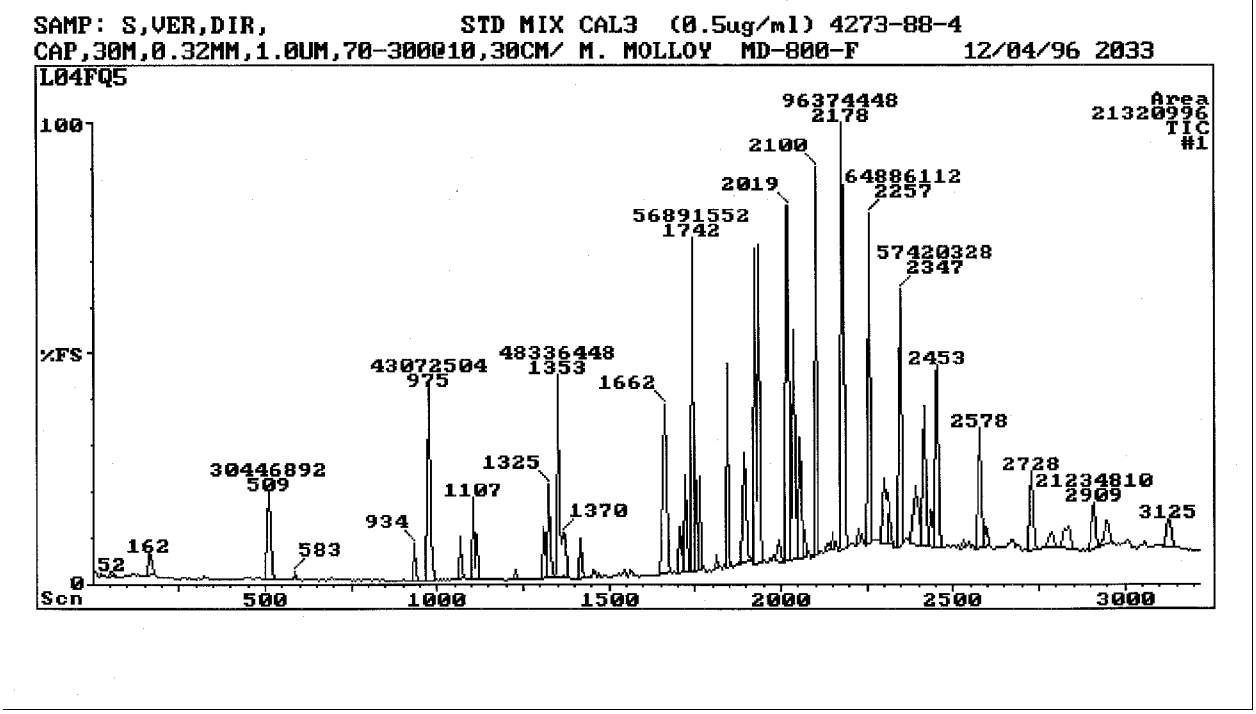


Figure 16. Total ion chromatogram (TIC) of Compendium Method TO-13A target PAHs.

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PARTICULATES NOT OTHERWISE REGULATED, TOTAL 0500

DEFINITION: total aerosol mass CAS: NONE RTECS: NONE

METHOD: 0500, Issue 2 **EVALUATION: FULL** **Issue 1: 15 February 1984**
Issue 2: 15 August 1994

OSHA: 15 mg/m^3
NIOSH: no REL
ACGIH: 10 mg/m^3, total dust less than 1% quartz

PROPERTIES: contains no asbestos and quartz less than 1%

SYNONYMS: nuisance dusts; particulates not otherwise classified

SAMPLING

SAMPLER: FILTER (tared 37-mm, 5-µm PVC filter)

FLOW RATE: 1 to 2 L/min

VOL-MIN: 7 L @ 15 mg/m^3
-MAX: 133 L @ 15 mg/m^3

SHIPMENT: routine

SAMPLE STABILITY: indefinitely

BLANKS: 2 to 10 field blanks per set

BULK SAMPLE: none required

ACCURACY

RANGE STUDIED: 8 to 28 mg/m^3

BIAS: 0.01%

OVERALL PRECISION ($\hat{S}_{rt}$): 0.056 [1]

ACCURACY: ±11.04%

MEASUREMENT

TECHNIQUE: GRAVIMETRIC (FILTER WEIGHT)

ANALYTE: airborne particulate material

BALANCE: 0.001 mg sensitivity; use same balance before and after sample collection

CALIBRATION: National Institute of Standards and Technology Class S-1.1 weights or ASTM Class 1 weights

RANGE: 0.1 to 2 mg per sample

ESTIMATED LOD: 0.03 mg per sample

PRECISION ($\bar{S}_r$): 0.026 [2]

APPLICABILITY: The working range is 1 to 20 mg/m^3 for a 100-L air sample. This method is nonspecific and determines the total dust concentration to which a worker is exposed. It may be applied, e.g., to gravimetric determination of fibrous glass [3] in addition to the other ACGIH particulates not otherwise regulated [4].

INTERFERENCES: Organic and volatile particulate matter may be removed by dry ashing [3].

OTHER METHODS: This method is similar to the criteria document method for fibrous glass [3] and Method 5000 for carbon black. This method replaces Method S349 [5]. Impingers and direct-reading instruments may be used to collect total dust samples, but these have limitations for personal sampling.

EQUIPMENT:

1. Sampler: 37-mm PVC, 2- to 5-µm pore size membrane or equivalent hydrophobic filter and supporting pad in 37-mm cassette filter holder.
2. Personal sampling pump, 1 to 2 L/min, with flexible connecting tubing.
3. Microbalance, capable of weighing to 0.001 mg.
4. Static neutralizer: e.g., Po-210; replace nine months after the production date.
5. Forceps (preferably nylon).
6. Environmental chamber or room for balance (e.g., 20 °C ± 1 °C and 50% ± 5% RH).

SPECIAL PRECAUTIONS: None.

PREPARATION OF FILTERS BEFORE SAMPLING:

1. Equilibrate the filters in an environmentally controlled weighing area or chamber for at least 2 h.
   NOTE: An environmentally controlled chamber is desirable, but not required.
2. Number the backup pads with a ballpoint pen and place them, numbered side down, in filter cassette bottom sections.
3. Weigh the filters in an environmentally controlled area or chamber. Record the filter tare weight, W_1 (mg).
   a. Zero the balance before each weighing.
   b. Handle the filter with forceps. Pass the filter over an antistatic radiation source. Repeat this step if filter does not release easily from the forceps or if filter attracts balance pan. Static electricity can cause erroneous weight readings.
4. Assemble the filter in the filter cassettes and close firmly so that leakage around the filter will not occur. Place a plug in each opening of the filter cassette. Place a cellulose shrink band around the filter cassette, allow to dry and mark with the same number as the backup pad.

SAMPLING:

5. Calibrate each personal sampling pump with a representative sampler in line.
6. Sample at 1 to 2 L/min for a total sample volume of 7 to 133 L. Do not exceed a total filter loading of approximately 2 mg total dust. Take two to four replicate samples for each batch of field samples for quality assurance on the sampling procedure.

SAMPLE PREPARATION:

7. Wipe dust from the external surface of the filter cassette with a moist paper towel to minimize contamination. Discard the paper towel.
8. Remove the top and bottom plugs from the filter cassette. Equilibrate for at least 2 h in the balance room.
9. Remove the cassette band, pry open the cassette, and remove the filter gently to avoid loss of dust.
   NOTE: If the filter adheres to the underside of the cassette top, very gently lift away by using the dull side of a scalpel blade. This must be done carefully or the filter will tear.

CALIBRATION AND QUALITY CONTROL:

10. Zero the microbalance before all weighings. Use the same microbalance for weighing filters before and after sample collection. Maintain and calibrate the balance with National Institute of Standards and Technology Class S-1.1 or ASTM Class 1 weights.
11. The set of replicate samples should be exposed to the same dust environment, either in a laboratory dust chamber [7] or in the field [8]. The quality control samples must be taken with the same

equipment, procedures, and personnel used in the routine field samples. The relative standard deviation calculated from these replicates should be recorded on control charts and action taken when the precision is out of control [7].

MEASUREMENT:

12. Weigh each filter, including field blanks. Record the post-sampling weight, W_2 (mg). Record anything remarkable about a filter (e.g., overload, leakage, wet, torn, etc.)

CALCULATIONS:

13. Calculate the concentration of total particulate, C (mg/m^3), in the air volume sampled, V (L):

$$C = \frac{(W_2 - W_1) - (B_2 - B_1)}{V} \times 10^3, \text{ mg/m}^3,$$

where: W_1 = tare weight of filter before sampling (mg),
W_2 = post-sampling weight of sample-containing filter (mg),
B_1 = mean tare weight of blank filters (mg),
B_2 = mean post-sampling weight of blank filters (mg).

EVALUATION OF METHOD:

Lab testing with blank filters and generated atmospheres of carbon black was done at 8 to 28 mg/m^3 [2,6]. Precision and accuracy data are given on page 0500-1.

REFERENCES:

[1] NIOSH Manual of Analytical Methods, 3rd ed., NMAM 5000, DHHS (NIOSH) Publication No. 84-100 (1984).
[2] Unpublished data from Non-textile Cotton Study, NIOSH/DRDS/EIB.
[3] NIOSH Criteria for a Recommended Standard … Occupational Exposure to Fibrous Glass, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-152, 119–142 (1977).
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[5] NIOSH Manual of Analytical Methods, 2nd ed., V. 3, S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
[6] Documentation of the NIOSH Validation Tests, S262 and S349, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
[7] Bowman, J.D., D.L. Bartley, G.M. Breuer, L.J. Doemeny, and D.J. Murdock. Accuracy Criteria Recommended for the Certification of Gravimetric Coal Mine Dust Personal Samplers. NTIS Pub. No. PB 85-222446 (1984).
[8] Breslin, J.A., S.J. Page, and R.A. Jankowski. Precision of Personal Sampling of Respirable Dust in Coal Mines, U.S. Bureau of Mines Report of Investigations #8740 (1983).

METHOD REVISED BY:

Jerry Clere and Frank Hearl, P.E., NIOSH/DRDS.

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Description | Additional information

Description

General

ACS Vapor Monitor can be worn as a badge near the breathing zone to measure personal exposure, or placed in a room to measure area concentration.

Workplace Exposure Monitoring

Measurement of personal exposure is very easy. Clip the monitoring badge near the breathing zone of the worker. Record the start time and the stop time of the test on the pouch of the badge and return it to the laboratory for analysis in the mailer provided. The analysis report will show the average concentration of the worker's personal exposure level.

According to OSHA regulations, each person who uses a toxic gas as a regular part of their job must have their exposure monitored periodically.

Indoor Air Monitoring

Place or hang the badge in your home for 24 hours. Record the start and stop times and send the badge back to the laboratory for analysis in the mailer provided. The analysis report explains if the concentration level of your home is exceeding the exposure limit.

Principle of Operation

Air diffuses through a micro-porous membrane and collects vapor on special prepared adsorbents inside the monitoring badge.

	OSHA	NIOSH	ACGIH
TWA (PPM)	1	0.1	0.5

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STEL/CEIL (PPM)	5	1	2.5

AKA	
CAS#	71-43-2
ANALYTICAL METHOD	OSHA 1005, NIOSH 1501
ABSORBENT	Activated Carbon
MINIMUM LEVEL OF QUANTITATION	0.05 PPM for 8 Hours 1.6 PPM for 15 Minutes 0.02 PPM for 24 Hours
RECOMMENDED SAMPLING TIME	Workplace: 15 minutes to 8 hours Indoor Air: 24 hours – 48 hours
INTERFERENCES	No interferences are known
EFFECT OF TEMPERATURE	Less than 10% for each 10°C variation from 24°C
EFFECT OF HUMIDITY	No effects detected at 30% RH to 92% RH.
ACCURACY	Meets or exceeds OSHA accuracy requirements of +/- 25%
STORAGE CONDITIONS	Store at room temperature.
RECOMMENDED HOLDING TIME	Monitors need to be returned to Lab within two weeks after sampling.

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Naphthalene Vapor Monitor (OV-775)

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Naphthalene Vapor Monitor (OV-775)

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Description | Additional information

Description

General

ACS Vapor Monitor can be worn as a badge near the breathing zone to measure personal exposure, or placed in a room to measure area concentration.

Workplace Exposure Monitoring

Measurement of personal exposure is very easy. Clip the monitoring badge near the breathing zone of the worker. Record the start time and the stop time of the test on the pouch of the badge and return it to the laboratory for analysis in the mailer provided. The analysis report will show the average concentration of the worker's personal exposure level.

According to OSHA regulations, each person who uses a toxic gas as a regular part of their job must have their exposure monitored periodically.

Indoor Air Monitoring

Place or hang the badge in your home for 24 hours. Record the start and stop times and send the badge back to the laboratory for analysis in the mailer provided. The analysis report explains if the concentration level of your home is exceeding the exposure limit.

Principle of Operation

Air diffuses through a micro-porous membrane and collects vapor on special prepared adsorbents inside the monitoring badge.

	OSHA	NIOSH	ACGIH
TWA (PPM)	10	10	10

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Full Scan Organic (OV-00F) (https://acsbadge.com/product/full-scan-organic-vapor-monitor-ov-00f/)

STEL/CEIL (PPM)	15

AKA	Naphthalene
CAS#	91-20-3
ANALYTICAL METHOD	NIOSH S292 [4], NIOSH 1501
ABSORBENT	Activated Carbon
MINIMUM LEVEL OF QUANTITATION	0.05 PPM for 8 Hours 1.6 PPM for 15 Minutes 0.02 PPM for 24 Hours
RECOMMENDED SAMPLING TIME	Workplace: 15 minutes to 8 hours Indoor Air: 24 hours – 48 hours
INTERFERENCES	No interferences are known
EFFECT OF TEMPERATURE	Less than 10% for each 10°C variation from 24°C
EFFECT OF HUMIDITY	No effects detected at 30% RH to 92% RH.
ACCURACY	Meets or exceeds OSHA accuracy requirements of +/- 25%
STORAGE CONDITIONS	Store at room temperature.
RECOMMENDED HOLDING TIME	Monitors need to be returned to Lab within two weeks after sampling.

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Appendix D

Safety Data Sheets for
Contractor Materials

Dust Control 96% Calcium Chloride
Safety Data Sheet

SECTION 1: PRODUCT AND COMPANY IDENTIFICATION

Product Name: Dust Control 96% Calcium Chloride

Product Code(s): B-DCPXXX

Recommended Use: For industrial use. Agricultural. Dust Control. Brine treatment.

Company: Blendmagic Products, LLC
PO Box 220, East Islip, NY 11730
www.blendmagicproducts.com
T. 888-605-4332

Emergency Number: Chemtrec (800) 424-9300

Restriction on Use: Not Food Grade. Not for Ingestion

SECTION 2: HAZARDS IDENTIFICATION

Classification
Acute toxicity - Oral Category 4
Skin corrosion/irritation Category 2
Serious eye damage/eye irritation Category 2B

Label Elements None

Signal Word (GHS-US) Warning

Hazard Statements (GHS-US)
Harmful if swallowed
Causes skin irritation
Causes eye irritation

Precautionary Statements
Prevention – Wear protective gloves/protective clothing / eye protection / face protection.
Response – IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. If eye irritation persists: Get medical advice/attention.
IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/ shower.
IF SWALLOWED: Call a POISON CENTER or doctor if you feel unwell. Rinse mouth
Disposal – Dispose of contents/container to an approved waste disposal plant.

Other Hazards None Identified.

SECTION 3: COMPOSITION/INFORMATION ON INGREDIENTS

Component	CAS No.	Amount
Calcium Chloride	10043-52-4	94-100%

These components contain no substances or impurities which influence the classification of this product

.

SECTION 4: FIRST-AID MEASURES

General	If medical advice is needed, have product container or label at hand for the doctor in attendance.
Inhalation	If inhaled, remove to fresh air and keep at rest in a position comfortable for breathing. Give oxygen or artificial respiration if necessary. Obtain medical attention if breathing difficulty persists.
Skin Contact	Wash skin thoroughly with mild soap and water. Obtain medical attention if irritation develops or persists.
Eye Contact	Immediately rinse with water for a prolonged period (15 minutes) while holding the eyelids wide open including upper and lower lids. Remove contact lenses, if present and easy to do. Continue rinsing. Keep eye wide open while rinsing. Do not rub affected area. Get medical attention if irritation develops and persists.
Ingestion	Rinse mouth immediately. Do not induce vomiting. Administer water if patient is conscious. Seek medical attention if a large amount is swallowed. Get medical advice and attention if you feel unwell.

SECTION 5: FIRE FIGHTING MEASURES

5.1 EXTINGUISHING MEDIA

Suitable Extinguishing Media	Not Flammable. Non-Combustible. Isolate area and use extinguishing media appropriate for surrounding fire.
Unsuitable Extinguishing Media	None known.

5.2 SPECIFIC HAZARDS ARISING FROM PRODUCT

Fire Hazard	This material does not burn. Fight fire for other material that is burning. Use water spray to cool fire-exposed containers and structures. Heat is generated when product mixes with water. Isolate and restrict area access.
Explosion Hazard	Not considered an explosion hazard.

5.3 SPECIAL PROTECTIVE EQUIPMENT AND PRECAUTIONS FOR FIRE-FIGHTERS

Fire-fighting Instructions	Keep Upwind. Under conditions of fire this material may produce Calcium oxides; Hydrogen chloride gas.
Protection during fire-fighting	Wear full firefighting turn out gear (full Bunker gear) and respiratory protection (SCBA).
Other Information	Do not all run-off from firefighting to enter drains or water courses.

SECTION 6: ACCIDENTAL RELEASE MEASURES

6.1 PERSONAL PRECAUTIONS, PROTECTIVE EQUIPMENT AND EMERGENCY PROCEDURES

General Measures	Firefighters should wear self-contained breathing apparatus and full firefighting turnout gear. Use personal protection equipment.

Protective Equipment for Emergency & Non-Emergency Personnel	Wear suitable protective clothing, gloves and eye/face protection including tight fitting goggles in areas of high fume concentration. Wear NIOSH approved respiratory protective equipment when workplace conditions warrant use of respirator.
Spills	Isolate area, eliminate source and contain spilled material if possible, recover material and reuse or collect for disposal. Prevent spills from entering sewers or waterways.

6.2 ENVIRONMENTAL PRECAUTIONS

If spill could potentially enter any waterway, including intermittent dry creeks or in case of accident or road spill notify CHEMTREC at 800-424-9300 (in USA) or CANUTEC at 613-996-6666 (in Canada). In other countries call CHEMTREC at (International code) +1-703-527-3887.

6.3 METHODS AND MATERIALS FOR CONTAINMENT AND CLEANING UP

For Containment	Contain and collect all material. Do not allow into soils, ditches, drains or water courses or dispose of where ground or surface waters may be affected.
Methods for Cleaning Up	Recover the product by vacuuming or suitable tools / PME into suitable containers. If uncontaminated, recover and reuse as product.

SECTION 7: HANDLING AND STORAGE

7.1 PRECAUTIONS FOR SAFE HANDLING

Precautions for Safe or Handling: Heat developed during diluting or dissolving is very high. Use cool water when diluting dissolving (temperature less than 27°C). For industrial use only. Handle and open containers with care. Avoid contact with eyes, skin and clothing.

Do not ingest. Avoid inhalation of chemical. Empty containers may contain hazardous product residues. Keep the containers closed when not in use. Protect against physical damage. Use appropriate personnel protective equipment.

7.2 CONDITIONS FOR SAFE STORAGE

Storage Conditions: Protect against moisture. Keep containers tightly closed. Store in a cool, dry, well ventilated area.

SECTION 8: EXPOSURE CONTROLS/PERSONAL PROTECTION

8.1 CONTROL PARAMETERS

Chemical Name	Alberta OEL	BC OEL	Ontario	Quebec OEL	Exposure Limit - ACGIH	Immediately Dangerous to Life or Health - IDLH
Calcium Chloride	Not available	Not available	TWA: 5 mg/m^3	Not available	Not available	Not available

8.2 EXPOSURE CONTROLS

Appropriate engineering controls:	Provide general and/or local exhaust ventilation to control airborne levels below the exposure guidelines.
Personal protective equipment:	 Gloves. Safety glasses. Protective clothing.
Hand Protection:	Neoprene gloves. NOTICE: The selection of a specific glove for a particular application and duration of use in a workplace should also take into account all relevant workplace factors such as, but not limited to: Other chemicals which may be handled, physical requirements (cut/puncture protection, dexterity, thermal protection), potential body reactions to glove materials as well as the instructions/specifications provided by the glove supplier. Nitrile gloves. Vinyl gloves. Appropriate chemical resistant gloves should be worn. Polyvinylchloride (PVC) gloves.
Eye Protection:	Chemical safety glasses with side shields or splash proof goggles.
Skin and Body Protection:	Wear suitable protective clothing
Footwear:	Normal
Respiratory Protection:	If exposure exceeds occupational exposure limits, use an appropriate NIOSH approved respirator. In case of spill or leak resulting in unknown concentration, use a NIOSH approved supplied air respirator. Respirator must have a chemical cartridge and particulate filter.

SECTION 9: PHYSICAL AND CHEMICAL PROPERTIES

9.1 INFORMATION ON BASIC PHYSICAL AND CHEMICAL PROPERTIES

Physical State	Solid Pellets
Color	White
Odor	Odorless
pH	No data available
Melting Point	773ºC
Freezing Point	1424ºF
Boiling Point	>815ºC (1500ºF)
Flash Point	No data available
Self-Ignition temperature	Not flammable
Decomposition temperature	No date available
Flammability (solid, gas)	Not flammable
Vapor Pressure	<0.005 mm Hg @ 20ºC
Relative Vapor Density	No data available
Bulk Density	52 - 58 lb/ft3
Solubility	Soluble in water.

SECTION 10: STABILITY AND REACTIVITY

Reactivity/Chemical Stability	Stable at ambient temperature and under normal conditions of use.
Possibility of Hazardous Reactions	No additional remark.
Conditions to Avoid	Hygroscopic (absorbs moisture from the air). Moisture.
Incompatible Materials	Heat is generated when mixed with water. Spattering and boiling can occur. Sulphuric acid. Reaction of bromide impurity with oxidizing materials may generate trace levels of impurities such as bromate. Corrosive when wet. Flammable hydrogen may be generated from contact with metals such as zinc or sodium.
Hazardous Decomposition Products	Hydrogen chloride gas. Calcium oxide.

SECTION 11: TOXICOLOGICAL INFORMATION

11.1 Likely Routes of Exposure

Inhalation	May cause nose and throat irritation. Dust may be irritating to the respiratory tract. Vapors are unlikely due to physical properties.
Eye Contact	Dusts may cause severe irritation with corneal injury. Effect may be slow to heal. When dissolving, the heat produced may cause more intense effects as well as thermal burns.
Skin Contact	When dissolving, the heat produced may cause more intense effects as well as thermal burns. No significant irritation expected from a single short-term exposure. A single exposure is not likely to result in the material being absorbed through the skin in harmful amounts. May cause more severe response if skin is damp. Prolonged or repeated exposure may cause skin irritation, even a burn. May cause more severe response if confined to skin or skin is abraded (scratched or cut).
Ingestion	Ingestion may cause gastrointestinal irritation or ulceration. Harmful if large amounts are swallowed. Small amounts swallowed incidental to normal handling operations are not likely to cause injury. Single dose oral toxicity is low.

11.2 Toxicological Effects

Symptoms Potassium Chloride: In animals, effects have been reported on the following organs following ingestion: gastrointestinal tract, heart, kidney. Dose levels producing these effects were many times higher than any dose levels expected from exposure due to use.

11.3 Numerical Measures of Toxicity

Acute Toxicity The following values are calculated based on chapter 3.1 of the GHS document.
ATEmix (oral) 1,009.00 mg/kg ATEmix (dermal) 5,160.00 mg/kg

Chemical Name	Oral LD50	Dermal LD50	Inhalation LC50
Calcium Chloride 10043-52-4	= 1000 mg/kg (Rat)	> 5000 mg/kg (Rabbit)	Not available

11.4 Delayed and immediate effect as well as chronic effects from short and long-term exposure

Skin Corrosion / Irritation	When dissolving, the heat produced may cause more intense effects as well as thermal burns. No significant irritation expected from a single short-term exposure. A single exposure is not likely to result in the material being absorbed through the skin in harmful amounts. May cause more severe response if confined to skin or skin is abraded (scratched or cut). May cause more severe response if skin is damp. Prolonged or repeated exposure may cause skin irritation, even a burn.
Serious Eye Damage / Irritation	Dusts may cause severe irritation with corneal injury. Effects may be slow to heal. When dissolving, the heat produced may cause more intense effects as well as thermal burns.
Respirator or Skin Sensitization	No Information available.
Germ Cell Mutagenicity	No information available.
Carcinogenicity	No information available.
Reproductive Toxicity	In vitro genetic toxicity studies were negative.
S.T.O.T – Single Exposure	No information available.
S.T.O.T – Repeated Exposure	No information available.
Aspiration Hazard	No Information available.

SECTION 12: ECOLOGICAL INFORMATION

Chemical Name	Ecotoxicity - Freshwater Algae	Ecotoxicity - Fish Species Data	Toxicity to microorganisms	Crustacea
Calcium Chloride 10043-52-4	Not available	10650 mg/L LC50 (Lepomis macrochirus) 96 h static	Not available	LC50: 2280000 - 3948000µg/L (48h, Daphnia magna)

Persistence and degradability - No information available.
Bioaccumulation - No information available.
Other adverse effects - No information available

SECTION 13: DISPOSAL CONSIDERATIONS

Waste disposal recommendations: Dispose in accordance with local, state/provincial, and national requirements.

SECTION 14: TRANSPORT INFORMATION

	UN Number	UN Proper Shipping Name	Hazard Class	Packing Group	Environmental Hazard
DOT	None	Not Regulated	None	None	

SECTION 15: REGULATORY INFORMATION

Safety, health, and environmental regulations specific for the product in question.

CERCLA Section 103: Not Listed

SARA Hazard Category (311/312): None

SARA 313: This product contains the following chemicals subject to Annual Release Reporting Requirements
Under SARA Title III, Section 313 (40 CFR 372): None

EPA TSCA Inventory: All of the ingredients in this product are listed on the EPA TSCA Inventory.

California Proposition 65: This product contains the following substances known to the State of California to cause cancer and/or reproductive harm (birth defects): None

SECTION 16: OTHER INFORMATION

NFPA Health Hazard 1 – Exposure could cause irritation but only minor residual injury even if no treatment is given.

NFPA Fire Hazard 0 – Materials that will no burn.

NFPA Reactivity 0 – Normally stable, even under fire exposure conditions, and are not reactive with water.

Issue Date/ Revision: 2/26/21 **Prepared by:** Technical Department

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MATERIAL SAFETY DATA SHEET

ODOR KILL POWDER FUEL OIL DEODORIZER

FOR CHEMICAL EMERGENCY: Spill, Leak, Fire, Exposure, or Accident - Call **INFOTRAC** - Day or Night: **1-800-535-5053**
THIS MSDS COMPLIES WITH 29 CFR 1910.1200 (HAZARD COMMUNICATION STANDARD) **IMPORTANT**: Read this MSDS before handling & disposing of this product. Pass this information on to employees, customers and users of this product.

PRODUCT IDENTIFICATION

DOT Shipping name:	MAGNESIUM ALUMINUM SILICATE	**CAS NO.:** MIXTURE
Chemical Family:	ATTAPULGITE	**UN/NA #:** N/A
DOT Hazard Class:	NONE	**DATE OF ISSUE**: 12/16

SECTION I - HAZARDOUS INGREDIENTS/EXPOSURE LIMITS

Hazardous Ingredients:	CAS #	TLV/PEL	AGENCY	TYPE	SARA-313(% Range)
NONE					

SECTION II - EMERGENCY AND FIRST AID PROCEDURES

EYE CONTACT: Move person away from exposure and into fresh air. If irritation or redness develops, flush eyes with clean water and seek medical attention. **Seek medical attention**.

SKIN CONTACT: Remove contaminated shoes and clothing and cleanse affected areas thoroughly by washing with mild soap and water. If irritation persists **seek medical attention.**

INHALATION: (breathing) Not expected to be a hazard under normal operating conditions.

INGESTION: (swallowing) Rinse mouth with water**. Seek medical advice.**

SECTION III - HEALTH HAZARDS / ROUTES OF ENTRY

EYE CONTACT: One or more components of this material may be an eye irritant.

SKIN CONTACT: Continuous contact with this material may cause drying of skin. Chemical itself is an irritant or sensitizer.

SKIN ABSORPTION: No symptoms of toxicity are anticipated by this route .

INHALATION: (breathing) Not expected to be a hazard under normal operating conditions.

INGESTION: (swallowing) Ingestion of large quantities may cause irritation of digestive tract, intestinal obstruction or constipation.

SECTION IV - SPECIAL PROTECTION INFORMATION

VENTILATION: Adequate ventilation is recommended.

RESPIRATORY PROTECTION: Not necessary under normal use.

PROTECTIVE GLOVES: The use of gloves impermeable to the specific material handled is advised to prevent possible irritation.

EYE PROTECTION: Approved eye protection to safeguard against potential eye contact, irritation or injury is recommended.

SECTION V - REACTIVITY DATA

STABILITY: Stable

INCOMPATIBILITY: (materials to avoid) This product is incompatible with: *Oxidizing agents, strong acids and bases*

HAZARDOUS DECOMPOSITION PRODUCTS: Thermal decomposition in the presence of air may yield carbon monoxide, carbon dioxide.

HAZARDOUS POLYMERIZATION: Will not occur

SECTION VI - SPILL OR LEAK PROCEDURES

PRECAUTIONS IN CASE OF LEAK OR SPILL: If a spill occurs, material should be cleaned up with an appropriate absorbent.

WASTE DISPOSAL METHOD: Dispose of product in accordance with local, county, state and federal regulations.

SECTION VII - STORAGE AND SPECIAL PRECAUTIONS

HANDLING AND STORAGE PRECAUTIONS: Do not get in eyes, skin or clothing. Keep containers tightly closed and dry. Use and store this product with adequate ventilation. Use good personal hygiene practices. Dispose of containers in an environmentally safe manner and in accordance with governmental regulations.

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SECTION VIII - FIRE AND EXPLOSION HAZARD DATA

EXTINGUISHING MEDIA: Extinguish with dry chemical, CO_2, or a universal type foam.

FIRE AND EXPLOSION HAZARD: May decompose during contact with flames, heating elements, or in combustion engines releasing irritating gases.

FIRE FIGHTING PROCEDURES: Wear appropriate protective equipment including respiratory protection as conditions warrant (**see Section IV**). Stop spill/release if it can be done without risk. Water spray may be useful in minimizing or dispersing vapors and cooling equipment exposed to heat and flame.

SECTION IX - PHYSICAL DATA

APPROXIMATE BOILING POINT (DEG F):	N/A	**PER CENT VOLATILE:**	N/A
SPECIFIC GRAVITY (68 F):	2.4	**FLASH POINT (TCC, DEG F):**	NON-FLAMMABLE
RELATIVE EVAPORATION RATE (ESTIMATED):	N/A	**PER CENT SOLUBILITY IN WATER:**	0
VAPOR PRESSURE @20C mmHg (CALCULATED):	N/A		

SECTION X - OTHER REGULATORY DATA

SARA

SECTION 302:	NOT LISTED
SECTION 311 & 312:	NOT LISTED
SECTION 313:	See Section I.I

HMIS

Health:	0
Flammability:	0
Reactivity:	0

TSCA

All components are in full compliance with the TSCA inventory.

CALIFORNIA PROPOSITION 65

NOT LISTED

RCRA

Waste material would be a D001

CERCLA

NOT LISTED

CARCINOGENICITY:

NOT LISTED with NTP or IARC.

NOTICE

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