OHM Remediation Services Corp.
A Subsidiary of OHM Corporation

October 2, 1998

EPA Region 5 Records Ctr.
351506

Mr. A. Keith Watson
Kerr-McGee Chemical, LLC
123 Robert S. Kerr Avenue
Oklahoma City, OK 73102

VIA: Federal Express

RE: Toledo Tie Treatment Site, Toledo, OH

Subject: **Project Submittals: Work Plan**
Letter No. KMC-005

Dear Mr. Watson:

On September 29, 1998, OHM Remediation Services Corp. (OHM) submitted a draft Work Plan for the Toledo Tie Treatment Site in Toledo, Ohio. OHM has incorporated the comments that we received from Kerr-McGee Chemical LLC (KMC) and Hull & Associates, Inc. (HAI) into the enclosed final Work Plan. The Work Plan describes the following topics:

- Site description
- Project staffing, organization, and responsibilities
- Mobilization and set up activities
- Utility management, removal, maintenance and replacement
- Excavation
- Storm water management
- Sediment removal and stabilization
- French drain construction
- Transportation and disposal
- Backfill and site restoration

OHM will submit a Traffic Plan under separate cover to supplement the work plan. The Traffic Plan will satisfy the requirements of Section 4.4.4 of the Time Critical Removal Plan prepared by HAI.

16406 U.S. Route 224 East ■ P.O. Box 551 ■ Findlay, OH 45839-0551 ■ 419-423-3526

If you have any questions or require additional information, please feel free to contact me at (419) 578-4065 or Mr. Patrick Bolger at (312) 715-5904.

Sincerely,
OHM Remediation Services Corp.

William Spedding

William Spedding
Site Manager

Enclosure

pc: Scott Lockhart, HAI
Patrick Bolger, OHM
Peter Goetz, KMC w/ Enclosure
KMC/HAI/Chron Files

WORK PLAN FOR THE TIME-CRITICAL REMOVAL AT THE TOLEDO TIE TREATMENTSITE IN TOLEDO, OHIO

Submitted to:

Kerr-McGee Chemical, LLC
123 Robert S. Kerr Avenue
Oklahoma City, Oklahoma 73102

Submitted by:

OHM Remediation Services Corp.

Prepared by:

William Spedding
Site Manager

Approved by:

Patrick Bolger
Project Manager

October 2, 1998
Project 776773

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1.0 INTRODUCTION

This work plan was developed by OHM Remediation Services Corp. (OHM) to achieve Kerr-McGee Chemical LLC's (KMC) objectives for the time-critical task order at the Toledo Tie Treatment Site located in Toledo, Ohio in a moderately populated area of Lucas County. The work order is issued under a Master Agreement with Kerr-McGee. The work has been divided into tasks for ease of implementation. Actions to be taken and necessary resources are identified and detailed by task.

1.1 SITE DESCRIPTION AND HISTORY

The Site encompasses an area over 50 acres in size and is located in the City of Toledo, Lucas County, Ohio. The Site was a railroad tie treating facility owned and operated by Federal Creosoting Company (FCC) from approximately 1923 to 1959, and the American Creosoting Corporation (ACC) from 1959 to 1962. Operations ceased in 1962 when the Site was sold to the City of Toledo. In 1969, the Site was sold to Arco Realty, Inc., who subdivided the Site into a number of parcels and developed the area into a business and industrial park.

While operated by FCC and ACC, wooden railroad ties were treated with coal tar creosote at the Site. A site map of the general wood treating operations is included as Figure 1. Based on a review of aerial photographs from years 1950, 1957, 1963 and 1969, it appears that untreated lumber was stored on the eastern section of the Site, and treated wood was stored on the western section of the Site. An above ground tank farm was located in the central southern section of the Site, south of the old access road formerly known as Creosote Road. The Ohio Environmental Protection Agency (OEPA) reported in the Site Inspection report (SI, 1993) that the tank farm consisted of two 500,000 gallon, three 30,000 gallon, and four 150,000 gallon creosote tanks, and one 150,000 gallon zinc chloride tank. Suspected waste lagoons are located in the central section of the Site, north of the old access road. The suspected lagoons are located east of Arco Drive. One is directly under and two are south of the current location of Frenchmens Road. Based on a review of aerial photographs, it appears that the suspected lagoons were filled between 1969 and 1972. A currently unoccupied distribution warehouse is currently situated over an area, which the US EPA's Environmental Sciences Division reports as an irregular shaped depression. (US EPA, Aerial Photographic Analysis, Toledo Tie Treatment Site, May 1998). This area has however historically been referred to as a lagoon. This irregular shaped depression is reported by the US EPA as collecting probable overflow from the adjacent impoundment. Further investigation into the presence and distribution of potential residual contaminants beneath and adjacent to the former distribution warehouse will be conducted during the Engineering Evaluation/Cost Analysis (EE/CA) phase of the project.

Williams Ditch serves as the natural drainage in the area. When the Site operated as a wood treating facility, the ditch ran southwest to northeast along the western section of the Site. The ditch generally intersected what are now known as Arco Drive and Frenchmens Road, at

approximately a 45-degree angle. A review of aerial photos indicate the impacted portion of the ditch was rerouted to its current location during the redevelopment of the area.
The Site is located on a relatively level piece of property approximately 4,500 feet north of Swan Creek and 8,000 feet south of the Ottawa River. The Site gently slopes toward Williams Ditch, which crosses the Site from southwest to northeast. Elevations across the site range from 620 to 625 mean sea level (msl). Elevations are referenced to the Lucas County Datum.

The Site lies within the Eastern Lake Plains of the Central Lowland physiographic province of North America. This glaciolacustrine landscape typically possesses low relief and low elevation. This flat surface was created due to several widely spaced periods of continental glaciation that supplied the largely unsorted, unstratified surficial drift deposits that cover the land in this area of the state. During the most recent stages of ice retreat, released water became trapped between the retreating ice mass to the north and the glacial deposits to the south and proglacial lakes formed. These lakes produced a thin veneer of lacustrine deposits over the glacial tills.

More specifically, the surficial lacustrine deposits consist of two distinct types: silt and clay deposits representing quiet water deposition; and sand deposits representing higher energy environments (i.e. near shore). The lacustrine deposits are approximately 12 to 14 feet thick at the Site and range from silt to clay to sand.

The Ohio Department of Natural Resource (ODNR), Division of Geological Survey, Drift Thickness Map of Lucas County, Ohio (ODNR, 1985) indicates that the Site sits on the southern slope of buried valley where the drift thickness is approximately 125 feet. The buried valley trace is from the southwest to the northeast and reaches a maximum depth of approximately 150 feet north of the Site. The glacial drift overlies Devonian limestone or dolomite bedrock.

The ODNR Ground-Water Resources Map of Lucas County indicates that the principal aquifer beneath the Site is a thin, discontinuous sand and gravel lenses interbedded in the clay till filling the preglacial valley. Yields of approximately 10 to 20 gallons per minute (gpm) are encountered at depths of 120 feet or less.

Higher yields may be obtained from the underlying carbonate aquifer. The area in the vicinity of the Site is served by a municipal water supply system, and local use of ground water for potable consumption is expected to be minimal or non-existent. A detailed review of available well record data, City of Toledo water account information, and the municipal water supply system pertinent to the Site will be conducted during the Engineering Evaluation/Cost Analysis (EE/CA) phase of the project.

1.2 STATUS OF SITE REMOVAL ACTIVITIES

A number of environmental investigations were conducted at the Site from 1987 to 1995. Key documents were describing site conditions include the "Initial Investigation and Preliminary Risk Assessment" report dated June 27, 1990, by Midwest Environmental Consultants, "The

Hydrogeology and Creosote Contamination of an Abandoned "Wood Preserving Plant Site at Toledo, Ohio," report dated December 1995, by Greg Victor Lesniak of the University of Toledo, and the 1993 Ohio EPA Site Inspection Report (SI). Results of soil, groundwater, and surface water samples collected from the Site during these investigations indicated contamination from creosote compounds existed near the suspected lagoons, former process area, and Williams Ditch. Some of the major individual polynuclear hydrocarbons (PAHs) detected were naphthalene, benzo(a)pyrene, phenanthrene, chrysene, fluoranthene, acenaphthalene, pyrene, and dibenzo(a,h)anthracene. The SI in the Administrative Record reports that concentrations were detected in the range of 100s to 1,000s of parts per million (ppm) in the soil, sediment, and surface water. Investigations conducted by Ohio EPA in 1993, and the Ohio Department of Health in 1995, determined that sediments in some areas of Williams Ditch were saturated with creosote.

The Administrative Record for the Toledo Tie Treatment Site reports that on September 25, 1997, following a significant rain event in Toledo, Ohio, the National Response Center was notified of the presence of a sheen of an unknown oil in Williams Ditch. On October 1, 1997, representatives of the U.S. EPA Emergency Response Branch evaluated conditions in Williams Ditch and observed an oil sheen upgradient of the National Super Service storm sewer outfall to Williams Ditch. U.S. EPA documented that the sheen was heavy in the ditch east of Arco Drive (50 to 100 feet) and north (50 to 100 feet) of the location of the suspected creosote lagoon areas. This area of heavy sheening is where a storm sewer apparently runs through the suspected lagoon area to Williams Ditch.

At the request of the U.S. EPA, KMC initiated abatement activities to preclude sheen migration in Williams Ditch on October 10, 1997, and continued these efforts until the issuance of a Unilateral Administrative Order (UAO), on December 24, 1997. Pursuant to the terms of the UAO, KMC prepared work plans for conducting field investigations for the time critical phase and for the Engineering Evaluation/Cost Analysis (EE/CA). A site specific Health & Safety Plan (HASP) was also prepared, which addressed anticipated removal activities. Field investigations to collect data on surface water, soil, sediment, and air at the site were conducted between April 27, 1998 and June 1, 1998, in accordance with the Removal Action Work Plan prepared by Hull & Associates, Inc. (HAI, April 1998, approved by the U.S. EPA on April 29, 1998). A second air sampling event, outside the original work plan, was conducted on August 13, 1998. These investigations are briefly described in Section 2.0.

Currently, creosote related contamination that accumulates on the surface of water in Williams Ditch is recovered on a weekly basis. Two siphon dams were constructed and are functioning as intended to control the downstream migration of visible oil and oil sheen in Williams Ditch. The site has been secured with high-visibility fencing and warning signs. Support facilities and equipment, including a site trailer and a dedicated tractor to maintain the grass, have been mobilized. Site visits are conducted on a regular basis to document the site's security status and to conduct perimeter ambient air monitoring.

1.3 PROJECT OBJECTIVES

Section IV, Item 6 of the UAO identifies the project objectives, pursuant to Section 300.415(b)(2) of the National Oil and Hazardous Substances Pollution Contingency Plan (NCP), driving the implementation of time critical removal activities at the Toledo Tie Treatment Site. The proposed remedy addresses the project objectives by mitigating potential migration and exposure pathways through source removal and engineering controls. The time-critical remedy selected by KMC is:

- Excavation of sediment to remove the most immediate source of contamination to the waters of Williams Ditch.
- Removal of immediate source areas in and around the two westernmost lagoons and Williams Ditch.
- Removal and replacement of a storm sewer which passes through the suspected former lagoon which lies beneath the current location of Frenchmens Road.
- Installation of engineering controls in the form of a subsurface barrier system with a french drain to control potential future migration to Williams Ditch and to address areas of residual contamination.

Removal of the immediate source of creosote contamination in Williams Ditch and installation of engineering controls, as described above, are consistent with Section V, Items 3.5 and 3.7 of the UAO.

1.4 REGULATORY FRAMEWORK

This project will be conducted under Section 106 of the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA).

1.5 PERTINENT PAST PERFORMANCE

OHM and the task order team members have pertinent past experience performing remedial actions at wood treatment facilities and other facilities contaminated with wood treatment chemicals throughout the United States. Table 1.1 presents several of these projects and the work performed.

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TABLE 1.1

PERTINENT EXPERIENCE

PROJECT NAME	TASK 1-Mobilization	TASK 2-Site Stabilization	TASK 3- Waste Sampling/ Disposal Profiling	TASK 4-Drum Handling	TASK 5-Removal of Liquids/Sludge	TASK 6-Decon/Removal of Vessels, USTs, and Process Equipment	TASK 7-Scrap Tanks/Vessels and Process Equipment	TASK 8-Debris Segregation	TASK 9-Soil Excavation	TASK 10-Construct Storage Cell	TASK 11-T&D	TASK 12-Backfill/Restoration
Defense Logistics Agency Defense Depot, TN	✓	✓	✓	✓	✓	✓		✓	✓		✓	✓
Emergency Response to a Fire at a Wood Treatment Facility, CA	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓
American Creosote Works, TN	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Joseph Forest Products Site, OR	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓
Havertown PCP NPL Site, PA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Madisonville Creosote Facility, LA	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓
Escambia Wood Treating Superfund Site, FL	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Escambia Camilla Wood Preserving Site, GA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Old Midland Products Site, AR	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓
Former Wickes Forest Industries Site, NC	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Selma Pressure Treating Superfund Site, CA	✓	✓	✓					✓	✓	✓	✓	✓
Cape Fear Wood Preserving Site, NC	✓	✓	✓		✓	✓	✓	✓	✓	✓	✓	✓

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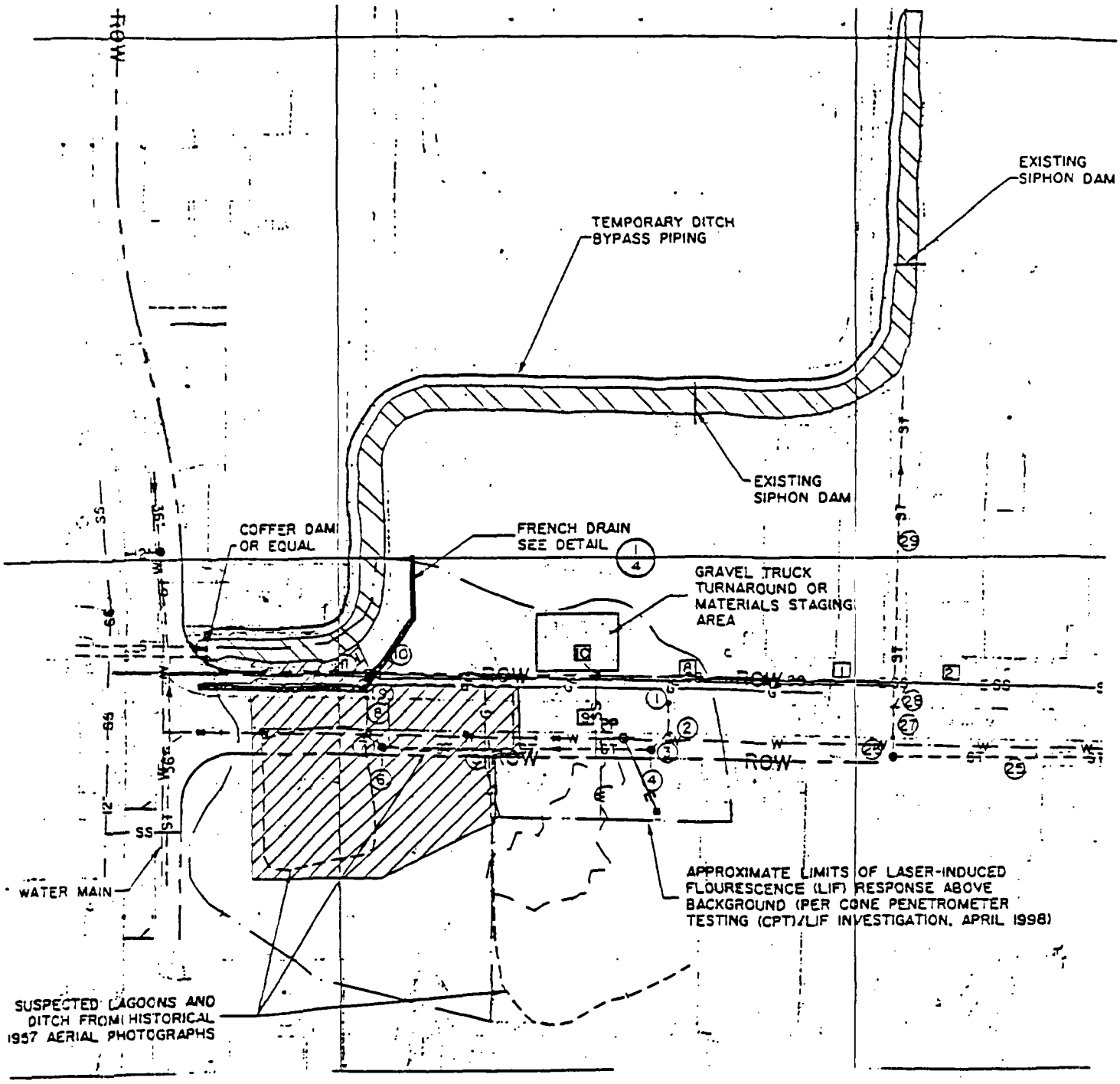

FIGURE 1.1
TIME CRITICAL REMOVAL
TOLEDO TIE TREATMENT SITE
CITY OF TOLEDO, OHIO
SITE MAP

2.0 MANAGEMENT APPROACH

The following sections describe OHM's management approach to completing the time-critical task order to the satisfaction of KMC. The key elements in OHM's management approach are the project organization, personnel, communication, cost control, schedule, resources, and subcontract management.

2.1 PROJECT ORGANIZATION

OHM's approach to project management is to place the management at a level close to the client. OHM's project manager works directly with the client to achieve the client's satisfaction with the project. Therefore, the project manager will have overall project responsibility to the client from a schedule, cost, and resources aspect. OHM has assigned a site manager to be responsible for accomplishing the work in the field. The site manager reports directly to the project manager. The site manager is responsible for the day-to-day activities in the field.

The project manager and site manager will jointly develop the project schedules and budgets and work to achieve these goals over the duration of the project. The schedule and budgets also include the resources required. The required resources will be reviewed with OHM's regional resource manager to schedule the necessary resources for the project. These activities are part of the initial planning activities and act as a baseline for measuring the progress of the project. The site manager will be responsible for project submittals and compliance with the specifications and the work plan.

The site safety officer (SSO) is responsible for implementing the Health and Safety Plan (HASP), which satisfies federal and local regulations and is consistent with site conditions. The SSO may take actions independent of the project group to stop the project, if required, for compliance with the HASP.

OHM will also provide a project accountant (PA) to assist in compiling the daily site costs and assist in procurement activities. The proposed project organization is presented in Figure 2.1. The duties and responsibilities of the project team members are defined in Section 2.2.

2.2 PERSONNEL – DUTIES AND RESPONSIBILITIES

2.2.1 OHM Responsibilities

The responsibilities of OHM are:

- Perform the remedial activities defined in the HAI plan and required under this work order.

- Conduct weekly progress meetings to discuss project safety, quality, schedule, and operations. Previous activities and accomplishments, planned activities, issues, etc. will be reviewed. Agendas and meeting minutes will be distributed to interested parties.
- Prepare and submit to KMC monthly status reports containing such information regarding percentage of completion, unresolved delays (encountered or anticipated) that may affect the schedule and a description of efforts made to mitigate those delays or anticipated delays, revised construction schedule, listing of activities scheduled for the next month, and other information relating to the progress of construction as is customary in the industry.
- Initiate, maintain, and supervise all safety precautions and programs in connection with the work.
- If conflict, error, or discrepancy is found in the contract documents, OHM will obtain a written interpretation or clarification from KMC.
- Notify KMC of any physical conditions encountered that differ materially from those specified or indicated.
- Accurately track project cost.
- Coordinate internal resources to meet the project schedule.
- Communicate with KMC's representative so that project information can be disseminated to interested parties.

2.2.2 Responsibilities of OHM's Project Management Team

A project-dedicated team that is responsible for the management and completion of the overall project and primary components of design and remediation will lead OHM's removal action. The organization chart defines the primary "chain of command".

The project manager will have the overall responsibility for project efforts including technical, schedule, and budget aspects. The project manager will be responsible for the day-to-day management and integration of all elements of the project and will be accountable for each activity. Supporting the project manager in the field will be the site manager, project superintendent and PA.

Responsibilities and authority of the project manager and supporting field personnel fundamental to the project are discussed in the following sections.

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2.2.3 Project Manager

The project manager is the person in charge of the overall project and has full authority for coordination and direction of the project. The project manager will communicate directly with KMC. Specific responsibilities of the project manager include:

- Interpret and plan overall work effort
- Approve work products, plans, deliverables
- Develop, review and meet work schedule and budget objectives
- Ensure technical adequacy of field, laboratory, data management, and construction activities
- Document the need for contract modification, if needed
- Discuss significant project issues with the client

To carry out these functions, the project manager will have the authority to:

- Allocate additional personnel as needed
- Establish work budgets and schedules with milestones
- Maintain OHM quality standards

2.2.4 Site Manager

The site manager is the OHM contact at the site and is responsible for performing the remediation activities in accordance with the work plan and other project plans and specifications. The site manager will also be responsible for:

- Managing the day-to-day execution of the project at the site including administrative and procurement activities
- Review and approve invoices
- Respond to resource requirements by defining resource needs and securing the commitments for staff and equipment
- Communicate with the site superintendent about day-to-day activities

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- Monitoring work progress and schedule, and advise project manager of variances
- Make work assignments for staff and subcontractors
- Monitor subcontract performance, schedules, budgets, and invoices
- Approve subcontractor work and invoices
- Reviewing results of on-site testing and inspection reports
- Performing, or causing to be performed, daily inspections and tests of the scope and character necessary to achieve the quality of construction outlined in the plans and specifications for work under the contract performed on or off site
- Maintaining the latest applicable drawings and specifications with amendments and/or approved modifications at the job site and assure that they are used for shop drawings, fabrication, construction, inspections, and testing
- Establishing and maintaining a Rework Item List of work that does not conform to specifications. Tracking and monitoring the items on the list to assure the rework inspection and testing activities and frequencies are in accordance with the contract requirements
- Confirming the quality and quantity of materials delivered to the site as referenced by the project plan and/or design drawings

2.2.5 Project Superintendent

The project superintendent's responsibilities include, but are not limited to:

- Day-to-day coordination of field activities
- Implementing the day-to-day aspects of the HASP and Work Plan
- Reporting to the site manager changes desired in the contract documents so that required review and approval can be accomplished prior to when the change is made, and reporting for review and approval changes necessitated by unanticipated site conditions.
- Procuring, with approval of the site manager, subcontractor services, vendor services, and materials.

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2.2.6 Site Safety Officer

The SSO is responsible for implementing the HASP that satisfies federal and local regulations and is consistent with site conditions. The SSO may take actions independent of the project group to stop the project, if required, for compliance with the HASP.

The project superintendent is responsible for the day-to-day implementation of the HASP during site activities. The SSO will oversee this day-to-day implementation, including the following responsibilities:

- Directing the entrance and exit medical physical requirements, if required
- Approval of personal protective equipment and safety procedures specified in the HASP
- Overseeing the maintenance and use of field monitoring equipment necessary to define on-site hazards associated with remediation
- Designating appropriate personnel protection level; determine protection level upgrades and downgrades as site conditions permit
- Providing necessary guidance to the project staff so they can safely perform their functions in accordance with federal and state regulations
- Identifies areas of non-compliance
- Ensures that corrective actions are implemented

2.2.7 Project Accountant

The responsibilities of the PA are:

- Accrue daily costs
- Assist the project manager in preparation of schedules, budgets, and invoices
- Maintain tracking systems to track costs and budget variances
- Provide weekly progress reports on budget and schedule status to the project manager
- Prepare daily report deliverables
- Audit weekly postings of charges to work budgets

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- Assist site manager in procurement activities
- Finalize costs for invoices to KMC
- Perform on site administrative duties

2.2.8 Transportation and Disposal Coordinator

OHM will assign a transportation and disposal (T&D) coordinator to the project team to manage the T&D of the various wastes, as necessary. The T&D coordinator will not be on site during the project. The T&D coordinator will be responsible for assisting with manifest preparation, and for obtaining cost-effective T&D options and disposal facility approval. The T&D coordinator will work closely with the site manager to ensure timely scheduling of trucks.

The responsibilities of the T&D coordinator are:

- Characterize and identify waste streams
- Ensure regulatory compliance
- Coordinate off-site disposal activities
- Prepare Material Profile Data Sheets for waste streams
- Prepare CERCLA Off-Site Disposal Reports

2.2.9 Buyer

The buyer will solicit quotations from potential vendors and subcontractors. The quotations will be evaluated for completeness and cost. The lowest responsible bidder will receive the award. The buyer will not be on site during the project.

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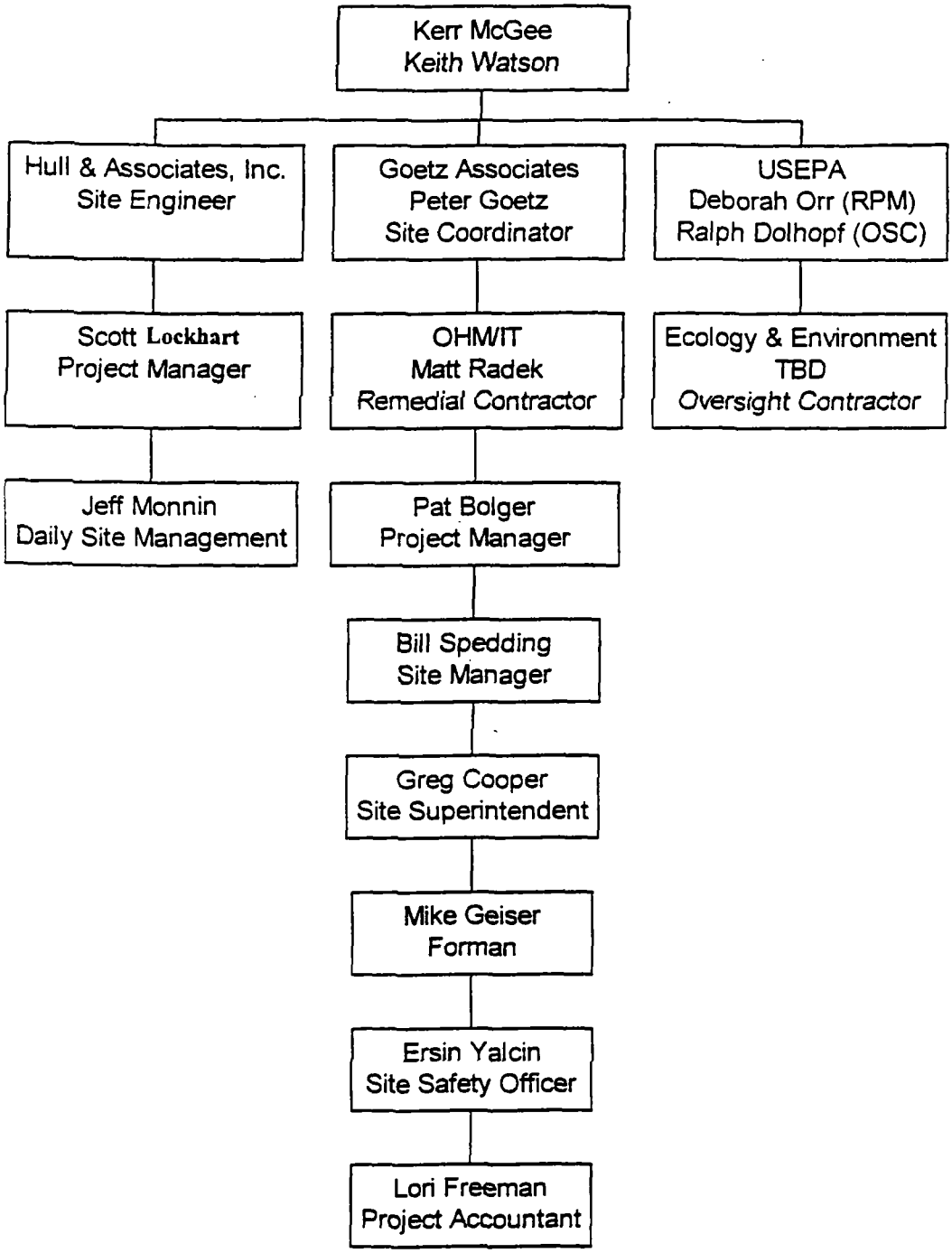

FIGURE 2.1
ORGANIZATION CHART

2.3 PROJECT COMMUNICATION

Clear and constant communication with KMC from work plan development through project completion is vital to the safe, efficient, and effective execution of this project. Ongoing communication will include standard and formal contract deliverables and informal project briefings with KMC as required. OHM's on-site Site Manager (SM) will ensure daily contact with KMC is maintained. KMC will be consulted at all decision points that require guidance or input. Decision points may include, but are not limited to, site resources, technical approach options, schedule, cost or billing issues, cleanup levels, analytical data review, transportation and disposal, planning, and potential changing site conditions.

OHM's PM will be KMC's point of contact for all project related activities and is empowered to commit resources on behalf of OHM. A phone and pager list of all site personnel and key off-site support personnel will be developed and used to maintain communication throughout the project. The SM will also maintain communication with the off-site Program Support Group on an as-needed basis to discuss resources, upcoming tasks, required technical support, and other issues. OHM is aware that the KMC is the project lead with respect to external communication and OHM personnel will defer all questions regarding the project to the KMC.

2.4 COST CONTROL

Cost control is paramount in providing cost-effective and proper service to KMC. All personnel involved with this cleanup will play a vital role in cost control. The PM will review with the KMC all cleanup requirements including procurement of equipment, personnel, materials, and subcontractors. Contract requirements in regards to cost control will be known and followed. OHM is committed to cost savings under this contract. These cost saving measures and others will ensure that OHM provides the best value service at the best price possible to KMC.

OHM has the ability to provide a cost-effective service for many reasons. OHM has a fleet of owned equipment fully maintained and ready to respond immediately. Secondly, OHM has a vast database/network of vendors and subcontractors that can be easily procured in emergency or time-critical situations. Three bids will be received for most purchases, rentals, and services. The decision to mobilize owned equipment or rent locally will be based on cost effectiveness as directed by the KMC.

2.5 PROJECT SCHEDULE

The proposed project schedule is presented in Figure 2.2. Sufficient resources will be allocated to compress the project into the shortest possible schedule, while maintaining the quality required to meet the project objectives. Effective schedule reduction will allow for optimum utilization of administrative and support resources and will result in significant cost savings. The schedule also incorporates overlapping tasks in order to optimize time on site, prevent multiple mobilizations, and reduce risks associated with the site in the shortest possible time period.

Activity ID	Activity Description	Orig Dur	Early Start	Early Finish
500	EPA Plan Approval	0	01SEP98	
1000	Contractor Plans	18	10SEP98	05OCT98
2000	Mobilization and Site	25	22SEP98	28OCT98
3000	Williams Ditch Rerouting	10	13OCT98	28OCT98
4000	Clear and Grubbing	5	06OCT98	12OCT98
5000	Staging Areas- Williams	10	08OCT98	19OCT98
6000	Water Treatment System	10	29SEP98	12OCT98
7000	Health and Safety- Air	67	22SEP98	28DEC98
8000	Utility Management,	65	22SEP98	23DEC98
9000	Soil Excavation (8,500 cy)	46	20OCT98	24DEC98
10000	Williams ditch sediment	35	27OCT98	18DEC98
11000	Road removal	10	18DEC98	04JAN99
12000	French Drain Installation	10	11DEC98	24DEC98
13000	Backfill and site	10	11DEC98	24DEC98
14000	Demobilization and final	10	22DEC98	08JAN99
15000	Project Management	73	22SEP98	08JAN99
16000	Transportation and	49	20OCT98	30DEC98
17000	Project complete	0		08JAN99

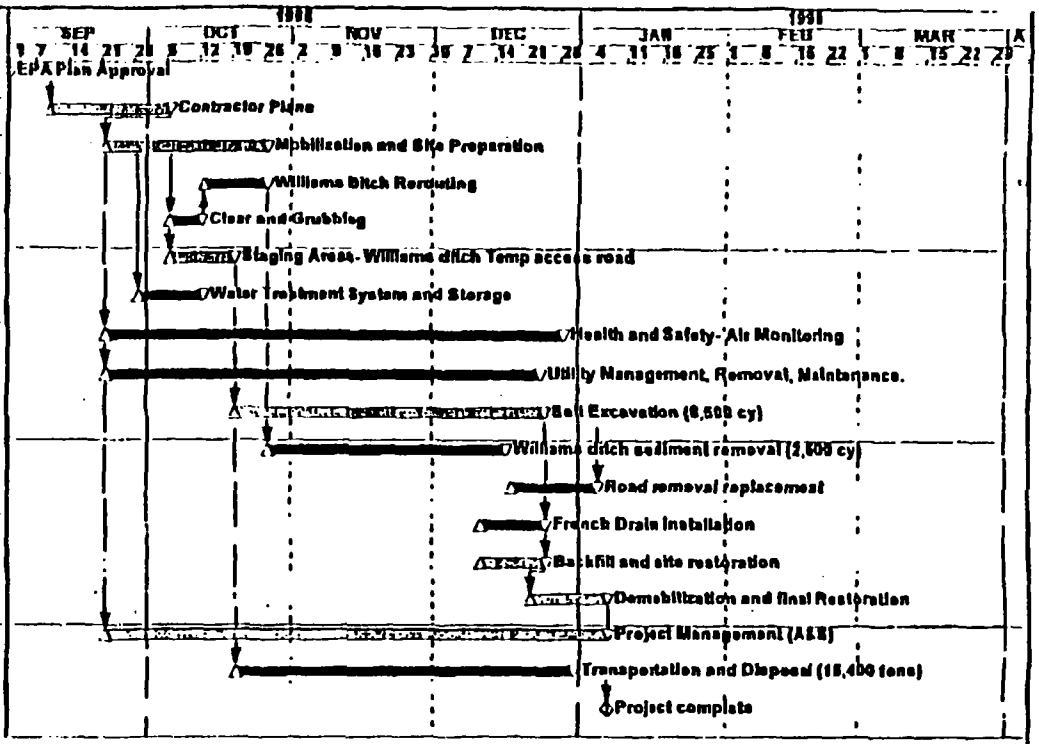

FIGURE 2.2
SUMMARY SCHEDULE

2.6 PROJECT RESOURCES

Utilization of resources will be optimized throughout the project by performing tasks concurrently and by sharing resources between tasks. This approach will minimize mobilization, labor, and equipment costs.

2.7 SUBCONTRACT MANAGEMENT

OHM will utilize several subcontractors during the task order. The subcontractors will provide the following services: ditch rerouting and dewatering, laboratory analytical services, electrical and telephone installation, transportation and disposal of wastes, soil and stone material delivery, and liner installation. The procurement of these services will follow strict adherence to OHM's procurement policies that are based upon prudent and ethical business practices. Any onsite subcontractor, other than utilities, will be submitted to KMC and EPA for approval.

OHM's procurement process is streamlined to facility emergency responses and designed to minimize administrative burdens for ease in project site execution, while maintaining contract compliance.

OHM's acquisition strategy:

- Identifies as many sources of supplies and services as possible
- Promotes competition for all procurement through the use of pre-qualified subcontractors
- Provides acquisitions that are managed to ensure control and resource availability
- Ensures OHM controls all subcontracted items

OHM maintains full responsibility for the proper administration of all subcontractors in accordance with the terms and provisions of the contract. The contract requirements are included on purchase orders, subcontract agreements, and other procurement documents issued by OHM. Any flow down requirements of the prime contract will be incorporated into the subcontracts. OHM will submit the qualifications of each subcontractor to KMC for approval.

OHM monitors the performance of the subcontractors and requires them to meet the same level of performance that is required of OHM. Status review meetings with major subcontractors are held during those periods when their performance and adherence to the project schedule is crucial to the successful execution of the project and as may be necessary, allowing significant deviations or problems to be promptly reported.

Prior to any work being performed on site, all subcontractors will be required to attend an orientation meeting. The orientation meeting is to outline the responsibilities of the subcontractor

while on site, review the site-specific health and safety plan and discuss the work to be performed. Subcontractor personnel will sign a safety plan acknowledgement.

OHM's proven acquisition system assures the overall strategy, implementation, and management to provide the best value to KMC. This structured approach ensures both quality and value are factored into each step of the procurement process.

2.8 PROJECT DOCUMENTATION

OHM understands the importance of proper documentation of all project activities. The on-site management team will provide concise documentation to KMC in a timely manner. The primary means of project documentation include but are not limited to the following:

- Daily Work Reports
- Maintenance of Project Schedule
- Sampling Logs
- Analytical Reports
- CERCLA Off-Site Disposal Reports
- Site Progress Reports
- Waste Profiles and Acceptance Letters
- Manifests and Bills of Lading

2.9 SUMMARY

OHM's professional staff will be the key to meeting KMC's objectives for successful project completion within the prescribed cost and time limits. The OHM project management structure combines the various functional areas performing work on a project into a coordinated team. OHM's project team will produce quality work that meets the contract requirements. To ensure that KMC is appraised of site activities, progress, and issues, OHM will provide the following during the Toledo Tie Treatment Site remediation

- A project kickoff meeting prior to intrusive activities with representatives of the project team members. The kickoff meeting will be held at a time and location that is convenient to all project members.

- Weekly progress meetings to discuss project safety, quality, schedule, and operations. Previous activities and accomplishments, planned activities, issues, etc. will be reviewed. Agendas and meeting minutes will be distributed to interested parties.

- Daily Reports that document site activities, subcontractor activities, site personnel, equipment use, quality control inspections and tests, and safety.

- Monthly status reports containing an updated construction schedule, percentage of completion, delays that may affect the schedule and a description of efforts made to mitigate

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those delays, listing of activities scheduled for the next month, and other information relating to construction progress.

- A final report describing the work, variances from the project plans, the final construction schedule. The final report will contain copies of any work plan and HASP revisions, daily reports, weekly summaries, analytical reports, disposal documentation (e.g., waste profiles, T&D log, manifests, etc.).

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3.0 PREMOBILIZATION AND MOBILIZATION ACTIVITIES

OHM will complete several tasks prior to mobilization. These tasks will include conducting an on-scene survey and developing project-planning documents including the Work Plan and Health and Safety Plan (HASP). The following sections briefly describe the premobilization and mobilization tasks.

3.1 ON-SCENE SURVEY

Prior to mobilizing the field crews, OHM will visit the project site to accomplish an on-scene survey. During the on-scene survey, representatives of KMC/HAI and OHM will discuss the project objectives; mobilization timing; required levels of protection; and equipment, personnel, materials, and subcontractor requirements. At the discretion of KMC, other project team members may be included in the on-scene survey. The information obtained during the on-scene survey will be disseminated to the team members for use in preparing project plans and procurement of the necessary project materials and equipment.

3.2 PROJECT PLANS

Per Section 4.4 of the HAI's plan, OHM will submit the plans listed in this section to supplement this work plan.

3.2.1 Health and Safety Plan

OHM will submit a HASP that addresses anticipated site health and safety concerns for OHM's employees and subcontracts and specifically focuses on project tasks. The plan presents required information including, but not limited to: identification of key personnel and lines of communication, training, medical surveillance, site hazards, work zones, personal safety, ambient and personal air monitoring, respiratory clearance, equipment decontamination, material safety data sheets, heat stress monitoring for permeable and non-permeable clothing, respirable contaminant action levels, levels of PPE, and emergency contingencies (i.e., injury, chemical release, etc.). OHM's HASP is provided separately.

OHM will submit tabulated copies of all training records in a bound document. All persons entering the site must show proof of OSHA 40-hour training, 8-hour refresher training, medical surveillance, and respirator clearance before they will be permitted to enter the site.

HAI prepared a HASP addressing the requirements for the anticipated activities at the former Toledo Tie Treatment Site. This document is located in Appendix B of HAI's Removal Action Work Plan. OHM's HASP is as stringent as the one prepared by HAI.

3.2.2 Storm Water Management Plan

OHM is responsible for ensuring that all storm water is properly managed and segregated. Water storage will be used to contain storm water from excavations and decontamination water. The storm water plan is described in Section 4.2.2 of this work plan.

3.2.3 Dust/Odor Control Plan

OHM will implement dust control measures to keep airborne dust particulates to a minimum during site activities. Visible dust emissions will be addressed by applying misted water or using an equivalent approach. Traffic patterns will be restricted to limit the area of soil disturbance. Where possible, existing vegetation will remain in place.

OHM plans to leave the saturated ditch sediments uncovered to facilitate air-drying. However, the staging area will be covered if necessary to control dust. Dry stockpiled site soil will be covered. When necessary, stockpiled soil will be covered with a waterproof cover and secured. Each section of the cover will be overlapped. Where practical, the toe or edge of the cover will be located on the outside of the stockpile area's perimeter berm.

All over the road trucks will be covered with a tarp prior to departure from the site.

A supply of chemical suppressant will be mobilized to the site to control odors if needed. OHM intends to suppress isolated excavation odor sources with site soil. If the odor source is pervasive, OHM will apply a chemical odor suppressant to control odors.

Determination of the need for dust or odor control will be the responsibility of the OHM site manager in consultation with the SSO. The HASP contains further details on air monitoring.

3.2.4 Air Monitoring Plan

OHM will conduct ambient and personal air monitoring throughout the duration of all intrusive construction activities. OHM will conduct a baseline air-monitoring program to establish background levels of VOCs and PAHs. Since benzene was detected during HAI's May and August 1998 air sampling events, organic vapor badges will be worn by the worker(s) with the greatest potential for exposure. In addition, organic vapor badges will be placed at the perimeter to evaluate whether benzene is present. Personal sampling for PAHs will also be implemented. Real time air monitoring will be performed during both intrusive and soil handling activities. OHM will develop action levels based upon site specific chemical data prior to initiating excavation activities.

Section 7.0 of OHM's HASP provided in Appendix A contains the Air Monitoring Plan.

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3.2.5 Dewatering/Stabilization Plan

OHM will implement a program to handle and manage contaminated sediments/soils. The program will include a liquids management program incorporating at a minimum, the recovery, management, treatment and disposal of potentially contaminated water. Anticipated sources of potentially contaminated water include, but are not limited to, decontamination water, water from Williams Ditch, water entering excavations which contacts the immediate source, and water draining from excavated soils or sediments. The water treatment system is described in Section 3.6 of this work plan. If the analytical results indicate that the water quality satisfies the discharge criteria, then the water will be discharged to Williams Ditch downstream of the work area.

3.2.6 Contingency Plan

The site contingency plan will be amended as needed to address such things as on-site or off-site spills of materials leaving the site, traffic emergencies, etc. The plan will be distributed to the parties previously contacted. Prior to initiating construction, the plan will be reviewed with them.

Section 8.0, Emergency Response, of OHM's HASP satisfies the Contingency Plan requirements.

3.2.7 Traffic Plan

OHM will provide a traffic plan that identifies the proposed routing of trucks, staging of equipment, materials, contaminated soils/sediments, etc. and establish a schedule of the number and types of trucks, etc.

The Traffic Plan will be submitted under separate cover.

3.2.8 Project Schedule and Management Plan

OHM will provide a project schedule that parallels to the degree practicable, the preliminary schedule shown in Figure 8 of the HAI plan. Written documentation regarding cost control procedures, project personnel, the chain of command with defined decision-making authority, and regular progress reports are to be supplied.

Section 2.0 of the work plan contains OHM's Project Schedule and Management Plan.

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3.3 MOBILIZATION

Pre-mobilization authorization will be obtained from KMC. OHM will mobilize the personnel, equipment, and resources necessary to complete the project as defined in this work plan. Project resources will be primarily mobilized from OHM's Findlay, Ohio, operations office.

Other activities that will be accomplished during site setup will be installation of phones and office equipment; construction of a temporary decontamination pad; receipt on site of temporary sanitary facilities and common trash dumpsters; establishment of work zones and transition zones for safe entry and exit from work areas, and placement of eyewash stations, safety showers, first aid kits, fire extinguishers, and spill kits. In addition, a health and safety review meeting with on-site personnel will be conducted.

Prior to beginning work on site, a training meeting will be conducted to brief all site personnel on the HASP and other relevant site-specific plans. Site hazards and conditions will be discussed and all personnel will acknowledge their understanding and compliance with all activities and the plan by signing an acceptance form.

3.3.1 Temporary Facilities

OHM will establish an office trailer, a crew support trailer, a decontamination trailer, a tool trailer and temporary sanitary facilities at the site for the entire length of the project. OHM will install telephone and electrical service for use during the project.

3.3.2 Work Zones

OHM will mark the Support Zone, Contamination Reduction Zone (CRZ), and Exclusion Zone (EZ), in accordance with Occupational Safety Health and Administration (OSHA) guidelines. All specific work zones will be delineated with orange plastic fencing with metal posts and appropriate warning signs will be strategically placed. Caution tape, roping and fencing devices will be used as specific project tasks require.

3.3.3 Stockpile Areas

OHM will construct an onsite soil/sediment staging area to accommodate soil drying and storage. The stockpile will include a drainage system necessary to allow gravity dewatering of the excavated soils. The dimensions of the stockpile area will be approximately 75 feet by 125 feet. The welded HDPE liner will line the entire bermed stockpile area. The staging area will include two layers of 60-mil HDPE liner, a 6-inch layer of soil between the liners, and a perimeter berm. OHM will construct an equipment access ramp into and out of the stockpile area.

OHM will place a sump at a low point in the stockpile area. Accumulated water will be sent to the onsite water treatment system.

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Ditch sediments will be transferred from the ditch to the staging area. Sediment will then be allowed to drain. At this time it is unknown how fast and complete the ditch sediments will drain to meet transportation and disposal specifications.

Although OHM plans to leave the stream sediment uncovered to facilitate air drying, the staging area will be covered if the air monitoring readings indicate that it is required or during rain events. When necessary, the stockpiled soil will be covered with a minimum 6-mil polyethylene sheet and secured.

3.4 INITIAL SITE SURVEY

OHM has assumed that HAI will supply a state licensed land surveyor to perform an initial site survey and establish a construction baseline. To track progress, OHM will implement an excavation control grid for the lagoon excavation. The construction baseline will be established on the western and southern boundary of the project site. The control grid will be on 25' stationing, with the western line identified as N0+00, N0+25, N0+50, etc, and the northern line as E0+00, E0+25, E0+50, etc. The area between the stations will be identified as A, B, C, etc. for the western baseline and as 1, 2, 3, etc. for the northern baseline. The baselines will be used as a quick reference for location or replacement of the initial grid survey. The grids will be identified in the following manner A1, A2, B1, B2, etc. The example covers the first 2-stations north and east. In addition the initial site survey will establish a project benchmark (elevation) for vertical control. In addition to the project benchmark a number of temporary benchmarks (TBM) will be established within the line of sight of the active excavation/backfill area(s).

Horizontal and vertical control of the excavation will be necessary. This can be accomplished using a construction grade level. The level will be set up and calibrated each shift from the project benchmark or a temporary benchmark. OHM will record in the log the approximate area(s) and depths of excavation per shift/day. Horizontal control of the excavation area(s) will be as established by the grid layout and referenced to the construction baselines.

OHM will employ a stationing system to track the progress of the sediment removal from Williams Ditch. The activity specific stationing system will follow Williams Ditch. Station 0+00 will be located along Williams Ditch at the upstream end of the remediation. Subsequent stations will be placed at increments of one hundred feet (e.g., 1+00, 2+00, etc.). Ditch elevations will be recorded to verify the removal of two feet of sediment.

3.5 CLEARING AND GRUBBING

OHM will clear and grub the vegetation from the planned remediation areas. Clearing and grubbing includes the removal of roots, trees, brush, and stumps from work areas. Where practical, OHM will leave large trees in place. Prior to commencing clearing and grubbing operations, necessary erosion and sediment control measures will be in place.

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Trees and brush will be cut and placed in piles for chipping by utilizing a tree chipper. The chipper will process approximately 95 percent of all onsite trees. Oversized trees will be processed by hand to reduce their size. Chips will be stockpiled for use on-site (e.g., haul road materials, etc.) or will be disposed offsite. An excavator will be used to remove stumps and roots. Stumps, roots, and oversized logs will be disposed offsite.

3.6 ON-SITE WATER TREATMENT

OHM has assumed three primary sources of contact water: decontamination water, water from Williams Ditch, and excavation water. Decontamination water will be generated during equipment and personnel decontamination activities. Excavation water will result from direct rainfall into an excavation and/or groundwater infiltration into the excavation of the impacted soil. All accumulated excavation water, Williams Ditch water, and decontamination water will be treated prior to discharge.

OHM has assumed that the removal of free product and total suspended solids from contact water will be accomplished through filtration with sand cells and polishing with granular activated carbon.

The wastewater treatment plant (WWTP) will consist of the following components (listed in order of configuration):

- Two (2) 50,000 gallon contact water storage pools with double liners
- One (1) sand filter
- Two (2) carbon cells (one primary and one backup)
- Two (2) 50,000 gallon treated water storage pools

Bag filters may be added to the system as required.

Treated water will be discharged in accordance with the letter dated September 23, 1998, from the Ohio EPA. Ohio EPA has permitted continuous discharge, with confirmation laboratory analysis, to Williams Ditch. To demonstrate system performance prior to and throughout the operation of the water treatment system, effluent samples will be collected and analyzed per Ohio EPA's requirements.

During the first week of operation, the effluent will be analyzed during each day of operation. During the next eight weeks, the effluent will be sampled and analyzed once per week. Beginning with the ninth week of operation, the effluent will be sampled every other week.

The initial two batches of water (e.g., approximately 30,000 gallons) will be treated and analyzed prior to discharge. If both batches satisfy the discharge criteria, treated water will subsequently be discharged directly to Williams Ditch on a continuous basis.

If a substantial quantity of free product is recovered from the surface of the pools, then a temporary holding vessel (e.g., a drum) will be set up to temporarily store product prior to transfer into transport vehicles.

The WWTP will not be decommissioned until all of the site soil has been shipped offsite. At the end of the project, the WWTP sand and sediment will be removed for disposal at an approved offsite facility. The spent carbon will be sent either for regeneration or disposal at an approved offsite facility. To facilitate disposal of the WWTP materials, a stabilization additive may be necessary.

The pool liners will be decontaminated and transported to an OHM facility if possible. If the liners can not be decontaminated, they will be disposed offsite with the other WWTP materials.

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4.0 OPERATIONAL APPROACH

The following sections describe OHM 's operational approach for completing the scope of work at the Toledo Tie Treatment Site. This approach has been developed from information obtained during the on-scene survey, information provided in the HAI plan, and input from KMC.

4.1 UTILITY MANAGEMENT, REMOVAL, MAINTENANCE, AND REPLACEMENT

There are several subsurface and overhead utilities located within the excavation area. Sheet 3 of the HAI plan set and Section 4.1.2 of the HAI Time Critical Removal Plan provide details regarding the protection, removal and replacement of utilities.

Whenever possible, it is preferable to take an affected utility out of service and/or to relocate it temporarily. If it is not possible to take a utility out of service and it is necessary to excavate impacted soil from around, including beneath, the utility, a temporary (or permanent) shoring system will be engineered. OHM will not conduct any excavation below the utility pipe's mid-point, unless special precautions have been taken to prevent undermining. After the existing utilities have been marked, OHM will protect them during site work by maintaining three feet of cover over all utilities to allow for the passage of heavy equipment.

Although the HAI sheets provide the location of subsurface utilities, OHM will contact the Ohio Utility Protection Service prior to commencing intrusive operations. In addition, OHM will directly contact each utility prior to excavation. Once the utilities have located their services (water, gas, sewer, storm, telephone and electrical lines), OHM will verify the location (vertical and horizontal) of each utility by uncovering the utility by hand (assisted by limited mechanical excavation).

The following briefly describes the utility work:

> Electric - The existing electrical service running through the site on the north side of the Frenchmens Road and the service to the warehouse will present challenges during the excavation. OHM will discuss with Toledo Edison the temporary relocation of the east/west electrical line to north of the impacted area. This would require the temporary relocation of approximately six utility poles. If this approach proves to be impractical, temporary pole supports would be an acceptable alternative. A third approach would be to leave "islands" of site soil around each power pole. Per Toledo Edison, existing utility poles require a minimum of three to four feet of undisturbed soil around the pole. To provide a factor of safety, a minimum of five feet of undisturbed soil will be left around the pole. The soil will also be sloped according to the soil's classification (e.g., Type C, etc.). OHM will confer with Toledo Edison prior to work around the power poles.

The existing overhead electrical wires along Frenchmens Road are also a safety hazard. OHM will request that Toledo Edison place "sleeves" over low overhead electrical wires.

Storm Water - Storm sewer pipes #6-#11, a portion of #5 and the associated fixtures will be removed. Storm water controls will be reinstated per HAI's plan to insure proper management of storm water in the area. Removed concrete pipes will be disposed off-site. As the storm water manholes were cast in place, OHM anticipates that new manholes will be required. Per the City of Toledo, catch basins, manhole covers, etc. that are in good condition may be decontaminated, if necessary, and reused.

Water - The City of Toledo Department of Utilities requires the 8-inch water main be removed prior to excavation of the lagoon areas and replaced upon completion of removal activities. The City will cut and cap the water main outside of the remediation area. The US EPA or a designated representative will be onsite to observe the City cut and cap the water line. After the contaminated material has been removed, OHM will replace the 8-inch water main and all appurtenances (e.g., fire hydrants, etc.) in accordance with the City of Toledo Department of Utilities' Specifications. OHM will coordinate the installation of a temporary water service to the Support Zone to coincide with the decommissioning of the water main. The service will include a stub, valve, and water meter.

Natural Gas - Columbia Gas has already abandoned and capped their 4-inch low-pressure gas line. At this time it is not anticipated that the gas line will be replaced.

Telephone - Approximately 260 feet of the Ameritech's underground line runs through the lagoon excavation. Although it is currently anticipated that the line will remain in place, OHM will discuss the temporary relocation of the line with Ameritech. If it is not possible to relocate the line, OHM will support the line during excavation and backfill in accordance with Ameritech's specifications.

OHM will coordinate the installation of replacement utilities with the City of Toledo Department of Utilities, Ameritech, Toledo Edison, and Columbia Gas.

4.2 SOIL EXCAVATION

4.2.1 Introduction

The area indicated on Sheet 2 of the HAI plan set will be excavated to the subsurface lacustrine clay layer, which is at an average depth of approximately eight feet, or to the depth at which the immediate source is no longer visually distinguishable. Per the HAI plan, an immediate source is

defined as free flowing, creosote product or soils/sediment saturated with creosote product that is encountered within the limits shown on the plan set. Areas where there may be water with residual creosote contamination or visible sheening, but no readily visible or distinguishable presence of an immediate source will be noted and addressed during the implementation of the non-time critical activities.

If the excavated material has visual evidence of creosote contamination as defined in the previous paragraph, the soil will be transferred to the contaminated soil stockpile or will be loaded directly into licensed over the road trucks for disposal. If the presence of creosote contamination in any excavated material is questionable, OHM will verify the soil's disposition with an HAI representative. If an HAI representative is not available, OHM will stockpile the questionable soil within the excavation for further inspection.

Should free flowing, creosote product or soils saturated with creosote product be observed at the perimeter of the excavation, OHM will notify HAI prior to proceeding with any additional soil removal.

As the immediate source and non-contaminated soil will be intermixed, the same heavy equipment will used to continuously handle contaminated source material, non-contaminated site soil, and questionable site soil. Equipment will be decontaminated prior to handling clean offsite material or site soil outside of the excavation limits.

4.2.2 Storm Water Management

The primary objective will be to prevent clean water from becoming contaminated. Surface drainage features will be diverted away from work areas. OHM also plans to minimize the open excavation area by placing backfill as the excavation proceeds.

Storm water will be diverted from the excavation through the use of temporary drainage ditches and/or earthen berms. Although the location, depths, heights and lengths of such ditches and berms will be determined in the field, OHM anticipates that perimeter berms with a nominal one-foot height will be sufficient. The perimeter berms may be relocated as the work progresses to minimize the drainage area. OHM plans to use clean site overburden material to construct perimeter berms where possible. If this is not possible, OHM will use clean imported fill material. In either case, the berm material will be backfilled in the excavation at the end of the project.

There are several roof drains that discharge storm water into the work area. OHM will attach temporary drain extensions to these roof drains to convey storm water away from the work.

After the required sections of the existing storm water system have been decommissioned, OHM will construct a temporary sump at the eastern limit of the excavation area (i.e., the western end

of the remaining storm sewer) to accommodate storm water flow in the remaining section of the storm sewer. The sump location is provided on Sheet 5 of the HAI plan set.
Per Section 4.1.3 of the HAI plan, accumulated storm water may be transferred to Williams Ditch provided no visible oil or oil sheen is observed. If visible oil or sheen is observed, the affected water will be transferred to the contaminated water storage pools for treatment. As an additional precautionary measure, a float will be attached to the suction hose. The float will maintain the suction hose below the water surface.

OHM will consult with an HAI representative prior to discharge to Williams Ditch whenever possible. If an HAI representative is unavailable and the possibility of flooding exists, OHM will examine the water for visible oil or oil sheen and transfer storm water to Williams Ditch or the contaminated water storage pools as appropriate.

During after hour storm events, OHM representatives will be dispatched to the Toledo Tie Treatment Site to monitor the storm water management system.

4.2.3 Excavation Approach

Following establishment of the storm water controls and the 25-foot grid, OHM will begin the excavation. OHM intends to start the excavation and impacted soil removal from the south side of the impacted area, advancing to the north. Although the soil will be monitored for visual creosote contamination, mass excavation techniques will be employed.

Clean surficial soils (i.e., overburden) will be removed prior to the removal of the contaminated subsurface soils. Surficial soils along with existing grasses, neither visually impacted by creosote nor exhibiting visual signs of being saturated by a water/creosote mixture, will be removed and used as backfill to replace excavated source material. In order to minimize the size of the open excavation, OHM will not remove all of the clean surficial soils at once. Surficial soil will be removed in sections as the excavation proceeds. Surficial soil will be stockpiled directly on clean soil or placed in the bottom of the excavation as the excavation proceeds. Some surficial soil may be used to construct the clean perimeter berms.

4.2.4 Excavation Safety

The excavation must meet the requirements of 40 CFR 1926 Subpart P (i.e., a competent person, daily excavation permits, sloping or benching, etc.) prior to entry. OSHA's excavation standard authorizes a competent person to determine safe slopes for excavations less than twenty (20) feet in depth. The minimum slope for Type C soil, the worst case, is 1.5H to 1V. Site soil will be considered Class C unless the competent person determines that the soil can be reclassified. The competent person will verify the slope requirements based on existing conditions. Unless a visible source is observed, excavation layback material will be considered clean site soil and will be backfilled at the bottom of the excavation.

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Except for existing utilities traversing the excavation area (e.g., the Ameritech line, etc.), OHM does not plan to use any shoring during the excavation. OHM will excavate the areas adjacent to the warehouse, the warehouse parking lot, and utilities to an appropriate angle of repose to the structure. So that the excavation will not affect the warehouse, OHM proposes to leave a minimum buffer distance of five feet adjacent to the warehouse. If KMC requires vertical excavation adjacent to the warehouse, OHM will require additional subsurface soil information and information on the warehouse foundations. OHM would then develop a new system of excavation that could possibly require engineered stabilization methods (e.g., post and beam, sheetpiling, etc).

Although OHM will minimize the open excavation area, portions of the excavation will remain open for extended periods of time. The excavation will be barricaded with high visibility construction fence or equivalent to control access.

4.2.5 Excavation Dewatering

OHM will install dewatering sumps in strategic locations within the excavation. These dewatering sumps will consist of a slotted pipe of sufficient length to extend below the planned terminus of the excavation (i.e., approximately 8 feet below the original ground surface). Although the location of the dewatering sumps will be determined in the field, approximately one sump per 2,500 square feet should be sufficient. Trenches may also be excavated along the bottom of the excavation to channel water to the sumps. A small submersible pump (e.g., 2-inch or 3-inch diameter) will be placed in any active sumps. Excavation water will be discharged to the contaminated water storage pools for treatment.

4.3 WILLIAMS DITCH

4.3.1 Ditch Rerouting

OHM intends to excavate the ditch sediments in the "dry." To accomplish this objective, OHM will provide pumping capacity to divert upstream water around the active remediation section. OHM will isolate Williams Ditch by installing a coffer dam or a low permeability soil plug upstream of the remediation area. A sump arrangement, fitted with two 12-inch pumps (i.e., a primary and secondary pump), will be used to convey water through a diversion pipe around the effected portion of Williams Ditch. The pumps will be connected to a piping system that will permit the use of both bypass pumps. A single 12-inch pump has the capacity to transfer approximately 6,000 GPM (i.e., 13.3 CFS). Conveyed water will be discharged onto a "scour pad" located downstream of the remediation area. The scour pad will consist of geotextile and rip rap.

To facilitate effective water management during the ditch remediation, OHM will construct dams downstream of the active remediation area. These dams will isolate discrete sections (e.g., 200 feet to 300 feet in length) of Williams Ditch. The dam will be approximately 3 feet wide at the

top. To prevent flooding in the area, the top of the dam will remain below the existing banks of Williams Ditch.

The dam will be covered with geotextile to reduce erosion in the event that the flow tops the dam during a significant storm event. The geotextile will be secured on both sides of the dam. Ground personnel will also be able to utilize the dams as an access way to the north side of Williams Ditch.

After an individual section of Williams Ditch has been remediated, the previous downstream dam will become the new upstream dam and a new downstream dam will be constructed. Depending on site conditions, the previous upstream dam may be removed immediately or at the end of the project. The dam's surficial soil will be removed and treated as contaminated soil. If the dam is removed during the course of the project, the dam's core soil may be used in subsequent dams.

No flow or low flow conditions would be ideal for the installation of the sump and the initial upstream dam. If the sump can not be constructed during favorable conditions, OHM will place and secure plywood sheets across the dual 48-inch culverts upstream of Arco Drive in an attempt to temporarily interrupt the flow. OHM will monitor the water level upstream of Arco Drive during the operation. Accumulated water that remains in the ditch will be pumped ahead of the isolated section and allowed to flow downstream through the existing siphon dams to facilitate the construction of the sump and initial upstream dam in the "dry". Any accumulated water that remains below the invert of the siphon will be pumped out. If there is a visible sheen, the water will be pumped to the water treatment system prior to discharge to a scour pad located in Williams Ditch.

After the flow has been interrupted, the initial upstream dam can be constructed. Prior to the placement of the upstream dam material, the area immediately downstream of the dual 48-inch culverts will be remediated to provide a clean area for the sump and the upstream dam. After the dam has been constructed, the plywood sheets will be removed.

Sumps will be installed upstream of the isolation dams to promote excavation dewatering. The location of the dewatering sumps will be adjusted as work progresses. Excavation water will be pumped to the water treatment system.

While the pumping system will handle "normal" rainfall events, unusually heavy rainfall and extended rainfall events may require temporary release of storm water to the isolated section of the ditch. In this event, OHM will temporarily cease excavation activities until the ditch section can be isolated again and excavation in the "dry" can continue. This approach should reduce the amount of sediment migrating down stream, which could result in contaminating additional lengths of stream.

Per the HAI plan, the siphon dams will remain in place until it has been demonstrated by visual observation that the water in Williams Ditch is free of visible oil or oil sheen. To allow the work to progress downstream, each siphon dam may have to be reconstructed upstream of the dam's original location OHM will consult with HAI regarding the disposition of the siphon dams.

4.3.2 Sediment Removal

Sediment will be removed from Williams Ditch beginning on the east side of Arco Drive and terminating halfway between sample locations SED-009 and SED-010. Sediment will be removed from the bottom of Williams Ditch (i.e., bottom of bank to bottom of bank). A minimum of 2 feet will be removed in all areas delineated on the HAI plan set. HAI estimates that 2,200 cy of material will be removed. Additional excavation beyond the minimum may be necessary. HAI will confirm the excavation depth using a combination of visual observation and field fluorescence techniques.

An excavator with a reach boom will be the primary excavation equipment. Due to the width of Williams Ditch (i.e., approximately 30 feet), the excavator will be positioned on "swamp" mats to reach the opposite bank. The excavator will load the sediment directly into a 30-ton off-road dump truck. The dump truck will be equipped with a tailgate to contain the wet sediment.

Where possible, the off-road dump truck haul route will parallel Williams Ditch. If field conditions warrant, a temporary haul road will be constructed. The haul road would be approximately 10 feet wide and would consist of geotextile and approximately 6-inches of gravel. The haul road stone material will satisfy the requirements of ODOT Item 203 Embankment Fill or ODOT Item 304 Aggregate Base. Unless KMC prefers to utilize the haul road during future remediation activities, the haul road would be removed and used as fill material under Frenchmens Road.

Through KMC, OHM will request that property owners remove obstructions (e.g., fence, etc.) along Williams Ditch. High visibility construction fence will be used as a temporary substitute for the removed permanent fence. Any permanent fence that is removed by OHM will be replaced at the end of the project.

If obstructions prevent access along the entire length of Williams Ditch, the off road truck will travel through paved areas (i.e., a parking lot and Frenchmens Road). To protect the pavement, a gravel transition (approximately 15 feet by 15 feet) will be constructed next to the pavement. A 1-foot layer of stone will be placed on geotextile. Although the gravel will also serve as a "tire scrubber", the pavement will have to be cleaned.

Special care will be necessary when loading wet sediment. The excavator operator will observe the following procedures to minimize sediment spills:

Carefully load the dump trucks;

Do not overload the dump trucks;

Balance the loads in the dump trucks.

The truck driver and other OHM personnel will monitor the haul route for spilled material. If material is spilled in a public area, the driver will turn on the truck's hazard lights and block the haul route until other OHM personnel arrive with the appropriate spill response supplies.

The City of Toledo Department of Utilities has indicated that backfilling of Williams Ditch to replace excavated materials is not necessary. Should KMC require additional restoration, the City of Toledo's Comprehensive Ditch Plan can be used as a guide.

4.3.2 Sediment Dewatering/Stabilization

Although OHM plans to excavate the Williams Ditch sediments in the dry, laboratory tests conducted by Peoria Disposal Company (PDC) for waste characterization and handling indicate that sediment, without dewatering, will not pass the standard paint filter test. Dewatering, soil bulking, or stabilization will be necessary prior to transportation off-site. Sediments will be allowed to air dry in the ditch and in the soil staging area located north of Frenchmens Road.

If air-drying proves to be insufficient, OHM will bulk the sediment with soil from the lagoon excavation. If soil bulking is impractical, OHM will condition the sediment with a solidification additive (e.g., quick lime, CKD, fly ash, Portland cement, etc.) to meet the specification. If the sediment is too wet to load into the dump truck, OHM may mix a solidification additive to the ditch sediments prior to removal. Although allowing the sediment to drain naturally is preferable; moisture conditioning of the sediments through the addition of solidification agents is likely to be necessary.

To be landfilled at the Peoria Disposal Company (PDC) in Illinois, stabilized materials must demonstrate an unconfined compressive strength of 2 TSF in accordance with the requirements of Title 35 – Illinois Environmental Protection Act, Subpart C 729.310 (b)(3).

4.4 ROAD REMOVAL AND REPLACEMENT

The removal of the immediate source necessitates the removal and replacement of a portion of Frenchmens Road. Contributory drainage areas adjacent to Frenchmens Road will be regraded to ensure that the City of Toledo stormwater control and pipe sizing requirements are met.

The road will be replaced according to the HAI design alignment, width, elevation and construction. The sub-base will be placed and compacted in accordance with the HAI design and the Ohio DOT requirements. As the remediation is not expected to be completed until late fall, the asphalt will be placed during the spring of 1999. A gradual transition will be constructed between the remaining pavement and the sub-base. Pavement along the transition that is damaged during the winter will be replaced.

4.5 FRENCH DRAIN INSTALLATION

OHM will construct a french drain to address the residual contamination that is not removed during the excavation of the time critical action and to provide a mechanism to protect sediment and surface water in Williams Ditch. The work will progress from the western end to the eastern end of the french drain. The western and eastern sections of the french drain will be graded to flow toward a sump near the middle of the french drain. The sump will be constructed as the work progresses from west to east.

The french drain will consist of geotextile, 60 mil HDPE FML or equal, ODOT 57 Natural (non-crushed) washed stone bedding or equal, and a 6-inch perforated CMP or equal.

The sump will consist of a 48-inch diameter HDPE manhole with an automated pumping system. In addition to the 6-inch perforated pipe that discharges into sump, the middle section of the sump is perforated to collect water.

Sheet 4 of the HAI plan set provides the layout and details.

4.6 SOIL LOADOUT

Soil that meets the landfill disposal requirements will be loaded into licensed over the road trucks for transportation to an appropriate disposal facility. A rubber tired loader will load soil from the soil stockpile area into the disposal trucks. The excavator will directly load the remaining soil into the trucks.

Prior to loading the trucks, the truck beds will be lined with plastic. If necessary, each truck will be decontaminated prior to departure. Each truck will be weighed, on non-certified scales, prior to departure to ensure that the trucks meet DOT requirements. If a truck is significantly under the allowable limit, additional soil will be loaded into the truck. The truck's tare weight will be used to determine the approximate load. If necessary, trucks will be weighed prior to loading. Each truck will be covered with a tarp prior to departure for the disposal facility. OHM will inspect the disposal trucks for the necessary paperwork, placards, contamination, etc.

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4.7 TRANSPORTATION AND DISPOSAL

Upon completion of the required analysis, an OHM T&D Coordinator will use the sample analytical data and the pertinent site information to evaluate packaging, transportation, treatment, and disposal options for all of the wastestreams. OHM's T&D coordinators maintain regular communications with licensed and permitted transporters and treatment, storage, and disposal (TSD) facilities. The core objective of this evaluation process is to select the most compliant and cost-effective waste management approach. Only qualified and authorized firms will be allowed to bid.

When the selection has been made, an OHM T&D Coordinator will complete waste profiles and interact with state regulators and TSDF technical staff during the approval process. After approval letters have been obtained from the disposal facilities, the T&D Coordinator will work with the KMC and the project team to efficiently schedule shipments of waste.

The following briefly summarizes the waste approval process:

- Identify individual waste streams
- Submit representative samples of all waste streams for analysis
- Data review and interpretation
- Waste code designation
- Competitive T&D bidding
- Selection of T&D vendors
- Profiling and initiation of the approval process
- Approval
- Scheduling and shipping of waste to TSDFs

HAI estimated that approximately 10,000 to 11,000 CY (i.e., approximately 14,000 to 15,000 tons) of contaminated soil and approximately 2,400 CY (i.e., approximately 3,400 tons) of sediment might have to be disposed off-site. The disposal facility will be Peoria Disposal Company.

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Although the final disposition of each waste material will be determined based on a preliminary visual observation and subsequent analytical testing, the following briefly describes possible waste management approaches for the other site waste streams:

- WWTP Carbon: regeneration or landfill in a Subtitle D TSDF
- WWTP Sand: landfill in a Subtitle D TSDF
- RCP (e.g., Storm water, etc.): landfill in a Subtitle D TSDF
- Steel water pipe: decontaminate, satisfy debris standard, transport to scrap yard or landfill in a Subtitle D TSDF
- Plastic conduit and piping: landfill in a Subtitle D TSDF
- Curb: landfill in a Subtitle D TSDF
- Asphalt concrete: Recycle or landfill in a Subtitle D TSDF
- Wood chips: leave onsite
- Miscellaneous waste (e.g., contaminated PPE, water treatment system bag filters, absorbent pads and booms, contaminated polyethylene plastic, etc.): disposed off-site with the soil.

4.8 BACKFILL

A 60 MIL HDPE flexible membrane liner (FML) will be placed along the sides of the excavation to serve as a barrier. The liner location is illustrated on Sheet 3 of the HAI plan set. The liner will be placed in accordance with HAI's High Density Polyethylene (HDPE) Flexible Membrane Liner specification. Liner sheets will be placed such that there is a minimum overlap of three feet shingled in the direction of flow. The liner will also extend a minimum of one foot along the bottom of the excavation. A geosynthetic boot attached to the conduit where the subsurface utilities penetrate the FML will be used to address potential migration pathways along subsurface utility corridors. HAI will document the liner installation for inclusion in the record drawings.

Backfill placement will commence after a sufficient quantity of liner has been installed. The source of fill material will be from non-contaminated soils that were excavated as well as off-site fill material. OHM will begin the backfill by first placing any clean site soil (i.e., surficial soil, etc.) at the terminal depth of the excavation, followed by clean imported fill material. Off-site material must satisfy the Ohio EPA's Generic Direct Contact Commercial Standard. Where practical, backfill activities will be performed concurrently with the soil excavation activities.

All excavations will be backfilled to the design grade with non-contaminated soil. Backfill will be placed with heavy equipment. In the area of the underground utilities, backfill will be placed in accordance with the requirements of the individual utility. Backfill placed underneath Frenchmens Road will be placed to meet Ohio DOT specifications. The compaction will be accomplished using a compactor in large, open excavations and using the excavator bucket or a hoe pack attached to an excavator in trenches or confined spaces. Any compaction testing will be performed by HAI.

In areas other than under Frenchmens Road or around utilities, backfill will be placed in 12-inch loose lifts. Compaction will be achieved by a minimum of three passes with heavy equipment (e.g., a dozer, etc.). Compaction testing will not be required. Lifts will be compacted at the end of each day to "seal" the lift in the event of rain.

If the excavation is saturated, OHM will employ backfill methods suitable for saturated conditions. OHM will place a porous material (e.g., gravel, etc.) to raise the grade above the water level. This material would be placed until backfill material can be placed in accordance with the requirements.

4.9 SITE RESTORATION

As the remediation is not expected to be completed until late fall, site restoration will be conducted during the spring of 1999. Prior to demobilization, temporary erosion and sediment controls including silt fence, etc will be placed to protect the site during the winter. Affected areas will be revegetated during the spring.

4.10 DEMOBILIZATION

Upon completion of the project objectives and at the direction of KMC, OHM personnel will decontaminate all equipment that contacted site contaminants prior to demobilization. The equipment will be cleaned using high-pressure washing. Residual decontamination wastewater and personal protective equipment (PPE) may be containerized, sampled, and profiled for off-site disposal. Rented equipment will be returned to its respective vendor. All OHM personnel and equipment will be dispatched to their original locations.

Derenzo and Associates, Inc.
Environmental Consultants

August 21, 1998

Mr. Scott Lockhart
HULL & ASSOCIATES, INC.
2726 Monroe Street
Toledo, Ohio 43606

Subject: Results of Ambient Air Monitoring At the Toledo Tie Treatment in Toledo, Ohio

Dear Mr. Lockhart:

Derenzo and Associates, Inc. is forwarding four copies of the report of the *Results of Ambient Air Monitoring At the Toledo Tie Treatment Site in Toledo, Ohio.* The ambient air sampling and analyses were performed to obtain information on the concentrations of air pollutants in the vicinity of the clean-up site of the Toledo Tie Treatment Site.

Please contact me if you have any questions.

Sincerely,

DERENZO AND ASSOCIATES, INC.

Kristopher Simonian
Field Service Manager

enclosure

39201 Schoolcraft, Suite B-10 • Livonia, MI 48150 • (734) 464-3880 • FAX (734) 464-4368

Derenzo and Associates, Inc.
Environmental Consultants

RESULTS
OF
AMBIENT AIR MONITORING
AT THE TOLEDO
TIE TREATMENT SITE
IN
TOLEDO, OHIO

Project No. 9804002

August 21, 1998

39201 Schoolcraft, Suite B-10 • Livonia, MI 48150 • (734) 464-3880 • FAX (734) 464-4368

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Derenzo and Associates, Inc.

RESULTS OF AMBIENT AIR MONITORING AT THE TOLEDO TIE TREATMENT SITE IN TOLEDO, OHIO

1.0 INTRODUCTION

Hull & Associates, Inc. (HAI) retained Derenzo and Associates, Inc. to conduct a short term ambient air monitoring program around the Toledo Tie Treatment Site located on Frenchmens Street in Toledo, Ohio. The monitoring program was used to satisfy requirements of the site's Health and Safety Plan. The monitoring program collected information on the ambient air concentrations of volatile organic compounds (VOC), Polynuclear Aromatic Hydrocarbons (PAH) and Particulate Matter having diameters less than 10 microns (PM-10).

The ambient air sampling was performed on three consecutive dates May 27, 28 and 29, 1998.

Messrs. Kristopher Simonian, Field Services Manager, and Daniel Rochette, Environmental Consultant of Derenzo and Associates, Inc. performed the air sampling for the project. Mr. Scott Lockhart, Senior Project Manager for Hull & Associates, Inc. coordinated the project activities.

2.0 PROGRAM DESIGN

The ambient air monitoring program was conducted to provide background air concentrations of pollutants in the area around the Toledo Tie Treatment Site that had treated wood products with wood preserving materials (mainly creosote). The facility has been closed for a number of years and the area developed into a business park. Analytical data collected by the Ohio Environmental Protection Agency (OEPA) and HAI indicate that the soil and sediment in the area had been impacted by wood preserving materials at various locations at the site.

Hull & Associates, Inc. was retained to develop a site remediation strategy, and as part of the sites Health and Safety Plan, a short term ambient air monitoring program was designed to determine the general ambient air concentrations of the pollutants (VOC, PAH and PM-10) around the clean-up site and to collect data on the ambient air pollutant concentrations during a controlled site excavation. A "worst case" location, based on previous investigations was selected by HAI to represent expected remedial activities.

Four PM-10, PAH (PUF) and VOC samplers were positioned around the perimeter of the clean-up site during the background pollutant sampling events.

Two separate twenty four-hour sampling events were conducted to establish background ambient air pollutant concentrations (VOC, PAH and PM-10).

Positions of the samplers were determined by Derenzo and HAI based on evaluation of the areas past wind direction data, sampling day wind predications and field personnel experience.

During the controlled site excavation only the PAH samplers and the VOC samplers were operated since one objective of the "worse case" sampling was to quantify the potential fence line pollutant concentrations at the clean-up site. One of the PAH and VOC samplers were moved during the site excavation sampling to collect a better down wind sample during the "worse case" testing. In addition, one VOC sample was also taken at the controlled excavation location. This was conducted to correlate readings obtained by hand held instruments for the potential worker exposure concentrations and to identify potential VOC emissions which may occur during excavation.

The controlled site excavation sampling event (worse case) was conducted, to determine if the excavation activities would have an impact on the fence line pollutant concentrations. The VOC and PAH samplers at the perimeter were operated before and after the site excavation for a period of approximately eight-hours. This sampling time frame was used for the following reasons:

1. It was determined that a minimum of eight hours of operation was needed for the PAH samplers to collect a significant amount of air volume to meet the analytical detection limits for the target compounds.

2. The eight hour period would be a similar time frame for the daily site clean-up activities when these activities begin.

The VOC samples were conducted over a one-hour period during the controlled excavation. This timeframe was chosen to show a correlation between the pollutant VOC concentrations collected at the excavation pit and potential pollutant VOC concentrations at the fence line. Given the unknown natures of the contaminants, one-hour was chosen to limit potential exposure and to allow a potential greater number of contaminates to volatilize.

There were no PM-10 samples collected during the controlled site excavation activities, these samplers were used to establish background PM-10 concentrations only.

Meteorological data was collected by Hull & Associates, Inc. during the sampling activities. In addition Metar Weather data recorded hourly, was retrieved from the National Weather Service. Copies of the meteorological data is presented in Appendix A.

Figure 2.0 illustrates the locations of the ambient air samplers during the background and the controlled site excavation activities.

3.0 TESTING AND ANALYSES

The ambient air monitoring and analyses were performed using procedures outlined in United States Environmental Protection Agency (USEPA) reference sampling methods.

USEPA Method TO-13, *The Determination of Benzo(a)pyrene and Other Polynuclear Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatographic (GC) and High Performance Liquid Chromatographic (HPLC) Analysis*, was used to collect ambient air samples for Naphthalene, Phenanthrene, Fluorene, Fluoranthene, Anthracene and Benzo (a)pyrene. These compounds were selected because they are the major components of creosole which was use at the former wood treating facility.

Background samples were collected over a twenty four-hour time sampling period. The site excavation samples were collected over an eight-hour sampling period. During the controlled site excavation the samplers were started before the excavation activities commenced and operated after the excavation period had been stopped. The PAH samples were analyzed by Triangle Laboratories, Inc. in Durham, North Carolina.

USEPA Method TO-14, *Determination of Volatile Organic Compounds in Ambient Air using Summa Passivated Canister Sampling and Gas Chromatographic Analysis*, was used to collect ambient air samples for VOC's. Samples collected in 6 liter SUMMA canisters and were analyzed by gas chromatography/mass spectrometry (GC/MS) procedures.

Background samples were collected over a twenty four-hour time period. The site excavation sample was collected over an one-hour sampling period, while perimeter samplers were started before the excavation activities commenced and stopped after the excavation had been finished. The VOC samples were analyzed by Air Toxics Laboratories, Ltd. in Folsum, California.

USEPA reference method (40 CFR Part 53, Subpart D 52) for collection of particulate matter (i.e., particulates having diameters less than 10 microns, PM_{10}) in ambient air was used to collect background PM-10 samples. Particulates (PM_{10}) on the high-volume sampler filters were analyzed by gravimetric procedures. The PM-10 samplers were operated on two days, the first was obtained the day before the site excavation and the second the day after the site excavation. The PM-10 samples were analyzed by Fibertec Environmental Services. in Holt, Michigan.

Appendix B provides copies of USEPA TO-13 & 14, and PM-10 Reference Sampling Methods.

3.1 Quality Assurance/Quality Control

USEPA Quality Assurance/Quality Control guidelines for ambient air sampling were followed during the short term ambient air monitoring program.

Copies of the laboratory data were sent to Environmental Standards, Inc. for data validation.

Copies of the laboratory results are presented in Appendix C. Individual laboratory Statement of Qualifications are presented in Appendix D.

4.0 RESULTS

4.1 Measured Ambient Air VOC Concentrations

Table 4.1 presents the ambient air pollutant VOC measurements made on each sampling event by monitoring site.

4.2 Measured Ambient Air PAH Concentrations

Table 4.2 presents the ambient air PAH measurements made on each sampling event by monitoring site.

4.3 Measured Ambient Air PM-10 Concentrations

Table 4.3 presents the ambient air PM-10 measurements made on each sampling event by monitoring site.

Report Prepared By:

Kristopher Simonian
Field Service Manager

Report Reviewed By:

Daniel Rochette
Environmental Consultant

Table 4.1 Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Site 1	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Freon 12		<1.0	< 0.96	< 0.96
Freon 114		<1.0	< 0.96	< 0.96
Chloromethane		**1.2**	**1.50**	**1.10**
Vinyl Chloride		<1.0	< 0.96	< 0.96
Bromomethane		<1.0	< 0.96	< 0.96
Chloroethane		<1.0	< 0.96	< 0.96
Freon 11		<1.0	< 0.96	< 0.96
1,1-Dichloroethene		<1.0	< 0.96	< 0.96
Freon 113		<1.0	< 0.96	< 0.96
Methlyene Chloride		<1.0	< 0.96	< 0.96
1,1-Dichloroethane		<1.0	< 0.96	< 0.96
cis-1,2-Dichloroethene		<1.0	< 0.96	< 0.96
Chloroform		<1.0	< 0.96	< 0.96
1,1,1-Trichloroethane		<1.0	< 0.96	< 0.96
Carbon Tetrachloride		<1.0	< 0.96	< 0.96
Benzene		<1.0	< 0.96	< 0.96
1,2-Dichloroethane		<1.0	< 0.96	< 0.96
cis-1,3-Dichloropropene		<1.0	< 0.96	< 0.96
Toluene		**1.7**	**2.20**	**1.90**
trans-1,3-Dichloropropene		<1.0	< 0.96	< 0.96
1,1,2-Trichloroethane		<1.0	< 0.96	< 0.96
Tetrachloroethene		<1.0	< 0.96	< 0.96
Ethylene Dibromide		<1.0	< 0.96	< 0.96
Chlorobenzene		<1.0	< 0.96	< 0.96
Ethyl Benzene		<1.0	< 0.96	< 0.96
m,p-Xylene		<1.0	< 0.96	< 0.96
o-Xylene		<1.0	< 0.96	< 0.96
Styrene		<1.0	< 0.96	< 0.96
1,1,2,2-Tetrachloroethane		<1.0	< 0.96	< 0.96
1,3,5-Trimethylbenzene		<1.0	< 0.96	< 0.96
1,2,4-Trimethylbenzene		<1.0	< 0.96	< 0.96
1,3-Dichlorobenzene		<1.0	< 0.96	< 0.96
1,4-Dichlorobenzene		<1.0	< 0.96	< 0.96
Chlorotoluene		<1.0	< 0.96	< 0.96
1,2-dichlorbenzene		<1.0	< 0.96	< 0.96
1,2,4-Trichlorobenzene		<1.0	< 0.96	< 0.96
Hexachlorobutadiene		<1.0	< 0.96	< 0.96
Propylene		<4.0	< 3.8	< 3.8
1,3-Butadiene		<4.0	< 3.8	< 3.8

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Site 1	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Acetone		**7.5**	**6.5**	**6.5**
Carbon Disulfide		<4.0	< 3.8	< 3.8
2-Propanol		<4.0	< 3.8	< 3.8
trans-1,2-Dichloroehtene		<4.0	< 3.8	< 3.8
Vinyl Acetate		<4.0	< 3.8	< 3.8
2-Butanone (Methyl Ethyl Ketone)		<4.0	< 3.8	< 3.8
Haxane		<4.0	< 3.8	< 3.8
Tetrahydrofuran		<4.0	< 3.8	< 3.8
Cyclohexane		<4.0	< 3.8	< 3.8
1,4-Dioxane		**7.0**	**4.4**	**7.8**
Bromdichloromethane		<4.0	< 3.8	< 3.8
4-Methyl-2-Pentanone		<4.0	< 3.8	< 3.8
2-Hexanone		<4.0	< 3.8	< 3.8
Dibromochloromethane		<4.0	< 3.8	< 3.8
Bromoform		<4.0	< 3.8	< 3.8
4-Ethyltoluene		<4.0	< 3.8	< 3.8
Ethanol		**5.1**	< 3.8	**6.0**
Methyl tert-Butyl Ether		<4.0	< 3.8	< 3.8
Heptane		<4.0	< 3.8	< 3.8

Tentatively Identified Compounds (TICs) (ppbv)	Site 1	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Heptane, 4-ethyl-2,2,6,6-tetramethyl-		6.1		
Heptane, 2,2,4-trimethyl-				
Naphthalene				
Aniline				
Undecane, 3,6-dimethyl				
Undecane, 2,6-dimethyl				3.9
Benzene, (1-methylethenyl)-				
Benzofuran				
Benzene, 1,2,3-trimethyl-				
1H-Indene, 2,3-dihydro-				
Benzene, 1-propenyl-				
Benzene , (1-methyl-1-propenyl)-, (E)-				
Benzofuran, 2-methyl-				
2,3-Dihydro-1-methylindene				
1H-Indene, 2,3-dihydro-2-methyl-				
Benzene, (1-methyl-2-cyclopropen-1-yl)-				
Benzothiophene				

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv) Site 2	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Freon 12	<1.1	< 0.94	**0.89**
Freon 114	<1.1	< 0.00	< 0.84
Chloromethane	**1.2**	**2.90**	**0.95**
Vinyl Chloride	<1.1	< 0.94	< 0.84
Bromomethane	<1.1	< 0.94	< 0.84
Chloroethane	<1.1	< 0.94	< 0.84
Freon 11	<1.1	< 0.94	< 0.84
1,1-Dichloroethene	<1.1	< 0.94	< 0.84
Freon 113	<1.1	< 0.94	< 0.84
Methlyene Chloride	<1.1	< 0.94	< 0.84
1,1-Dichloroethane	<1.1	< 0.94	< 0.84
cis-1,2-Dichloroethene	<1.1	< 0.94	< 0.84
Chloroform	<1.1	< 0.94	< 0.84
1,1,1-Trichloroethane	<1.1	< 0.94	< 0.84
Carbon Tetrachloride	<1.1	< 0.94	< 0.84
Benzene	**1.6**	< 0.94	< 0.84
1,2-Dichloroethane	<1.1	< 0.94	< 0.84
cis-1,3-Dichloropropene	<1.1	< 0.94	< 0.84
Toluene	**3.4**	**3.50**	**2.30**
trans-1,3-Dichloropropene	<1.1	< 0.94	< 0.84
1,1,2-Trichloroethane	<1.1	< 0.94	< 0.84
Tetrachloroethene	<1.1	< 0.94	< 0.84
Ethylene Dibromide	<1.1	< 0.94	< 0.84
Chlorobenzene	<1.1	< 0.94	< 0.84
Ethyl Benzene	<1.1	< 0.94	< 0.84
m,p-Xylene	<1.1	**0.99**	**0.91**
o-Xylene	<1.1	< 0.94	< 0.84
Styrene	<1.1	< 0.94	< 0.84
1,1,2,2-Tetrachloroethane	<1.1	< 0.94	< 0.84
1,3,5-Trimethylbenzene	<1.1	< 0.94	< 0.84
1,2,4-Trimethylbenzene	<1.1	< 0.94	< 0.84
1,3-Dichlorobenzene	<1.1	< 0.94	< 0.84
1,4-Dichlorobenzene	<1.1	< 0.94	< 0.84
Chlorotoluene	<1.1	< 0.94	< 0.84
1,2-dichlorbenzene	<1.1	< 0.94	< 0.84
1,2,4-Trichlorobenzene	<1.1	< 0.94	< 0.84
Hexachlorobutadiene	<1.1	< 0.94	< 0.84
Propylene	<4.3	< 3.7	**3.4**
1,3-Butadiene	<4.3	< 3.7	< 3.4

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Site 2	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Acetone		**8.6**	**10.0**	**6.3**
Carbon Disulfide		<4.3	< 3.7	< 3.4
2-Propanol		<4.3	< 3.7	< 3.4
trans-1,2-Dichloroehtene		<4.3	< 3.7	< 3.4
Vinyl Acetate		<4.3	< 3.7	< 3.4
2-Butanone (Methyl Ethyl Ketone)		<4.3	< 3.7	< 3.4
Haxane		<4.3	< 3.7	< 3.4
Tetrahydrofuran		<4.3	< 3.7	< 3.4
Cyclohexane		<4.3	< 3.7	< 3.4
1,4-Dioxane		**8.2**	< 3.7	< 3.4
Bromdichloromethane		<4.3	< 3.7	< 3.4
4-Methyl-2-Pentanone		<4.3	< 3.7	< 3.4
2-Hexanone		<4.3	< 3.7	< 3.4
Dibromochloromethane		<4.3	< 3.7	< 3.4
Bromoform		<4.3	< 3.7	< 3.4
4-Ethyltoluene		<4.3	< 3.7	< 3.4
Ethanol		**6.2**	< 3.7	**5.3**
Methyl tert-Butyl Ether		<4.3	< 3.7	< 3.4
Heptane		<4.3	< 3.7	< 3.4

Tentatively Identified Compounds (TICs) (ppbv)	Site 2	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Heptane, 4-ethyl-2,2,6,6-tetramethyl-				
Heptane, 2,2,4-trimethyl-				6.6
Naphthalene				
Aniline		6.0		
Undecane, 3,6-dimethyl				
Undecane, 2,6-dimethyl				
Benzene, (1-methylethenyl)-				
Benzofuran				
Benzene, 1,2,3-trimethyl-				
1H-Indene, 2,3-dihydro-				
Benzene, 1-propenyl-				
Benzene , (1-methyl-1-propenyl)-, (E)-				
Benzofuran, 2-methyl-				
2,3-Dihydro-1-methylindene				
1H-Indene, 2,3-dihydro-2-methyl-				
Benzene, (1-methyl-2-cyclopropen-1-yl)-				
Benzothiophene				

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Site 3	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Freon 12		<0.94	< 0.98	< 0.98
Freon 114		<0.94	< 0.98	< 0.98
Chloromethane		**1.1**	**1.30**	**1.20**
Vinyl Chloride		<0.94	< 0.98	< 0.98
Bromomethane		<0.94	< 0.98	< 0.98
Chloroethane		<0.94	< 0.98	< 0.98
Freon 11		<0.94	< 0.98	< 0.98
1,1-Dichloroethene		<0.94	< 0.98	< 0.98
Freon 113		<0.94	< 0.98	< 0.98
Methlyene Chloride		<0.94	< 0.98	< 0.98
1,1-Dichloroethane		<0.94	< 0.98	< 0.98
cis-1,2-Dichloroethene		<0.94	< 0.98	< 0.98
Chloroform		<0.94	< 0.98	< 0.98
1,1,1-Trichloroethane		<0.94	< 0.98	< 0.98
Carbon Tetrachloride		<0.94	< 0.98	< 0.98
Benzene		**1.1**	< 0.98	**1.50**
1,2-Dichloroethane		<0.94	< 0.98	< 0.98
cis-1,3-Dichloropropene		<0.94	< 0.98	< 0.98
Toluene		**1.7**	**1.70**	**1.70**
trans-1,3-Dichloropropene		<0.94	< 0.98	< 0.98
1,1,2-Trichloroethane		<0.94	< 0.98	< 0.98
Tetrachloroethene		<0.94	< 0.98	< 0.98
Ethylene Dibromide		<0.94	< 0.98	< 0.98
Chlorobenzene		<0.94	< 0.98	**1.10**
Ethyl Benzene		<0.94	< 0.98	< 0.98
m,p-Xylene		<0.94	< 0.98	< 0.98
o-Xylene		<0.94	< 0.98	< 0.98
Styrene		<0.94	< 0.98	< 0.98
1,1,2,2-Tetrachloroethane		<0.94	< 0.98	< 0.98
1,3,5-Trimethylbenzene		<0.94	< 0.98	< 0.98
1,2,4-Trimethylbenzene		<0.94	< 0.98	< 0.98
1,3-Dichlorobenzene		<0.94	< 0.98	< 0.98
1,4-Dichlorobenzene		<0.94	< 0.98	< 0.98
Chlorotoluene		<0.94	< 0.98	< 0.98
1,2-dichlorbenzene		<0.94	< 0.98	< 0.98
1,2,4-Trichlorobenzene		<0.94	< 0.98	< 0.98
Hexachlorobutadiene		<0.94	< 0.98	< 0.98
Propylene		<3.7	< 3.9	< 3.9
1,3-Butadiene		<3.7	< 3.9	< 3.9

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Site 3	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Acetone		**6.0**	**5.7**	**8.3**
Carbon Disulfide		<3.7	< 3.9	< 3.9
2-Propanol		<3.7	< 3.9	< 3.9
trans-1,2-Dichloroehtene		<3.7	< 3.9	< 3.9
Vinyl Acetate		<3.7	< 3.9	< 3.9
2-Butanone (Methyl Ethyl Ketone)		<3.7	< 3.9	< 3.9
Haxane		<3.7	< 3.9	< 3.9
Tetrahydrofuran		<3.7	< 3.9	< 3.9
Cyclohexane		<3.7	< 3.9	< 3.9
1,4-Dioxane		**6.9**	**17.0**	< 3.9
Bromdichloromethane		<3.7	< 3.9	< 3.9
4-Methyl-2-Pentanone		<3.7	< 3.9	< 3.9
2-Hexanone		<3.7	< 3.9	< 3.9
Dibromochloromethane		<3.7	< 3.9	< 3.9
Bromoform		<3.7	< 3.9	< 3.9
4-Ethyltoluene		<3.7	< 3.9	< 3.9
Ethanol		**7.6**	< 3.9	**8.4**
Methyl tert-Butyl Ether		<3.7	< 3.9	< 3.9
Heptane		<3.7	< 3.9	< 3.9

Tentatively Identified Compounds (TICs) (ppbv)	Site 3	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Heptane, 4-ethyl-2,2,6,6-tetramethyl-				
Heptane, 2,2,4-trimethyl-				
Naphthalene				
Aniline				
Undecane, 3,6-dimethyl		6.8		
Undecane, 2,6-dimethyl				
Benzene, (1-methylethenyl)-				
Benzofuran				
Benzene, 1,2,3-trimethyl-				
1H-Indene, 2,3-dihydro-				
Benzene, 1-propenyl-				
Benzene , (1-methyl-1-propenyl)-, (E)-				
Benzofuran, 2-methyl-				
2,3-Dihydro-1-methylindene				
1H-Indene, 2,3-dihydro-2-methyl-				
Benzene, (1-methyl-2-cyclopropen-1-yl)-				
Benzothiophene				

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Site 4	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Freon 12		< 1.0	< 0.98	<1.1
Freon 114		< 1.0	< 0.98	<1.1
Chloromethane		**1.1**	**1.10**	**1.7**
Vinyl Chloride		< 1.0	< 0.98	<1.1
Bromomethane		< 1.0	< 0.98	<1.1
Chloroethane		< 1.0	< 0.98	<1.1
Freon 11		< 1.0	< 0.98	<1.1
1,1-Dichloroethene		< 1.0	< 0.98	<1.1
Freon 113		< 1.0	< 0.98	<1.1
Methlyene Chloride		< 1.0	< 0.98	<1.1
1,1-Dichloroethane		< 1.0	< 0.98	<1.1
cis-1,2-Dichloroethene		< 1.0	< 0.98	<1.1
Chloroform		< 1.0	< 0.98	<1.1
1,1,1-Trichloroethane		< 1.0	< 0.98	<1.1
Carbon Tetrachloride		< 1.0	< 0.98	<1.1
Benzene		< 1.0	< 0.98	**3.0**
1,2-Dichloroethane		< 1.0	< 0.98	<1.1
cis-1,3-Dichloropropene		< 1.0	< 0.98	<1.1
Toluene		**1.0**	**1.80**	**1.6**
trans-1,3-Dichloropropene		< 1.0	< 0.98	<1.1
1,1,2-Trichloroethane		< 1.0	< 0.98	<1.1
Tetrachloroethene		< 1.0	< 0.98	<1.1
Ethylene Dibromide		< 1.0	< 0.98	<1.1
Chlorobenzene		< 1.0	< 0.98	<1.1
Ethyl Benzene		< 1.0	< 0.98	<1.1
m,p-Xylene		< 1.0	< 0.98	<1.1
o-Xylene		< 1.0	< 0.98	<1.1
Styrene		< 1.0	< 0.98	<1.1
1,1,2,2-Tetrachloroethane		< 1.0	< 0.98	<1.1
1,3,5-Trimethylbenzene		< 1.0	< 0.98	<1.1
1,2,4-Trimethylbenzene		< 1.0	< 0.98	<1.1
1,3-Dichlorobenzene		< 1.0	< 0.98	<1.1
1,4-Dichlorobenzene		< 1.0	< 0.98	<1.1
Chlorotoluene		< 1.0	< 0.98	<1.1
1,2-dichlorbenzene		< 1.0	< 0.98	<1.1
1,2,4-Trichlorobenzene		< 1.0	< 0.98	<1.1
Hexachlorobutadiene		< 1.0	< 0.98	<1.1
Propylene		< 4.1	< 4.0	<4.3
1,3-Butadiene		< 4.1	< 4.0	<4.3

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Site 4	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Acetone		**7.0**	**5.4**	**11.0**
Carbon Disulfide		< 4.1	< 4.0	<4.3
2-Propanol		< 4.1	< 4.0	<4.3
trans-1,2-Dichloroehtene		< 4.1	< 4.0	<4.3
Vinyl Acetate		< 4.1	< 4.0	<4.3
2-Butanone (Methyl Ethyl Ketone)		< 4.1	< 4.0	<4.3
Haxane		< 4.1	< 4.0	<4.3
Tetrahydrofuran		< 4.1	< 4.0	<4.3
Cyclohexane		< 4.1	< 4.0	<4.3
1,4-Dioxane		**4.2**	**20.0**	**8.2**
Bromdichloromethane		< 4.1	< 4.0	<4.3
4-Methyl-2-Pentanone		< 4.1	< 4.0	<4.3
2-Hexanone		< 4.1	< 4.0	<4.3
Dibromochloromethane		< 4.1	< 4.0	<4.3
Bromoform		< 4.1	< 4.0	<4.3
4-Ethyltoluene		< 4.1	< 4.0	<4.3
Ethanol		**8.2**	< 4.0	**6.2**
Methyl tert-Butyl Ether		< 4.1	< 4.0	<4.3
Heptane		< 4.1	< 4.0	<4.3

Tentatively Identified Compounds (TICs) (ppbv)	Site 4	27-May 24-hour	28-May 8-Hour	29-May 24-Hour
Heptane, 4-ethyl-2,2,6,6-tetramethyl-				
Heptane, 2,2,4-trimethyl-				
Naphthalene				
Aniline				
Undecane, 3,6-dimethyl				
Undecane, 2,6-dimethyl				
Benzene, (1-methylethenyl)-				6.2
Benzofuran				
Benzene, 1,2,3-trimethyl-				
1H-Indene, 2,3-dihydro-				
Benzene, 1-propenyl-				
Benzene , (1-methyl-1-propenyl)-, (E)-				
Benzofuran, 2-methyl-				
2,3-Dihydro-1-methylindene				
1H-Indene, 2,3-dihydro-2-methyl-				
Benzene, (1-methyl-2-cyclopropen-1-yl)-				
Benzothiophene				

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Pit	28-May 1-Hour
Freon 12		< 1.0
Freon 114		< 1.0
Chloromethane		**1.7**
Vinyl Chloride		< 1.0
Bromomethane		< 1.0
Chloroethane		< 1.0
Freon 11		< 1.0
1,1-Dichloroethene		< 1.0
Freon 113		< 1.0
Methlyene Chloride		< 1.0
1,1-Dichloroethane		< 1.0
cis-1,2-Dichloroethene		< 1.0
Chloroform		< 1.0
1,1,1-Trichloroethane		< 1.0
Carbon Tetrachloride		< 1.0
Benzene		**19.0**
1,2-Dichloroethane		< 1.0
cis-1,3-Dichloropropene		< 1.0
Toluene		**27.0**
trans-1,3-Dichloropropene		< 1.0
1,1,2-Trichloroethane		< 1.0
Tetrachloroethene		< 1.0
Ethylene Dibromide		< 1.0
Chlorobenzene		< 1.0
Ethyl Benzene		**39.0**
m,p-Xylene		**51.0**
o-Xylene		**21.0**
Styrene		**2.9**
1,1,2,2-Tetrachloroethane		< 1.0
1,3,5-Trimethylbenzene		**10.0**
1,2,4-Trimethylbenzene		**19.0**
1,3-Dichlorobenzene		< 1.0
1,4-Dichlorobenzene		< 1.0
Chlorotoluene		< 1.0
1,2-dichlorbenzene		< 1.0
1,2,4-Trichlorobenzene		< 1.0
Hexachlorobutadiene		< 1.0
Propylene		< 4.0
1,3-Butadiene		< 4.0

Table 4.1 (Cont.) Measured Ambient Air VOC Concentrations

VOC Compound (ppbv)	Pit	28-May 1-Hour
Acetone		**8.7**
Carbon Disulfide		< 4.0
2-Propanol		< 4.0
trans-1,2-Dichloroehtene		< 4.0
Vinyl Acetate		< 4.0
2-Butanone (Methyl Ethyl Ketone)		< 4.0
Haxane		< 4.0
Tetrahydrofuran		< 4.0
Cyclohexane		< 4.0
1,4-Dioxane		**7.4**
Bromdichloromethane		< 4.0
4-Methyl-2-Pentanone		< 4.0
2-Hexanone		< 4.0
Dibromochloromethane		< 4.0
Bromoform		< 4.0
4-Ethyltoluene		**17.0**
Ethanol		< 4.0
Methyl tert-Butyl Ether		< 4.0
Heptane		< 4.0

Tentatively Identified Compounds (TICs) (ppbv)	Pit	28-May 1-Hour
Heptane, 4-ethyl-2,2,6,6-tetramethyl-		
Heptane, 2,2,4-trimethyl-		
Naphthalene		290.0
Aniline		
Undecane, 3,6-dimethyl		
Undecane, 2,6-dimethyl		
Benzene, (1-methylethenyl)-		
Benzofuran		7.2
Benzene, 1,2,3-trimethyl-		7.8
1H-Indene, 2,3-dihydro-		76.0
Benzene, 1-propenyl-		35.0
Benzene , (1-methyl-1-propenyl)-, (E)-		7.2
Benzofuran, 2-methyl-		7.1
2,3-Dihydro-1-methylindene		5.6
1H-Indene, 2,3-dihydro-2-methyl-		6.7
Benzene, (1-methyl-2-cyclopropen-1-yl)-		5.1
Benzothiophene		19.0

Summary of Ambient Air Sampling Events, May 1998
Kerr McGee Chemical, LLC
Toledo Tie Treatment Site

	Baseline Emissions Samples[1]					Controlled Excavation Test Samples[2]					Baseline Emission Samples[3]				
	Site 1	Site 2	Site 3	Site 4		Site 1	Pit	Site 2	Site 4	Site 3	Site 1	Site 2	Site 3	Site 4	
	27-May	27-May	27-May	27-May		28-May	28-May	28-May	28-May	28-May	29-May	29-May	29-May	29-May	
Measured Ambient Air Contaminants	24-Hr	24-Hr	24-Hr	24-Hr	Ave	8-Hr	1-Hr	8-Hr	8-Hr	8-Hr	24-Hr	24-Hr	24-Hr	24-Hr	Ave
Freon 12												0.89			0.9
Chloromethane	1.2	1.2	1.1	1.1	1.2	1.5	1.7	2.9	1.1	1.3	1.1	0.95	1.2	1.7	1.2
Benzene		1.6	1.1		1.4		19						1.5	3	2.3
Toluene	1.7	3.4	1.7	1.0	2.0	2.2	27	3.5	1.8	1.7	1.9	2.3	1.7	1.6	1.9
Chlorobenzene													1.1		
Ethyl Benzene							39								
Xylenes							72	0.99				0.91			0.9
Styrene							2.9								
1,3,5-Trimethylbenzene							10								
1,2,4-Trimethylbenzene							19								
Propylene												3.4			
Acetone	7.5	8.6	6.0	7	7.3	6.5	8.7	10	5.4	5.7	6.5	6.3	8.3	11	8.0
1,4-dioxane	7	8.2	6.9	4.2	6.6	4.4	7.4		20	17	7.8			8.2	8.0
4-ethyltoluene							17								
Ethanol	5.1	6.2	7.6	8.2	6.8						6	5.3	8.4	6.2	6.5
Heptane, 4-ethyl-2,2,6,6-tetramethyl-	6.1				6.1										
Heptane, 2,2,4-trimethyl-												6.6			6.6
Naphthalene[4]	0.0055	0.013	0.011	0.004	0.008	0.022	290	0.018	0.004	0.006	0.017	0.037	0.050	0.009	0.028
Aniline		6													
Undecane, 2,6-dimethyl											3.9				3.9
Benzene, (1-methylethenyl)-														6.2	6.2
Benzofuran							7.2								
Benzene, 1,2,3-trimethyl-							7.8								
1H-Indene, 2,3-dihydro							76								
Benzene, 1-propenyl-							35								
Benzene, (1-methyl-1-propenyl)-, (E)-							7.2								
Benzofuran, 2-methyl-							7.1								
2,3-dihydro-1-methylindene							5.6								
1H-Indene, 2,3-dihydro-2-methyl-							6.7								
Benzene, (1-methyl-2-cyclopropen-1-yl)-							5.1								
Benzithiophene							19								
Fluorene[4]	0.001	0.004	0.004	0.003	0.0032047	0.003		0.010	0.001	0.003	0.003	0.006	0.011	0.003	0.005697
Phenanthrene[4]	0.007	0.011	0.013	0.012	0.011	0.005		0.029	0.009	0.008	0.009	0.019	0.032	0.009	0.017
Anthracene[4]								0.001				0.001	0.001		0.0013
Fluoranthene[4]	0.001	0.001	0.001	0.001	0.0012	0.001		0.004	0.002	0.001	0.001	0.002	0.004	0.001	0.0020

Footnotes
1 Prevailing Wind Direction 230° from METAR Data at Toledo, Ohio Station
2 Prevailing Wind Direction 250° from METAR Data at Toledo, Ohio Station
3 Prevailing Wind Direction 260° from METAR Data at Toledo, Ohio Station
4 Estimated ppb's from Ambient Air PAH Samples with a standard temp of 70°F

Table 4.2 Measured Ambient Air PAH Concentrations

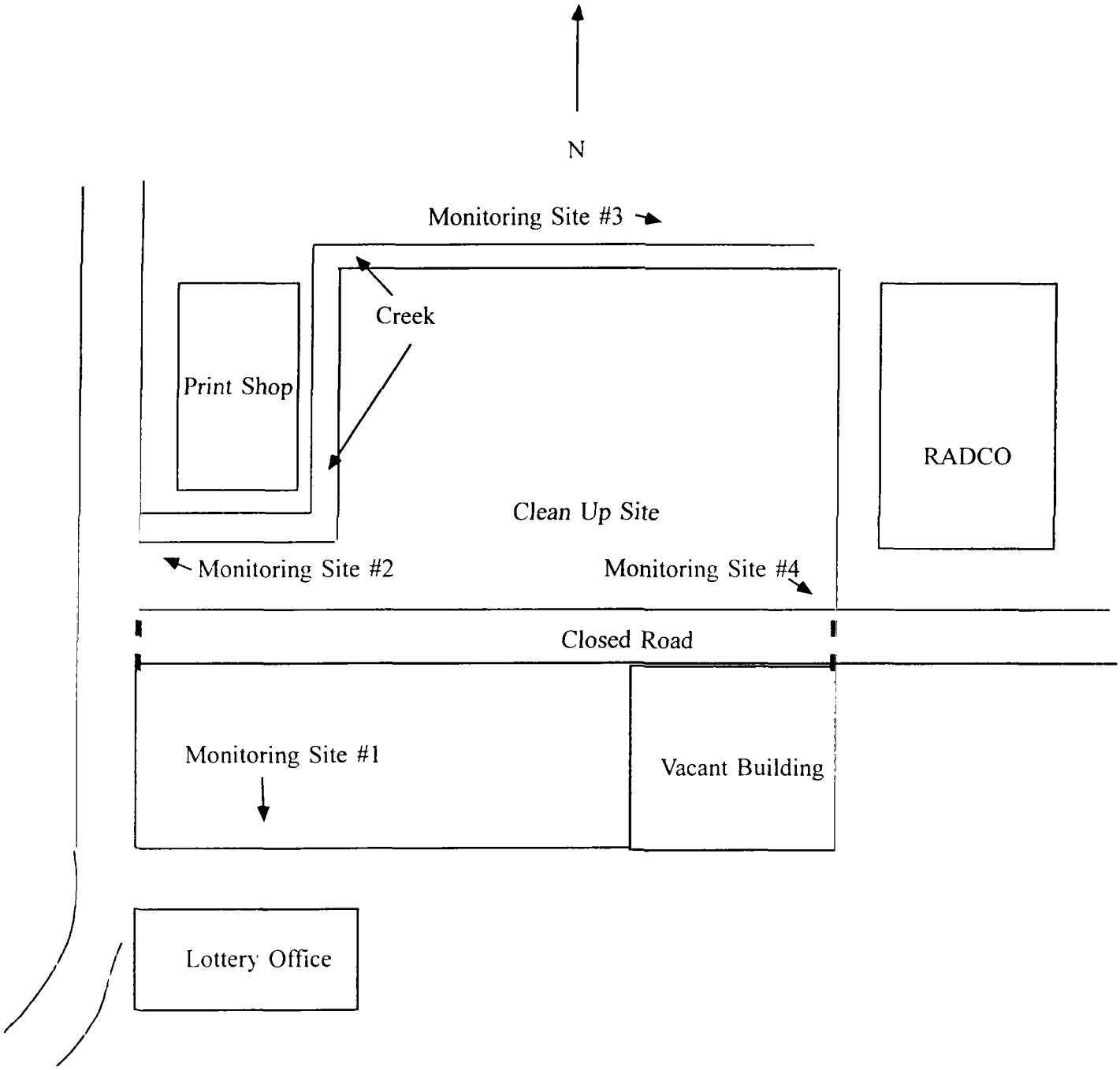
Compound	Site Number	27-May 24-Hour (ug/m3)	28-May 8-Hour (ug/m3)	29-May 24-Hour (ug/m3)
Naphthalene	1	0.03	0.12	0.09
Fluorene		0.01	0.02	0.02
Phenanthrene		0.05	0.04	0.07
Anthracene		<DL	<DL	<DL
Fluoranthene		0.01	0.01	0.01
Benzo(a)pyrene		<DL	<DL	<DL
Naphthalene	2	0.07	0.10	0.20
Fluorene		0.03	0.07	0.04
Phenanthrene		0.08	0.22	0.14
Anthracene		<DL	0.01	0.01
Fluoranthene		0.01	0.03	0.02
Benzo(a)pyrene		<DL	<DL	<DL
Naphthalene	3	0.06	0.03	0.27
Fluorene		0.03	0.02	0.08
Phenanthrene		0.10	0.06	0.24
Anthracene		<DL	<DL	0.01
Fluoranthene		0.01	0.01	0.03
Benzo(a)pyrene		<DL	<DL	<DL
Naphthalene	4	0.02	0.02	0.05
Fluorene		0.02	0.01	0.02
Phenanthrene		0.09	0.07	0.07
Anthracene		<DL	<DL	<DL
Fluoranthene		0.01	0.02	0.01
Benzo(a)pyrene		<DL	<DL	<DL

<DL=Less than Detection Limit

Table 4.3 Measured Ambient Air PM-10 Concentrations

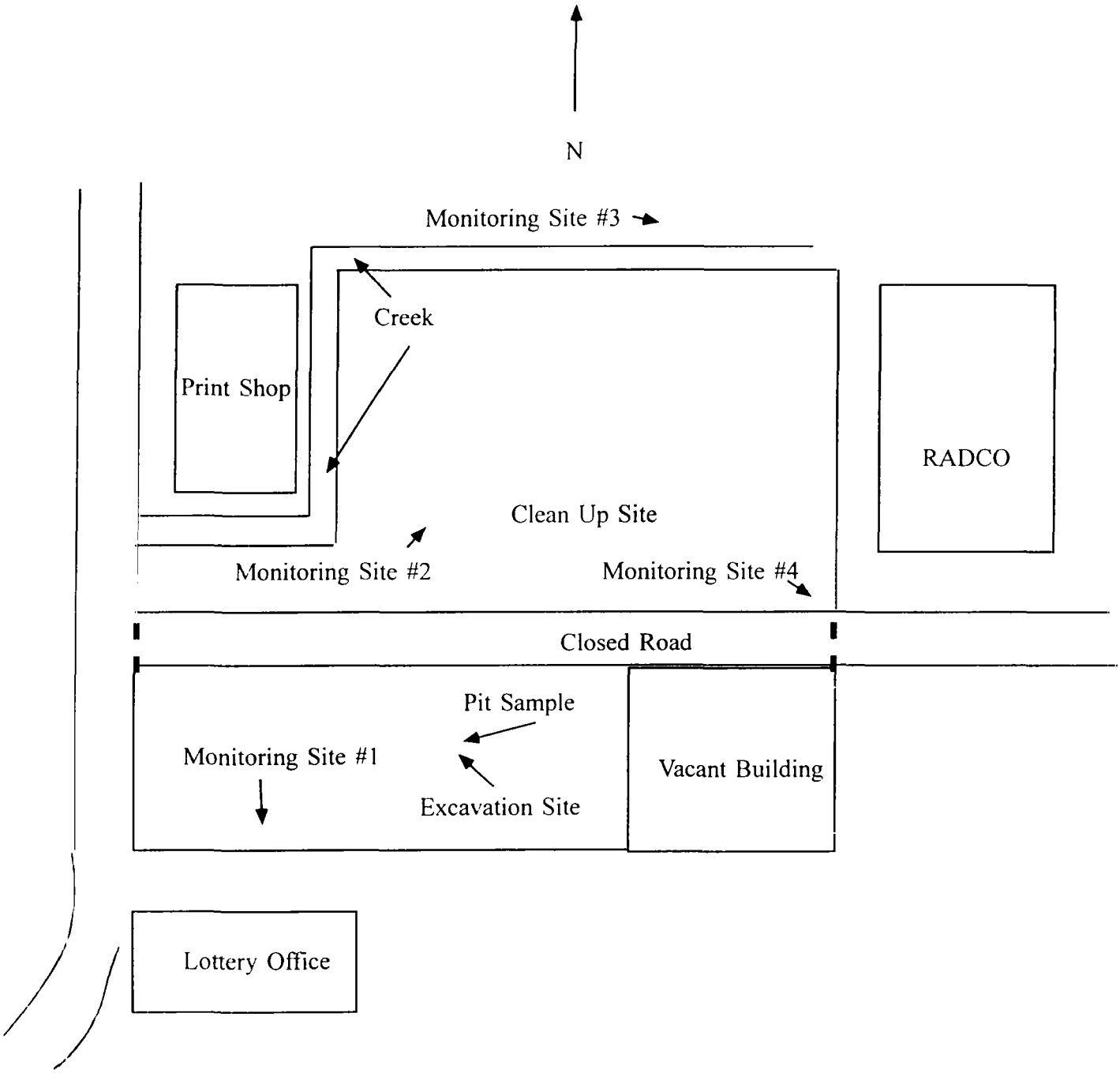
Site Number	27-May (ug/m3)	29-May (ug/m3)
1	61	49
2	68	45
3	54	41
4	63	43

Figure 2.0 Background Sampler Locations



BACKGROUND MONITORING SITE LOCATIONS

Figure 2.0 (Cont.) Excavation Sampler Locations



EXCAVATION MONITORING SITE LOCATIONS

APPENDIX A

METEROLOCIAL DATA

Kerr-McGee Toledo Tie Treatment Site
METAR Weather Data Observations

Date	Time	Met Stn.	Wind °	Wind Speed (m/s)	Prevailing Visibility (SM)	Cloud Amount	Cloud Height (ft)	Air Temp. (°C)	Dew-point Temp. (°C)	Altimeter inHg	Stability	
				Background Sampling Event								
5/27/98	11 52 27	KTOL	VRB	4	10	CLR		26.1	10.1	30.04	B	2
5/27/98	12 52.35	KTOL	220	8	10	FEW	75	26.7	9.4	30.03	C	3
5/27/98	13.52 37	KTOL	VRB	4	10	FEW	80	26.7	9.4	30.01	B	2
5/27/98	14:52:51	KTOL	150	4	10	FEW	90	26.1	10.1	30	B	2
5/27/98	15:52:26	KTOL	VRB	4	10	SCT	85	27.2	8 9	29.99	C	3
5/27/98	16:52.33	KTOL	VRB	3	10	CLR		27.2	8.9	29.99	B	2
5/27/98	17:52 27	KTOL	170	3	10	CLR		26.7	9.4	29.98	B	2
5/27/98	18:52:42	KTOL	210	5	10	SCT	90	25	12.2	29.98	C	3
5/27/98	19:52.27	KTOL	0	0	10	FEW	85	22.2	11.1	29.98	F	6
5/27/98	20:52:27	KTOL	140	4	10	CLR		20.6	11.1	29.99	F	6
5/27/98	21:52:33	KTOL	130	4	10	BKN	90	19.4	11.1	30	D	4
5/27/98	22.52.27	KTOL	150	3	10	FEW	90	18.3	10.6	30	E	5
5/27/98	23:52:33	KTOL	160	4	10	BKN	80	18.3	10.6	30	D	4
5/28/98	0 52 27	KTOL	0	0	10	CLR		17.2	10.6	30	F	6
5/28/98	1 52:35	KTOL	0	0	10	CLR		15	11.7	30	F	6
5/28/98	2 53:11	KTOL	0	0	10	CLR		14.4	11.1	30	F	6
5/28/98	3 52:27	KTOL	VRB	3	9	SCT	75	15	11.1	30.01	D	4
5/28/98	4:52:35	KTOL	200	3	7	SCT	75	15.6	11.7	30.01	D	4
5/28/98	5:52:27	KTOL	240	4	4	CLR		15	12.2	30.02	E	5
5/28/98	6:52:35	KTOL	200	4	4	CLR		18.3	13.9	30.02	E	5
5/28/98	7:52:27	KTOL	230	7	4	CLR		21.7	16.7	30.03	D	4
5/28/98	8 52 35	KTOL	240	8	4	CLR		23.9	17.8	30.03	D	4
5/28/98	9:52:27	KTOL	230	16	6	CLR		26.1	17.8	30.02	D	4
5/28/98	10.52 33	KTOL	220	15	6	FEW	41	26.7	17.2	30.02	D	4
5/28/98	11:52.28	KTOL	240	14	6	FEW	45	27.2	16.7	30.03	D	4
5/28/98	12.52:36	KTOL	250	17	9	FEW	55	28.9	16.7	30.01	D	4
		Mean Wind Speed		5.4								4.0
				Controlled Excavation Pit Sampling Event								
5/28/98	13 53:37	KTOL	230	13	10	CLR		28.9	16.7	29.95	C	
5/28/98	14 52:27	KTOL	230	15	10	FEW	60	30	16 1	29.94	D	
5/28/98	15 52 27	KTOL	230	13	10	CLR		29.4	16.1	29.92	D	
5/28/98	16 52:35	KTOL	220	12	10	CLR		30	16.7	30	D	
5/28/98	17 52:27	KTOL	250	12	10	BKN	70	30	16.1	29.91	D	
5/28/98	18 52.27	KTOL	240	11	10	CLR		29.4	17.2	29.91	D	
5/28/98	19 52:35	KTOL	210	7	10	CLR		27.8	17.2	29.91	D	
5/28/98	20 52:27	KTOL	210	7	10	CLR		26.1	17.2	29.91	D	
5/28/98	21:52:33	KTOL	220	9	10	CLR		25.6	17.2	29.93	D	
		Mean Wind Speed		11.0								
				Background Sampling Event								
5/29/98	9 52.27	KTOL	250	10	10	BKN	110	25.6	15	29.97	D	4
5/29/98	10 52:35	KTOL	260	15	10	CLR		27.8	15	29.96	C	3
5/29/98	11.53:11	KTOL	270	12	10	CLR		29.4	15	29.95	C	3
5/29/98	12 52 28	KTOL	280	16	10	CLR		31.1	14 4	29.93	C	3
5/29/98	13:52:35	KTOL	270	15	10	CLR		32.2	13.9	29.92	C	3
5/29/98	14:52.27	KTOL	290	18	10	CLR		31.7	14.4	29.9	D	4
5/29/98	15:52:36	KTOL	300	18	10	CLR		31.7	15	29.9	D	4
5/29/98	16.53:11	KTOL	290	15	10	CLR		31.1	14.4	29.89	D	4
5/29/98	17:52:27	KTOL	290	14	10	CLR		29.4	14.4	29.9	D	4
5/29/98	18:52:35	KTOL	290	8	10	SCT	120	27.2	15.6	29.91	D	4
5/29/98	19.52:27	KTOL	310	6	10	BKN	110	26.1	15 6	29.92	D	4
5/29/98	20:52:35	KTOL	260	4	10	SCT	110	25	15.6	29.94	D	4
5/29/98	21:52:27	KTOL	200	3	10	CLR		20.6	16 1	29.96	E	5
5/29/98	22:52:35	KTOL	0	0	10	CLR		18.3	16.1	29.98	F	6
5/29/98	23:52:27	KTOL	0	0	10	CLR		17.2	16.1	29 99	F	6
5/30/98	0.52.35	KTOL	0	0	10	CLR		16.1	15.6	29 99	F	6
5/30/98	1 53.11	KTOL	0	0	10	CLR		16.1	15.6	29.98	F	6
5/30/98	2:52:27	KTOL	0	0	8	CLR		16.1	15.6	29.9	F	6
5/30/98	3.52:33	KTOL	60	6	10	CLR		15	14.4	30	D	4
5/30/98	4 52:27	KTOL	80	6	10	CLR		15.6	13.9	30.01	D	4
5/30/98	5.52:33	KTOL	70	5	10	CLR		15.6	13.9	30.03	D	4
5/30/98	6:52:27	KTOL	110	6	10	FEW	95	17.2	13.3	30.05	D	4
5/30/98	7 53 11	KTOL	90	9	10	CLR		18 3	12.2	30.03	D	4
5/30/98	8:52 27	KTOL	110	8	10	CLR		20	11.7	30.03	D	4
5/30/98	9:52.33	KTOL	80	7	10	CLR		21.7	12.2	30.02	D	4
5/30/98	10:52:27	KTOL	120	6	10	CLR		23.3	13.3	30	C	3
5/30/98	11 52:27	KTOL	120	6	10	CLR		26.1	15	29.99	C	3
		Mean Wind Speed		7.9								4.2

SUMMARY TABLE OF WEATHER MONITORING DATA
KERR-McGEE CHEMICAL, LLC
TOLEDO TIE TREATMENT SITE

DATE	TIME	TEMPERATURE (degrees F)	BAROMETRIC PRESSURE (in. hg.)	WIND SPEED (mph)	WIND DIRECTION	RELATIVE HUMIDITY (%)
5/27/98	11:15	88.7	29.32	0-1	SW	N/A
5/27/98	15:51	86.5	29.51	0	S-SE	21
5/27/98	16:09	86.6	29.49	0	S	27
5/28/98	7:49	71.0	29.55	0-1	S	65
5/28/98	13:10	86.1	29.53	12	W-SW	32
5/28/98	14:01	83.7	29.51	5	S-SW	38
5/28/98	14:19	83.0	29.5	8	S-SW	38
5/28/98	14:29	82.7	29.48	20	S	37
5/28/98	14:44	85.5	29.46	11	SW	30
5/28/98	14:54	85.7	29.46	12	S	35
5/28/98	15:41	86.3	29.44	13	S	29
5/28/98	18:15	85.6	29.42	6	S	43
5/29/98	10:11	84.0	29.49	4	S-SW	47
5/29/98	10:30	81.0	29.49	4	S-SW	45
5/29/98	12:17	83.0	29.47	5-7	S-SW	46
5/29/98	12:32	83.2	29.48	5-6	S-SW	45
5/29/98	20:17	78.0	29.42	3-5	WSW-SSW	48
5/29/98	21:05	77.0	29.4	4-7	S-SW	48
5/30/98	9:02	70.0	29.53	0-1	E-NE	68
5/30/98	9:50	70.0	29.52	0-4	N-NW	50
5/30/98	10:21	73.0	29.51	2	N-NW	48
5/30/98	10:50	76.0	29.51	2	N-NE	45
6/1/98	9:18	62.0	29.43	2	N-NE	51
6/1/98	10:10	58.0	29.44	4-5	N-NW	40
6/1/98	10:23	62.0	29.43	1	N-NW	38
6/1/98	10:54	66.0	29.43	0	NW	37
6/1/98	11:25	70.0	29.42	4	E	35
6/1/98	11:42	66.0	29.41	0-2	W	33
6/1/98	12:32	66.0	29.38	0-3	W	33
6/1/98	13:32	69.0	29.37	5-6	S-SE	19
6/1/98	14:28	72.0	29.32	4	S-SW	31
6/1/98	14:56	74.0	29.31	2-3	S-SW	34
6/1/98	15:15	72.0	29.32	7-8	S-SW	22

APPENDIX B

USEPA TO-13 & 14
AND
PM-10 REFERENCE SAMPLING METHODS

METHOD TO-13

THE DETERMINATION OF BENZO(a)PYRENE [B(a)P] AND OTHER POLYNUCLEAR AROMATIC HYDROCARBONS (PAHs) IN AMBIENT AIR USING GAS CHROMATOGRAPHIC (GC) AND HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC (HPLC) ANALYSIS

1. Scope

1.1 Polynuclear 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, a reliable sampling and analytical method has been established. This document describes a sampling and analysis procedure for B[a]P and other PAHs involving a combination quartz filter/adsorbent cartridge with subsequent analysis by gas chromatography (GC) with flame ionization (FI) and mass spectrometry (MS) detection (GC/FI and GC/MS) or high resolution liquid chromatography (HPLC). The analytical methods are a modification 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 a carcinogenic constituent of coal tar (1-7). Fluorescent methods are capable of measuring subnanogram quantities of PAHs, but tend to be fairly non-selective. The normal spectra obtained tended to be intense and lacked resolution. Efforts to overcome this difficulty led to the use of ultraviolet (UV) absorption spectroscopy 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 (8). 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 spectra overlap required complete separation of sample components to insure accurate measurement of component levels. Hence, the use of UV absorption coupled with pre-speciation involving LC and TLC and fluorescence spectroscopy has declined and is now being replaced with the more sensitive high performance liquid chromatography (9) with UV/fluorescence detection and highly sensitive and specific gas chromatography with either flame

ionization detector or coupled with mass spectroscopy (10-11).

1.3 The choice of GC and HPLC as the recommended procedures for analysis of B[a]P and other PAHs are influenced by their sensitivity and selectivity, along with their ability to analyze complex samples. This method provides for both GC and HPLC approaches to the determination of B[a]P and other PAHs in the extracted sample.

1.4 The analytical methodology is well defined, but the sampling procedures can reduce the validity of the analytical results. Recent studies (12-15) 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 down stream of the filter to the back-up adsorbent. A wide variety of adsorbents such as Tenax GC, XAD-2 resin and polyurethane foam (PUF) have been used to sample B[a]P and other PAH vapors. All adsorbents have demonstrated high collection efficiency for B[a]P in particular. In general, XAD-2 resin has a higher collection efficiency (16-17) for volatile PAHs than PUF, as well as a higher retention efficiency. However, PUF cartridges are easier to handle in the field and maintain better flow characteristics during sampling. Likewise, PUF has demonstrated its capability in sampling organochlorine pesticides and polychlorinated biphenyls (Compendium Methods TO4 and TO10 respectively), and polychlorinated dibenzo-p-dioxins (Compendium Method TO9). However, PUF has demonstrated a lower recovery efficiency and storage capability for naphthalene and B[a]P, respectively, than XAD-2. There have been no significant losses of PAHs, up to 30 days of storage at 0°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 covers both the utilization of XAD-2 and PUF as the adsorbent to address post-collection volatilization problems associated with B[a]P and other reactive PAHs.

1.5 This method covers the determination of B[a]P specifically by both GC and HPLC and enables the qualitative and quantitative analysis of the other PAHs. They are:

Acenaphthene	Benzo(k)fluoranthene
Acenaphthylene	Chrysene
Anthracene	Dibenzo(a,h)anthracene
Benzo(a)anthracene	Fluoranthene
Benzo(a)pyrene	Fluorene

Benzo(b)fluoranthene	Indeno(1,2,3-cd)pyrene
Benzo(e)pyrene	Naphthalene
Benzo(g,h,i)perylene	Phenanthrene
	Pyrene

The GC and HPLC methods are applicable to the determination of PAHs compounds involving two-member rings or higher. Nitro-PAHs have not been fully evaluated using this procedure; therefore, they are not included in this method. When either of the methods are used to analyze unfamiliar samples for any or all of the compounds listed above, compound identification should be supported by both techniques.

1.6 With careful attention to reagent purity and optimized analytical conditions, the detection limits for GC and HPLC methods range from 1 ng to 10 pg which represents detection of B[a]P and other PAHs in filtered air at levels below 100 pg/m^3. To obtain this detection limit, at least 100 m^3 of air must be sampled.

2. Applicable Documents

2.1 ASTM Standards

2.1.1 Method D1356 — Definitions of Terms Relating to Atmospheric Sampling and Analysis.

2.1.2 Method E260 — Recommended Practice for General Gas Chromatography Procedures.

2.1.3 Method E355 — Practice for Gas Chromatography Terms and Relationships.

2.1.4 Method E682 — Practice for Liquid Chromatography Terms and Relationships.

2.1.5 Method D-1605-60 — Standard Recommended Practices for Sampling Atmospheres for Analysis of Gases and Vapors.

2.2 Other Documents

2.2.1 Existing Procedures (18-25)

2.2.2 Ambient Air Studies (26-28)

2.2.3 U.S. EPA Technical Assistance Document (29-32)

2.2.4 General Metal Works Operating Procedures for Model PS-1 Sampler, General Metal Works, Inc., Village of Cleves, Ohio.

3. Summary of Method

3.1 Filters and adsorbent cartridges (containing XAD-2 or PUF) are cleaned in solvents and vacuum-dried. The filters and adsorbent cartridges are stored in screw-capped jars wrapped in aluminum foil (or otherwise protected from light) before careful installation on a modified high volume sampler.

3.2 Approximately 325 m^3 of ambient air is drawn through the filter and adsorbent cartridge using a calibrated General Metal Works Model PS-1 Sampler, or equivalent (breakthrough has not shown to be a problem with sampling volumes of 325 m^3).

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

3.4 The filters and adsorbent cartridge are extracted by Soxhlet extraction with appropriate solvent. The extract is concentrated by Kuderna-Danish (K-D) evaporator, followed by silica gel clean-up using column chromatography to remove potential interferences prior to analysis.

3.5 The eluent is further concentrated by K-D evaporator, then analyzed by either gas chromatography equipped with FI or MS detection or high performance liquid chromatography (HPLC). The analytical system is verified to be operating properly and calibrated with five concentration calibration solutions, each analyzed in triplicate.

3.6 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 necessary, 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.

3.7 The samples and the blanks are analyzed and used (along with the amount of air sampled) to calculate the concentration of B[a]P in ambient air.

3.8 Other PAHs can be determined both qualitatively and quantitatively through optimization of the GC or HPLC procedures.

4. Significance

4.1 Several documents have been published which describe sampling and analytical approaches for benzo[a]pyrene and other PAHs, as outlined in Section 2.2. The attractive features of these methods have been combined in this procedure. This method has been validated in the laboratory; however, one must use caution when employing it for specific applications.

4.2 The relatively low level of B[a]P and other PAHs in the environment requires use of high volume (~6.7 cfm) sampling techniques 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 adsorbent cartridge which provide for efficient collection of most PAHs.

5. Definitions

Definitions used in this document and in any user-prepared standard operating procedures (SOPs) should be consistent with ASTM Methods D1356, D1605-60, E260, and E255. All abbreviations and symbols are defined within this document at point of use.

5.1 Sampling efficiency (SE) — ability of the sampling medium to trap vapors of interest. %SE is the percentage of the analyte of interest collected and retained by the sampling medium when it is introduced as a vapor in air or nitrogen 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.

5.2 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.

5.3 High Performance Liquid Chromatography — an analytical method based on separation of compounds of a liquid mixture through a liquid chromatographic column and measuring the separated components with a suitable detector.

5.4 Gradient elution — defined as increasing the strength of the mobile phase during a chromatographic analysis. The net effect of gradient elution is to shorten the retention time of compounds strongly retained on the analytical column. Gradient elution may be stepwise or continuous.

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 (KD) apparatus is a system for concentrating materials dissolved in volatile solvents.

5.7 Reverse phase liquid chromatography — reverse phase liquid chromatography involves a non-polar absorbent (C-18,ODS) coupled with a polar solvent to separate non-polar compounds.

5.8 Guard column — guard columns in HPLC are usually short (5cm) columns attached after the injection port and before the analytical column to prevent particles and strongly retained compounds from accumulating on the analytical column. The guard column should always be the same stationary phase as the analytical column and is used to extend the life of the analytical

column.

5.9 MS-SIM — the GC is coupled to a select ion mode (SIM) detector where the instrument is programmed to acquire data for only the target compounds and to disregard all others. This is performed using SIM coupled to retention time discriminators. The SIM analysis procedure provides quantitative results.

5.10 Sublimation — Sublimation is 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.

5.11 Surrogate standard — A surrogate standard is a chemically inert compound (not expected to occur in the environmental sample) which 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.

5.12 Retention time window — Retention time window is determined for each analyte of interest and is the time from injection to elution of a specific chemical from a chromatographic column. The window is determined by three injections of a single component standard over a 72-hr period as plus or minus three times the standard deviation of the absolute retention time for that analyte.

6. Interferences

6.1 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.

6.1.1 Glassware must be scrupulously cleaned (33). Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinsing with tap water and reagent water. It should then be drained dry, 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.

6.1.2 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.

6.1.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. Additional clean-up by column chromatography may be required (see Section 12.4).

6.2 The extent of interferences that may be encountered using liquid chromatographic techniques has not been fully assessed. Although GC and HPLC 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 or HPLC 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 is run for each batch of reagents used to determine if reagents are contaminant-free.

6.3 Although HPLC separations have been improved by recent advances in column technology and instrumentation, problems may occur with baseline noise, baseline drift, peak resolution and changes in sensitivity. Problems affecting overall system performance can arise (34). The user is encouraged to develop a standard operating procedure (SOP) manual specific for his laboratory to minimize problems affecting overall system performance.

6.4 Concern during sample transport and analysis is mentioned. Heat, ozone, NO_2 and ultraviolet (UV) light may cause sample degradation. These problems should be addressed as part of the user prepared standard operating procedure manual. Where possible, incandescent or UV-shield fluorescent lighting should be used during analysis.

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 data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified for the analyst (35-37).

7.2 Benzo[a]pyrene 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 whoever 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 (Table 1.0 and Figure 1.0).

7.3 Treat all selective polynuclear aromatic hydrocarbons 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. Regularly check counter tops and equipment with "black light" for fluorescence as an indicator of contamination.

7.4 Because the sampling configuration (filter and backup adsorbent) has demonstrated greater than 95% collection efficiency for targeted PAHs, no field recovery evaluation will occur as part of this procedure.

8. Apparatus

8.1 Sample Collection

8.1.1 General Metal Works (GMW) Model PS-1 Sampler, or equivalent [General Metal Works, Inc., 145 South Miami Ave., Village of Cleves, Ohio, 45002, (800-543-7412)].

8.1.2 At least two Model PS-1 sample cartridges and filters assembled for PS-1 sampler.

8.1.3 GMW Model PS-1 calibrator and associated equipment — General Metal Works, Inc., Model GMW-40, 145 South Miami Ave., Village of Cleves, Ohio, 45002, (800-543-7412).

8.1.4 Ice chest — to store samples at 0°C after collection.

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

8.1.6 Airtight, labeled screw-capped container sample cartridges (wide mouth, preferably glass with Teflon

seal or other noncontaminating seals) to hold filter and adsorbent cartridge during transport to analytical laboratory.

8.1.7 Portable Tripod Sampler (optional) — user prepared (38).

8.2 Sample Clean-up and Concentration

8.2.1 Soxhlet extractors capable of extracting GMW Model PS-1 filter and adsorbent cartridges (2.3" x 5" length), 500 mL flask, and condenser.

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.

8.2.3 Glass vial, 40 mL.

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, especially glassware that is reused, 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 amount of tap water containing detergent. Rinse with copious amount of tap water and several portions of distilled water. Drain, dry, and heat in a muffle furnace at 400°C for 2 to 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 Polyester gloves for handling cartridges and filters.

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

8.2.7 Stainless steel Teflon® coated spatulas and spoons.

8.2.8 Kuderna-Danish (KD) apparatus — 500 mL evaporation flask (Kontes K-570001-500 or equivalent), 10 mL graduated concentrator tubes (Knotes K-570050-1025 or equivalent) with ground-glass stoppers, and 3-ball

macro Snyder Column (Kontes K-5700010500, K-50300-0121, and K-569001-219, or equivalent).

8.2.9 Adsorption columns for column chromatography — 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 with chambers operating at 0°C and 4°C.

8.2.14 Boiling chips — solvent extracted, 10/40 mesh silicon carbide or equivalent.

8.2.15 Water bath — heated, with concentric ring cover, capable of temperature control (±5°C).

8.2.16 Vortex evaporator (optional).

8.3 Sample Analysis

8.3.1 Gas Chromatography with Flame Ionization Detection (FID).

8.3.1.1 Gas chromatography: Analytical system complete with gas chromatography suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak areas and/or peak heights is recommended.

8.3.1.2 Packed Column: 1.8-m x 2-mm I.D. glass column packed with 3% OV-17 on Chromosorb W-AW-DMCS (100/120 mesh) or equivalent (Supelco Inc., Supelco Park, Bellefonte, Pa. Supelco SPB-5).

8.3.1.3 Capillary Column: 30-m x 0.25-mm ID fused silica column coated with 0.25 u thickness 5% phenyl, 90% methyl siloxane (Supelco Inc., Supelco Park, Bellefonte, Pa.).

8.3.1.4 Detector: Flame Ionization (FI)

8.3.2 Gas Chromatograph with Mass Spectroscopy Detection Coupled with Data Processing System (GC/MS/DS).

8.3.2.1 The GC must be equipped for temperature programming, and all required accessories must be available, including syringes, gases, and a capillary column. The GC 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 1-3 uL injection volume is used consistently. With some GC injection ports, however, 1 uL injections may produce some improvement in precision and chromatographic separation. A 1 uL injection volume may be used if adequate sensitivity and precision can be achieved. [NOTE: If 1 uL is used as the injection volume, the injection volumes for all extracts, blanks, calibration solutions and performance check samples <u>must</u> be 1 uL.]

8.3.2.2 Gas Chromatograph-Mass Spectrometer Interface. The gas chromatograph is usually coupled directly to the mass spectrometer 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. 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. Graphic ferrules should be avoided in the GC injection area since they may adsorb PAHs. Vespel® or equivalent ferrules are recommended.

8.3.2.3 Mass Spectrometer. The static resolution of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer should be operated in the selected ion mode (SIM) with a total cycle time (including voltage reset time) of one second or less (Section 14.2).

8.3.2.4 Mass spectrometer: Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets all of the criteria (Section 14.5.1).

8.3.2.5 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.

8.3.2.6 GC Column. A fused silica column (50-m x 0.25-mm I.D.) HP Ultra #2 crosslinked 5% phenyl methylsilicone, 0.25 um film thickness (Hewlett-Packard Co., Crystal Lake, IL) is utilized to separate individual PAHs. Other columns may be used for determination of PAHs. Minimum acceptance criteria must be determined as per Section 14.2. 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.

8.3.2.7 Balance — Mettler balance or equivalent.

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

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

8.3.3 High Performance Liquid Chromatography (HPLC) System.

8.3.3.1 Gradient HPLC system — Consisting of acetonitrile and water phase reservoirs; mixing chamber; a high pressure pump; an injection valve (automatic sampler with an optional 25 uL loop injector); a Vydac C-18 bon and a data system or strip chart recorder. A Spectra Physics 8100 liquid chromatograph multi-microprocessor controlled, with ternary gradient pumping system, constant flow, autosampler injector (10 uL injection loop), and column oven (optional).

8.3.3.2 Guard column — 5-cm guard column pack with Vydac reverse phase C-18 material.

8.3.3.3 Reverse phase analytical column — Vydac or equivalent, C-18 bonded phase RP column (The Separation Group, P.O. Box 867, Hesperia, Ca., 92345), 4.6-mm x 25-cm, 5-micron particle diameter.

8.3.3.4 LS-4 fluorescence spectrometer, Perkin Elmer, separate excitation and emission, monochromator positioned by separate microprocessor-controlled flow cell and wavelength programming ability (optional).

8.3.3.5 Ultraviolet/visible detector, Spectra Physics 8440, deuterium Lamp, capable of programmable wavelengths (optional).

8.3.3.6 Dual channel Spectra Physics 4200 Computing Integrator, measures peak areas and retention times from recorded chromatographs. IBM PC XT will Spectra Physics Labnet system for data collection and storage (optional).

9. Reagents and Materials

9.1 Sample Collection

9.1.1 Acid-washed quartz fiber filter — 105 mm micro quartz fiber binderless filter (General Metal Works, Inc., Cat. No. GMW QMA-4, 145 South Miami Ave., Village of Cleves, Ohio, 45002 [800-543-7412] or Supelco Inc., Cat. No. 1-62, Supelco Park, Bellefonte, PA, 16823-0048).

9.1.2 Polyurethane foam (PUF) — 3 inch thick sheet stock, polyether type (density 0.022 g/cm^3) used in furniture upholstering (General Metal Works, Inc., Cat. No. PS-1-16, 145 South Miami Ave., Village of Cleves, Ohio, 45002 [800-543-7412] or Supelco Inc., Cat. No. 1-63, Supelco Park, Bellefonte, PA, 16823-0048).

9.1.3 XAD-2 resin — Supelco Inc., Cat. No. 2-02-79, Supelco Park, Bellefonte, PA, 16823-0048).

9.1.4 Hexane-rinsed aluminum foil — best source.

9.1.5 Hexane-reagent grade, best source.

9.2 Sample Clean-up and Concentration

9.2.1 Soxhlet Extraction

9.2.1.1 Methylene chloride — chromatographic grade, glass-distilled, best source.

9.2.1.2 Sodium sulfate, anhydrous — (ACS) granular anhydrous (purified by washing with methylene chloride followed by heating at 400°C for 4 hrs in a shallow tray).

9.2.1.3 Boiling chips — solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

9.2.1.4 Nitrogen — high purity grade, best source.

9.2.1.5 Ether — chromatographic grade, glass-distilled, best source.

9.2.1.6 Hexane — chromatographic grade, glass-distilled, best source.

9.2.1.7 Dibromobiphenyl — chromatographic grade, best source. Used for internal standard.

9.2.1.8 Decafluorobiphenyl — chromatographic grade, best source. Used for internal standard.

9.2.2 Solvent Exchange

9.2.2.1 Cyclohexane — chromatographic grade, glass-distilled, best source.

9.2.3 Column Clean-up

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9.2.3.1 Silica gel — high purity grade, type 60, 70-230 mesh; extracted in a Soxhlet apparatus with methylene chloride for 6 hours (minimum of 3 cycles per hour) and activated by heating in a foil-covered glass container for 24 hours at 130°C.

9.2.3.2 Sodium sulfate, anhydrous — (ACS) granular anhydrous (See Section 9.2.1.2).

9.2.3.3 Pentane — chromatographic grade, glass-distilled, best source.

Lobar Prepacked Column

9.2.3.4 Silica gel lobar prepacked column — E. Merck, Darmstadt, Germany [Size A(240-10) Lichroprep Si (40-63 um)].

9.2.3.5 Precolumn containing sodium sulfate — American Chemical Society (ACS) granular anhydrous (purified by washing with methylene chloride followed by heating at 400°C for 4 hours in a shallow tray).

9.2.3.6 Hexane — chromatographic grade, glass-distilled, best source.

9.2.3.7 Methylene chloride — chromatographic grade, glass-distilled, best source

9.2.3.8 Methanol — chromatographic grade, glass-distilled, best source.

9.3 Sample Analysis

9.3.1 Gas Chromatography Detection

9.3.1.1 Gas cylinders of hydrogen and helium — ultra high purity, best source.

9.3.1.2 Combustion air — ultra high purity, best source.

9.3.1.3 Zero air — Zero air may be obtained from a cylinder or zero-grade compressed air scrubbed with Drierite® or silica gel and 5A molecular sieve or activated charcoal, or by catalytic cleanup of ambient air. All zero air should be passed through a liquid argon cold trap for final cleanup.

9.3.1.4 Chromatographic-grade stainless steel tubing and stainless steel plumbing fittings — for interconnections. [Alltech Applied Science, 2051 Waukegan Road, Deerfield, IL, 60015, (312) 948-8600]. [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.*]

9.3.1.5 Native and isotopically labeled PAHs isomers for calibration and spiking standards-[Cambridge Isotopes, 20 Commerce Way, Woburn, MA, 01801 (617-547-1818)]. Suggested isotopically labeled PAH isomers are:

- perylene - d_{12}
- chrysene - d_{12}
- acenaphthene - d_{10}
- naphthalene - d_8
- phenanthrene - d_{10}

9.3.1.6 Decafluorotriphenylphosphine (DFTPP) — best source, used for tuning GC/MS.

9.3.2 High Performance Liquid Chromatography Detection

9.3.2.1 Acetonitrile — chromatographic grade, glass-distilled, best source.

9.3.2.2 Boiling chips — solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

9.3.2.3 Water — HPLC Grade. Water must not have an interference that is observed at the minimum detectable limit (MDL) of each parameter of interest.

9.3.2.4 Decafluorobiphenyl — HPLC grade, best source (used for internal standard).

10. Preparation of Sample Filter and Adsorbent

10.1 Sampling Head Configuration

10.1.1 The sampling head (Figure 2) consist of a filter holder compartment followed by a glass cartridge for retaining the adsorbent.

10.1.2 Before field use, both the filter and adsorbent must be cleaned to <10 ng/apparatus of B[a]P or other PAHs.

10.2 Glass Fiber Filter Preparation

10.2.1 The glass fiber filters are baked at 600°C for five hours before use. To insure acceptable filters, they are extracted with methylene chloride in a Soxhlet apparatus, similar to the cleaning of the XAD-2 resin (see Section 10.3).

10.2.2 The extract is concentrated and analyzed by either GC or HPLC. A filter blank of <10 ng/filter of B[a]P or other PAHs is considered acceptable for field use.

10.3 XAD-2 Adsorbent Preparation

10.3.1 For initial cleanup of the XAD-2, a batch of XAD-2 (approximately 60 grams) is placed in a Soxhlet apparatus [see Figure 3(a)] and extracted with methylene chloride for 16 hours at approximately 4 cycles per hour.

10.3.2 At the end of the initial Soxhlet extraction, the spent methylene chloride is discarded and replaced with fresh reagent. The XAD-2 resin is once again extracted for 16 hours at approximately 4 cycles per hour.

10.3.3 The XAD-2 resin is removed from the Soxhlet apparatus, placed in a vacuum oven connected to an ultra-purge nitrogen gas stream and dried at room temperature for approximately 2-4 hours (until no solvent odor is detected).

10.3.4 A nickel screen (mesh size 200/200) is fitted to the bottom of a hexane-rinsed glass cartridge to retain the XAD-2 resin.

10.3.5 The Soxhlet extracted/vacuum dried XAD-2 resin is placed into the sampling cartridge (using polyester gloves) to a depth of approximately 2 inches. This should require approximately 55 grams of adsorbent.

10.3.6 The glass module containing the XAD-2 adsorbent is wrapped with hexane-rinsed aluminum foil, placed in a labeled container and tightly sealed with Teflon® tape.

10.3.7 At least one assemble cartridge from each batch must be analyzed, as a laboratory blank, using the procedures described in Section 13, before the batch is considered acceptable for field use. A blank of <10 ng/cartridge of B[a]P on other PNA's is considered acceptable.

10.4 PUF Sampling Cartridge Preparation

10.4.1 The PUF adsorbent is a polyether-type polyurethane foam (density No. 3014 or 0.0225 g/cm^3) used for furniture upholstery.

10.4.2 The PUF insets 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 1). During cutting, the die is rotated at high speed (e.g., in a drill press) and continuously lubricated with water.

10.4.3 For initial cleanup, the PUF plug is placed in a Soxhlet apparatus [see Figure 3(a)] and extracted with acetone for 14-24 hours at approximately 4 cycles per hour. [Note: When cartridges are reused, 5% diethyl ether in n-hexane can be used as the cleanup solvent.]

10.4.4 The extracted PUF is placed in a vacuum oven connected to a water aspirator and dried at room temperature for approximately 2-4 hours (until no solvent odor is detected).

10.4.5 The PUF is placed into the glass sampling cartridge using polyester gloves. The module is wrapped with hexane-rinsed aluminum foil, placed in a labeled container, and tightly sealed.

10.4.6 At least one assembled cartridge from each batch must be analyzed, as a laboratory blank, using the

procedures described in Section 13, before the batch is considered acceptable for field use. A blank level of <10 ng/plug for single compounds is considered to be acceptable.

11. Sample Collection

11.1 Description of Sampling Apparatus

11.1.1 The entire sampling system can be a modification of a traditional high volume sampler (see Figure 4) or a portable sampler (see Figure 5). A unit specifically designed for this method is commercially available (Model PS-1 — General Metal Works, Inc., Village of Cleves, Ohio).

11.1.2 The sampling module consists of a glass sampling cartridge and an air-tight metal cartridge holder, as outlined in Section 10.1. The adsorbent (XAD-2 or PUF) is retained in the glass sampling cartridge.

11.2 Calibration of Sampling System

Each sampler is to 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%; 4) when a different sample collection media, other than that which the sampler was originally calibrated to, will be used for sampling; or 5) at the frequency specified in the user Standard Operating Procedure (SOP) manual in which the samplers are utilized.

11.2.1 Calibration of Flow Rate Transfer Standard

Calibration of the modified high volume air sampler in the field is performed using a calibrated orifice flow rate transfer standard. The flow rate transfer standard must be certified in the laboratory against a positive displacement rootsmeter (see Figure 6). Once certified, the recertification is performed rather infrequently if the orifice is protected from damage. Recertification of the orifice flow rate transfer standard is performed once per year utilizing a set of five (5) multihole resistance plates. [Note: The 5 multihole resistance plates are used to change the flow through the orifice so that several points can be obtained for the orifice calibration curve.]

11.2.1.1 Record the room temperature (t_1 in °C) and barometric pressure (P_b in mm Hg) on Orifice Calibration Data Sheet (see Figure 7). 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 6. 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, then there is a leak in the system or in the tape. 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 counterclockwise. 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 bottom of 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 hi-vol motor and let it run for five minutes to set the motor brushes.

11.2.1.8 Record both manometer readings-orifice water manometer (ΔH) and rootsmeter mercury manometer (ΔP). [Note: ΔH is the sum of the difference from zero (0) of

the two column heights.]

11.2.1.9 Record the time, in minutes, required to pass a known volume of air (approximately 200-300 ft^3 of air for each resistance plate) through the rootsmeter by using the rootsmeter's digital volume dial and a stopwatch.

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 11.2.1.10.

11.2.1.13 Repeat for each resistance plate. Note results on Orifice Calibration Data Sheet (see Figure 7). 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 with the following formula and record the standard volume on the Orifice Calibration Data Sheet:

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

where: V_{std} = standard volum

V_m = actual volume measured by the roots

P_1 = barometric pressure during calibration (mm Hg).

ΔP = differential pressure at inlet to volume meter (mm Hg).

P_{std} = 760 mm Hg.

T_{std} = 298 K.

T_1 = 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:

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

where: V_{std} = standard volum

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 value $\sqrt{\Delta H(P_1/P_{std})(298/T_1)}$ for each standard flow rate.

11.2.1.19 Plot each $\sqrt{\Delta H(P_1/P_{std})(298/T_1)}$ value (y-axis) versus its associated standar

11.2.1.20 Commercially available calibrator kits are available [General Metal Works, Inc., Model GMW-40, 145 South Miami Avenue, Village of Cleves, Ohio, 45002 (1-800-543-7412)].

11.2.2 Calibration of The High Volume Sampling System Utilizing Calibrated Multi-point Flow Rate Transfer Standard

11.2.2.1 The airflow through the sampling system can be monitored by a venturi/magnehelic assembly, as illustrated in Figure 4 or by a u-tube assembly connected to the high volume portable design as illustrated in Figure 5. The field sampling system must be audited every six months using a flow rate transfer standard, as described in the U.S. EPA High Volume Sampling Method, 40 CFR 50, Appendix B. A single-point calibration must be performed before and after each sample collection, using a transfer standard calibrated as described in Section 11.2.1.

11.2.2.2 Prior to initial multi-point calibration, a "dummy" adsorbent cartridge and filter

are placed in the sampling head and the sampling motor is activated. The flow control valve is fully opened and the voltage variator is adjusted so that a sample flow rate corresponding to 110% of the desired flow rate (typically 0.20-0.28 m^3/min) is indicated on the Magnehelic gauge (based on the previously obtained multi-point calibration curve). The motor is allowed to warm up for 10 minutes and then the flow control valve is adjusted to achieve the desired flow rate. Turn off the sampler. The ambient temperature and barometric pressure should be recorded on the Field Calibration Data Sheet (Figure 9).

11.2.2.3 The flow rate transfer standard is placed on the sampling head, and a manometer is connected to the tap on the transfer standard using a length of tubing. Properly align the retaining rings with filter holder and secure by tightening the three screw clamps. Set the zero level of the manometer. Attach the magnehelic gage to the sampler venturi quick release connections. Adjust the zero (if needed) using the zero adjust screw on the face of the gage.

11.2.2.4 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.

11.2.2.5 Adjust the flow control valve and repeat until six or seven uniformally spaced magnehelic readings are recorded spanning the range of approximately 40-70 in. Record the readings on the Field Calibration Data Sheet (see Figure 9). [Note: Use of some filter/sorbent media

combinations may restrict the airflow resulting in a maximum magnehelic reading of 60 in. or less. In such cases, a variable transformer should be placed in-line between the 110 volt power source and the sampler so that the line voltage can be increased sufficiently to obtain a maximum magnehelic reading approaching 70 in.].

11.2.2.6 Adjust the orifice manometer reading for standard temperature and pressure using the following equation:

$$X = \sqrt{\Delta H \frac{P_a}{P_{std}} \frac{T_{std}}{T_a}}$$

where: X = adjusted manom

ΔH = observed manometer reading (in wate

P_a = current barometric pressure (mm Hg).

P_{std} = 760 mm Hg.

T_a = current temperature (K), (K = °C + 273).

T_{std} = standard temperature (298 K).

11.2.2.7 Calculate the standard flow rate for each corrected manometer reading by the following equation:

$$Q_{std} = \frac{X - b}{M}$$

where: Q_{std} = standard flow

M = slope of flow rate transfer standar

X = corrected manometer reading from 11.2.2.6 (in water).

b = intercept of flow rate transfer standard calibration curve.

11.2.2.8 Adjust the magnehelic gage readings to standard temperature and pressure using the following equation:

$$M_{std} = \sqrt{\frac{(M)(P_a)}{P_{std}} \frac{T_{std}}{T_a}}$$

where: M_{std} = adjusted magne

M = observed magnehelic reading (inches

P_a = ambient atmospheric pressure (mm Hg).

P_{std} = standard pressure (760 mm Hg).

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

T_{std} = standard temperature (298 K).

11.2.2.9 Plot each M_{std} value (y-axis) versus its associated Q_{std} standard (x-axis) on arithmetic graph paper. Draw a line of best fit between the individual plotted points. This is the calibration curve for the venturi. Retain with sampler.

11.2.2.10 Record the corresponding Q_{std} for each M_{std} under Q_{std} column on Field Calibration Data Sheet, Figure 9.

11.2.3 Single-point Audit of The High Volume Sampling System Utilizing Calibrated Flow Rate Transfer Standard

11.2.3.1 A single point flow audit check is performed before and after each sampling period utilizing the Calibration Flow Rate Transfer Standard (Section 11.2.1).

11.2.3.2 Prior to single point audit, a "dummy" adsorbent cartridge and filter are placed in the sampling head and the sampling motor is activated. The flow control valve is fully opened and the voltage variator is adjusted so that a sample flow rate corresponding to 110% of the desired flow rate (typically 0.20-0.28 m^3/min) is indicated on the magnehelic gauge (based on the previously obtained multi-point calibration curve). The motor is allowed to warm up for 5 minutes and then the flow control valve is adjusted to achieve the desired flow rate. Turn off the sampler. The ambient temperature and barometric pressure should be recorded on a Field Test Data Sheet (Figure 10).

11.2.3.3 The flow rate transfer standard is placed on the sampling head.

11.2.3.4 Properly align the retaining rings with filter holder and secure by tightening the three screw clamps.

11.2.3.5 Using tubing, attach on 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 high volume motor and let run for five minutes.

11.2.3.8 Record the pressure differential indicated, ΔH, in inches of water. Be sure stable ΔH has been established.

11.2.3.9 Record the observed magnehelic gauge reading, in inches of water. Be sure stable M has been established.

11.2.3.10 Using previously established Flow Rate Transfer Standard curve, calculate Q_{std} (see steps 11.2.2.6-11.2.2.7).

11.2.3.11 Using previously established venturi calibration curve, calculate the indicated Q_{std} (Section 11.2.2.9).

11.2.3.12 A multi-point calibration of the Flow Rate Transfer Standard against a primary standard, must be obtained annually, as outlined in Section 11.2.1.

11.2.3.13 Remove Flow Rate Transfer Standard and dummy adsorbent cartridge and filter assembly.

11.3 Sample Collection

11.3.1 After the sampling system has been assembled and flow checked as described in Sections 11.1 and 11.2, it can be used to collect air samples, ad described in Section 11.3.2.

11.3.2 The samples should be located in an unobstructed area,

at least two 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.

11.3.3 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.

11.3.4 Detach the lower chamber of the rinsed sampling module. While wearing disposable clean lint-free nylon or powder-free surgical gloves, remove a clean glass cartridge/sorbent from its container (wide mouthed glass jar with a Teflon®-lined lid) and unwrap its aluminum foil covering. The foil should be replaced back in the sample container to be reused after the sample has been collected.

11.3.5 Insert the cartridge into the lower chamber and tightly reattach it to the module.

11.3.6 Using clean Teflon® tipped forceps, carefully place a clean fiber atop the filter holder and secure in place by clamping the filter holder ring over the filter using the three screw clamps. Insure that all module connections are tightly assembled. [Note: Failure to do so could result in air flow leaks at poorly sealed locations which could affect sample representativeness]. Ideally, sample module loading and unloading should be conducted in a controlled environment or at least a centralized sample processing area so that the sample handling variables can be minimized.

11.3.7 With the module 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.

11.3.8 Attach a "dummy" sampling module loaded with the exact same type of filter and sorbent media as that which will be used for sample collection.

11.3.9 With the sampler off, attach the Magnehelic gauge to the sampler. Turn the sampler on and adjust the flow control valve to the desired flow (normally as indicated by the cfm) magnehelic gauge reading and reference by the calibration chart. [Note:

Breakthrough has not been a problem for all PAHs outlined in Section 1.5 using this sampling method except anthracene and penanthrene]. Once the flow is properly adjusted, extreme care should be taken not to inadvertently alter its setting.

11.3.10 Turn the sampler off and remove both the "dummy" module and the Magnehelic gauge. The sampler is now ready for field use.

11.3.11 The zero reading of the sampler Magnehelic is checked. Ambient temperature, barometric pressure, elapsed time meter setting, sampler serial number, filter number, and adsorbent sample number are recorded on the Field Test Data Sheet (see Figure 10). Attach the loaded sampler module to the sampler.

11.3.12 The voltage variator and flow control valve are placed at the settings used in Section 11.2.2, and the power switch is turned on. The elapsed time meter is activated and the start time is recorded. The flow (Magnehelic setting) is adjusted, if necessary, using the flow control valve.

11.3.13 The Magnehelic reading is recorded every six hours during the sampling period. The calibration curve (Section 11.2.4) is used to calculate the flow rate. Ambient temperature, barometric pressure, and Magnehelic reading are recorded at the beginning and end of the sampling period.

11.3.14 At the end of the desired sampling period, the power is turned off. Carefully remove the sampling head containing the filter and adsorbent cartridge to a clean area.

11.3.15 While wearing disposable lint free nylon or surgical gloves, remove the sorbent cartridge from the lower module chamber and lay it on the retained aluminum foil in which the sample was originally wrapped.

11.3.16 Carefully remove the glass fiber filter from the upper chamber using clean Teflon® tipped forceps.

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

11.3.18 Wrap the combined samples in aluminum foil and place them in their original glass sample container. A sample label should be completed and affixed to the sample container. Chain-of-custody should be

maintained for all samples.

11.3.19 The glass containers should be stored in ice and protected from light to prevent possible photo-decomposition of collected analytes. If the time span between sample collection and laboratory analysis is to exceed 24 hours, sample must be kept refrigerated. [Note: Recent studies (13,16) have indicated that PUF does not retain, during storage, B[a]P as effectively as XAD-2. Therefore, sample holding time should not exceed 20 days.]

11.3.20 A final calculated sample flow check is performed using the calibration orifice, as described in Section 11.2.2. If calibration deviates by more than 10% from the initial reading, the flow data for that sample must be marked as suspect and the sampler should be inspected and/or removed from service.

11.3.21 At least one field filter/adsorbent blank will be returned to the laboratory with each group of samples. A field blank is treated exactly as a sample except that no air is drawn through the filter/adsorbent cartridge assembly.

11.3.22 Samples are stored at 0°C in an ice chest until receipt at the analytical laboratory, after which they are refrigerated at 4°C.

12. Sample Clean-up and Concentration

[Note: The following sample extraction, concentration, solvent exchange and analysis procedures are outlined for user convenience in Figure 11.]

12.1 Sample Identification

12.1.1 The samples are returned in the ice chest to the laboratory in the glass sample container containing the filter and adsorbent.

12.1.2 The samples are logged in the laboratory logbook according to sample location, filter and adsorbent 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-hrs., then the sample must be kept refrigerated. Minimize exposure of samples to fluorescence light. All samples should be extracted within one week after sampling.

12.2 Soxhlet Extraction and Concentration

12.2.1 Assemble the Soxhlet apparatus [see Figure 3(a)].

Immediately before use, charge the Soxhlet apparatus with 200 to 250 mL of methylene chloride and reflux for 2 hours. Let the apparatus cool, disassemble it, transfer the methylene chloride to a clean glass container, and retain it as a blank for later analysis, if required. Place the adsorbent and filter together in the Soxhlet apparatus (the use of an extraction thimble is optional) if using XAD-2 adsorbent in the sampling module. [Note: The filter and adsorbent are analyzed together in order to reach detection limits, avoid questionable interpretation of the data, and minimize cost.] Since methylene chloride is not a suitable solvent for PUF, 10% ether in hexane is employed to extract the PAHs from the PUF resin bed separate from the methylene chloride extraction of the accompanying filter rather than methylene chloride for the extraction of the XAD-2 cartridge.

12.2.1.1 Prior to extraction, add a surrogate standard to the Soxhlet solvent. A surrogate standard (i.e., a chemically inert 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 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 measured concentration falls within the acceptance limits. The following surrogate standards have been successfully utilized in determining matrix effects, sample process errors, etc. utilizing GC/FID, GC/MS or HPLC analysis.

Surrogate Standard	Concentration	Analytical Technique
Dibromobiphenyl	50 ng/uL	GC/FID
Dibromobiphenyl	50 ng/uL	GC/MS
Deuterated Standards	50 ng/uL	GC/MS
Decafluorobiphenyl	50 ng/uL	HPLC

[Note: The deuterated standards will be

added in Section 14.3.2. Deuterated analogs of selective PAHs cannot be used as surrogates for HPLC analysis due to coelution problems.] Add the surrogate standard to the Soxhlet solvent.

12.2.1.2 For the XAD-2 and filter extracted together, add 300 mL of methylene chloride to the apparatus and reflux for 18 hours at a rate of at least 3 cycles per hour.

12.2.1.3 For the PUF extraction separate from the filter, add 300 mL of 10 percent ether in hexane to the apparatus and reflux for 18 hours at a rate of at least 3 cycles per hour.

12.2.1.4 For the filter extraction, add 300 mL of methylene chloride to the apparatus and reflux for 18 hours at a rate of at least 3 cycles per hour.

12.2.2 Dry the extract from the Soxhlet extraction by passing it through a drying column containing about 10 grams of anhydrous sodium sulfate. Collect the dried extract in a Kuderna-Danish (K-D) concentrator assembly. Wash the extractor flask and sodium sulfate column with 100-125 mL of methylene chloride to complete the quantitative transfer.

12.2.3 Assemble a Kuderna-Danish concentrator [see Figure 3(b)] 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.]

12.2.4 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.

12.2.5 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 5 mL of cyclohexane.

12.3 Solvent Exchange

12.3.1 Replace the K-D apparatus equipped with a Snyder column back on the water bath.

12.3.2 Increase the temperature of the hot water bath to 95-100°C. Momentarily, remove the Snyder column, add a new boiling chip, and attach a two-ball micro-Snyder column. Prewet the Snyder column, using 1 mL of cyclohexane. Place the K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 15-20 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

12.3.3 When the apparatus is cool, remove the micro-Snyder column and rinse its lower joint into the concentrator tube with about 0.2 mL of cyclohexane. [Note: A 5 mL syringe is recommended for this operation]. Adjust the extract volume to exactly 1.0 mL with cyclohexane. Stopper the concentrator tube and store refrigerated at 4°C, if further processing will not be performed immediately. If the extract will be stored longer than 24 hours, it should be transferred to a Teflon®-sealed screw-cap vial.

12.4 Sample Cleanup By Solid Phase Exchange

Cleanup procedures may not be needed for relatively clean matrix samples. If the extract in Section 12.3.3 is clear, cleanup may not be necessary. If cleanup is not necessary, the cyclohexane extract (1 mL) can be analyzed directly by GC/FI detection, except the initial oven temperature begins at 30°C rather than 80°C for

cleanup samples (see Section 13.3), or solvent exchange to acetonitrile for HPLC analysis. If cleanup is required, the procedures are presented using either handpack silica gel column as prescribed in Method 610 (see Section 18.0, citation No. 18 and 22) or the use of a Lobar prepacked silica gel column for PAH concentration and separation. Either approach can be employed by the user.

12.4.1 Method 610 Cleanup Procedure [see Figure 3(c)]

12.4.1.1 Pack a 6-inch disposable Pasture pipette (10 mm I.D. x 7 cm length) with a piece of lass wool. Push the wool to the neck of the disposable pipette. Add 10 grams of activated silica gel in methylene chloride slurry to the disposable pipette. Gently tap the column to settle the silica gel and elute the methylene chloride. Add 1 gram of anhydrous sodium sulfate to the top of the silica gel column.

12.4.1.2 Prior to initial use, rinse the column with methylene chloride at 1 mL/min for 1 hr to remove any trace of contaminants. Preelute the column with 40 mL of pentane. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, transfer the 1 mL of the cyclohexane sample extract onto the column, using an additional 2 mL of cyclohexane to complete the transfer. Allow to elute through the column.

12.4.1.3 Just prior to exposure of the sodium sulfate layer to the air, add 25 mL of pentane and continue elution of the column. Discard the pentane eluate. [Note: The pentane fraction contains the aliphatic hydrocarbons collected on the filter/adsorbent combination. If interested, this fraction may be analyzed for specific aliphatic organics.] Elute the column with 25 mL of methylene chloride/pentane (4 + 6) (V/V) and collect the eluate in a 500 mL K-D flask equipped with a 10 mL concentrator tube. [Note: This fraction contains the B[a]P and other

moderately polar PAHs]. Elution of the column should be at a rate of about 2 mL/min. Concentrate the collected fraction to less than 10 mL by the K-D technique, as illustrated in Section 12.3 using pentane to rinse the walls of the glassware. The extract is now ready for HPLC or fraction may be analyzed for specific polar PAHs. However, additional cleanup by solid phase extraction may be required to obtain both qualitative and quantitative data due to complexity of the eluent.]

12.4.2 Lobar Prepacked Column Procedure

12.4.2.1 The setup using the Lobar prepacked column consists of an injection port, septum, pump, precolumn containing sodium sulfate, Lobar prepacked column and solvent reservoir.

12.4.2.2 The column is cleaned and activated according to the following cleanup sequence:

Fraction	Solvent Composition	Volume (mL)
1	100% Hexane	20
2	80% Hexane/20% Methylene Chloride	10
3	50% Hexane/50% Methylene Chloride	10
4	100% Methylene Chloride	10
5	95% Methylene Chloride/5% Methanol	10
6	80% Methylene Chloride/20% Methanol	10

12.4.2.3 Reverse the sequence at the end of the run and run to the 100% hexane fraction in order to activate the column. Discard all fractions.

12.4.2.4 Pre-elute the column with 40 mL of hexane, which is also discharged.

12.4.2.5 Inject 1 mL of the cyclohexane sample extract, followed by 1 mL injection of blank cyclohexane.

12.4.2.6 Continue elution of the column with 20 mL of hexane, which is also discharged.

12.4.2.7 Now elute the column with 180 mL of a 40/60 mixture of methylene chloride/hexane respectively.

12.4.2.8 Collect approximately 180 mL of the 40/60

methylene chloride/hexane mixture in a K-D concentrator assembly.

12.4.2.9 Concentrate to less than 10 mL with the K-D assembly as discussed in Section 12.2.

12.4.2.10 The extract is now ready for either HPLC or GC analysis.

13. Gas Chromatography Analysis with Flame Ionization Detection

13.1 Gas chromatography (GC) is a quantitative analytical technique useful for PAH identification. This method provides the user the flexibility of column selection (packed or capillary) and detector [flame ionization (FI) or mass spectrometer (MS)] selection. The mass spectrometer provides for specific identification of B(a)P; however, with system optimization, other PAHs may be qualitatively and quantitatively detected using MS (see Section 14.0). This procedure provides for common GC separation of the PAHs with subsequent detection by either FI or MS (see Figure 12.0). The following PAHs have been quantified by GC separation with either FI or MS detection:

Acenaphthene	Chrysene
Acenaphthylene	Dibenzo(a,h)anthracene
Anthracene	Fluoranthene
Benzo(a)anthracene	Fluorene
Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
Benzo(b)fluoranthene	Naphthalene
Benzo(e)pyrene	Phenanthrene
Benzo(g,h,i)perylene	Pyrene
Benzo(k)fluoranthene	

The packed column gas chromatographic method described here can not adequately resolve the following four pairs of compounds: anthracene and phenanthrene; chrysene and benzo(a)anthracene; benzo(b)fluoranthene and benzo(k)fluoranthene; and dibenzo(a,h)anthracene and indeno(1,2,3-cd)pyrene. The use of a capillary column instead of the packed column, also described in this method, should adequately resolve these PAHs. However, unless the purpose of the analysis can be served by reporting a quantitative sum for an unresolved PAH pair, either capillary gas chromatography/mass spectroscopy (Section 14.0) or high performance liquid chromatography (Section 15.0) should be used for these compounds. This section will address the use of GC/FI detection using packed or capillary columns.

13.2 To achieve maximum sensitivity with the GC/FI method, the extract must be concentrated to 1.0 mL, if not already concentrated to 1 mL. If not already concentrated to 1 mL, add a clean boiling chip to the methylene chloride extract in the concentrator tube. Attach a two-ball micro-Snyder column. Prewet the micro-Snyder

column by adding about 2.0 mL of methylene chloride to the top. Place the micro K-D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation the balls will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus. Drain and cool for at least 10 minutes. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a small volume of methylene chloride. Adjust the final volume to 1.0 mL and stopper the concentrator tube.

13.3 Assemble and establish the following operating parameters for the GC equipped with an FI detector:

	Capillary		Packed
	(A)	(B)	
Identification	SPB-5 fused silica capillary, 0.25 um 5% phenyl, methyl siloxane bonded	SPB-5 fused silica capillary, 0.25 um 5% phenyl, methyl siloxane bonded	Chromosorb W-AW-DMCS (100/120 mesh) coated with 3% OV-17
Dimensions	30-m x 0.25-mm ID	30-m x 0.25-mm ID	1.8-m x 2-mm ID
Carrier Gas	Helium	Helium	Nitrogen
Carrier Gas Flow Rate	*28-30 cm/sec* (1 cm/minute)	*28-30 cm/sec* (1 cm/minute)	*30-40 cm/minute*
Column Program	35°C for 2 min; program at 8°C/min to 280°C and hold for 12 minutes	80°C for 2 min; program at 8°C/min to 280°C and hold for 12 minutes	Hold at 100°C for 4 minutes; program at 8°C/min to 280°C and hold for 15 minutes
Detector	Flame Ionization	Flame Ionization	Flame Ionization

(A) Without column cleanup (see Section 12.4)
(B) With column cleanup (see Section 12.4.1)

13.4 Prepare and calibrate the chromatographic system using either the external standard technique (Section 13.4.1) or the internal standard technique (Section 13.4.2). Figure 13.0 outlines the following sequence involving GC calibration and retention time window determination.

13.4.1 External Standard Calibration Procedure — For each analyte of interest, including surrogate compounds for spiking, if used, prepare calibration standards at a

minimum of five concentration levels by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with methylene chloride. [Note: All calibration standards of interest involving selected PAHs, of the same concentration, can be prepared in the same flask.]

13.4.1.1 Prepare stock standard solutions at a concentration of 100 ug/uL by dissolving 0.100 gram of assayed PAH material in methylene chloride and diluting to vol-in a 10 mL volumetric flask. [Note: Larger volumes can be used at the convenience of the analyst.]

13.4.1.2 When compound purity is assayed to be 98% or greater, the weight can be used without correction to calculate the concentration of the stock standard. [Note: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.] Transfer the stock standard solutions into Teflon®-sealed screw-cap bottles.

13.4.1.3 Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards indicates a problem.

13.4.1.4 Calibration standards at a minimum of five concentration levels should be prepared through dilution of the stock standards with methylene chloride. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. [Note:

Calibration solutions must be replaced after six months, or sooner, if comparison with a check standard indicates a problem.]

13.4.1.5 Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (e.g., 1- to 3-uL injections). [Note: The same amount must be injected each time.]

13.4.1.6 Tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each analyte. [Note: Alternatively, for samples that are introduced into the gas chromatograph using a syringe, the ratio of the response to the amount injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration by the following equation:

$$\text{Calibration factor } (CF) = \frac{\text{Total Area of Peak}}{\text{Mass injected } (in \text{ nanogra}}$$

If the percent relative standard deviation (%RSD) c a l i b ration curve.]

13.4.1.7 The working calibration curve or calibration factor must be verified on each working day by the injection of one or more calibration standards. If the response for any analyte varies from the predicted response by more than ±20%, a new calibration curve must be prepared for that analyte. Calculate the percent variance by the following equation:

$$\text{Percent variance} = \frac{R_2 - R_1}{R_1} \times 100$$

where

R_2 = Calibration factor from succeeding analysis.

R_1 = Calibration factor from first analysis.

13.4.2 Internal Standard Calibration Procedure — To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Due to these limitations, no internal standard applicable to all samples can be suggested. [Note: It is recommended that the internal standard approach be used only when the GC/MS procedure is employed due to coeluting species.]

13.4.2.1 Prepare calibration standards at a minimum of five concentration levels for each analyte of interest by adding volumes of one or more stock standards to a volumetric flask.

13.4.2.2 To each calibration standard, add a known constant amount of one or more internal standard and dilute to volume with methylene chloride. [Note: One of the standards should be at a concentration near, but above, the method detection limit. The other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.]

13.4.2.3 Inject each calibration standard using the same introduction technique that will be applied to the actual samples (e.g., 1- to 3-uL injection).

13.4.2.4 Tabulate the peak height or area responses against the concentration of each compound and internal standard.

13.4.2.5 Calculate response factors (RF) for each compound as follows:

where:

$$\text{Response Factor } (RF) = (A_s C_{is}) / (A_{is} C_s)$$

A_s = Response for the analyte to be measured (area units or peak height).

A_{is} = Response for the internal standard (area units or peak height).

C_{is} = Concentration of the internal standard, (ug/L).

C_s = Concentration of the analyte to be measured, (ug/L).

13.4.2.6 If the RF value over the working range is constant (<20% RSD), the RF can be assumed to be invariant, and the average RF can be used for calculations. [Note: Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is} versus RF.]

13.4.2.7 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards.

13.4.2.8 If the response for any analyte varies from the predicted response by more than ±20%, a new calibration curve must be prepared for that compound.

13.5 Retention Time Windows Determination

13.5.1 Before analysis can be performed, the retention time windows must be established for each analyte.

13.5.2 Make sure the GC system is within optimum operating conditions.

13.5.3 Make three injections of the standard containing all compounds for retention time window determination. [Note: The retention time window must be established for each analyte throughout the course of a 72-hr period.]

13.5.4 The retention window is defined as plus or minus three times the standard deviation of the absolute retention times for each standard.

13.5.5 Calculate the standard deviation of the three absolute

retention times for each single component standard. In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a close eluting, similar compound to develop a valid retention time window.

13.5.6 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be noted and retained in a notebook by the laboratory as part of the user SOP and as a quality assurance check of the analytical system.

13.6 Sample Analysis

13.6.1 Inject 1- to 3-uL of the methylene chloride extract from Section 13.2 (however, the same amount each time) using the splitless injection technique when using capillary column. [Note: Smaller (1.0 uL) volumes can be injected if automatic devices are employed.]

13.6.2 Record the volume injected and the resulting peak size in area units or peak height.

13.6.3 Using either the internal or external calibration procedure, determine the identity and quantity of each component peak in the sample chromatogram through retention time window and established calibration curve. Table 2 outlines typical retention times for selected PAHs, using both the packed and capillary column technique coupled with FI detection, while Figure 14.0 illustrates typical chromatogram for a packed column analysis.

13.6.3.1 If the responses exceed the linear range of the system, dilute the extract and reanalyze. It is recommended that extracts be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

13.6.3.2 Establish daily retention time windows for each analyte. Use the absolute retention time for each analyte from Section 13.5.4 as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 13.5.4.

13.6.3.3 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. [Note: Confirmation may be required on a second GC column, or by GC/MS (if concentration permits) or by other recognized confirmation techniques if overlap of peaks occur.]

13.6.3.4 Validation of GC system qualitative performance is performed through the use of the midlevel standards. If the mid-level standard falls outside its daily retention time window, the system is out of control. Determine the cause of the problem and perform a new calibration sequence (see Section 13.4).

13.6.3.5 Additional validation of the GC system performance is determined by the surrogate standard recovery. If the recovery of the surrogate standard deviates from 100% by not more than 20%, then the sample extraction, concentration, clean-up and analysis is certified. If it exceeds this value, then determine the cause of the problem and correct.

13.6.4 Determine the concentration of each analyte in the sample according to Sections 17.1 and 17.2.1.

14. Gas Chromatography with Mass Spectroscopy Detection

14.1 The analysis of the extracted sample for benzo[a]pyrene and other PAHs is accomplished by an electron impact gas chromatography/mass spectrometry (EI GC/MS) in the selected ion monitoring (SIM) mode with a total cycle time (including voltage reset time) of one second or less. The GC is equipped with an ultra No. 2 fused silica capillary column (50-m x 0.25-mm I.D.) with helium carrier

gas for analyte separation. The GC column is temperature controlled and interfaced directly to the MS ion source.

14.2 The laboratory must document that the EI GC/MS system is properly maintained through periodic calibration checks. The GC/MS system should have the following specifications:

Mass range: 35-500 amu
Scan time: 1 sec/scan
GC Column: 50 m x 0.25 mm I.D. (0.25 um film thickness) Ultra No. 2 fused silica capillary column or equivalent
Initial column temperature and hold time: 40°C for 4 min
Column temperature program: 40-270°C at 10°C/min
Final column temperature hold: 270°C (until benzo[g,h,i]perylene has eluted)
Injector temperature: 250-300°C
Transfer line temperature: 250-300°C
Source temperature: According to manufacturer's specifications
Injector: Grob-type, splitless
EI Condition: 70 eV
Mass Scan: Follow manufacturer instruction for select ion monitoring (SIM) mode
Sample volume: 1-3 uL
Carrier gas: Helium at 30 cm/sec

The GC/MS is tuned using a 50 ng/uL 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.

14.3 The GC/MS operating conditions are outlined in Table 4. The GC/MS system can be calibrated using the external standard technique (Section 14.3.1) or the internal standard technique (Section 14.3.2). Figure 15.0 outlines the following sequence involving the GC/MS calibration.

14.3.1 External standard calibration procedure.

14.3.1.1 Prepare calibration standard of B[a]P or other PAHs at a minimum of five concentration levels by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with methylene chloride. The stock standard solution of B[a]P (1.0 ug/uL) must be prepared from pure standard materials or purchased as certified solutions.

14.3.1.2 Place 0.0100 grams of native B[a]P or other PAHs on a tared aluminum weighing disk and weigh on a Mettler balance.

14.3.1.3 Quantitatively, transfer to a 10 mL volumetric flask. Rinse the weighing disk with several small portions of methylene chloride. Ensure all material has been transferred.

14.3.1.4 Dilute to mark with methylene chloride.

14.3.1.5 The concentration of the stock standard solution of B[a]P or other PAHs in the flask is 1.0 ug/uL [Note: Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.]

14.3.1.6 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.

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

14.3.1.8 Calibration standards at a minimum of five concentration levels should be prepared. [Note: One of the calibration standards should be at a concentration near, but above the method detection limit; the others should correspond to the range of concentrations found in the sample but should not exceed the working range of the GC/MS system.] Accurately pipette 1.0 mL of the stock solution (1 ug/uL) into another 10 mL volumetric flask, dilute to mark with methylene chloride. This daughter solution contains 0.1 ug/uL of B[a]P or other PAHs.

14.3.1.9 Prepare a set of standard solutions by appropriately diluting, with methylene chloride, accurately measured volumes of the daughter solution (0.1 ug/uL).

14.3.1.10 Accurately pipette 100 uL, 300 uL, 500 uL, 700 uL and 1000 uL of the daughter solution (0.1 ug/uL) into each 10 mL volumetric flask, respectively. To each of these flasks, add an internal deuterated standard to give a final concentration of 40 ng/uL of the internal deuterated standard (Section 14.3.2.1). Dilute to mark with methylene chloride.

14.3.1.11 The concentration of B[a]P in each flask is 1 ng/uL, 3 ng/uL, 5 ng/uL, 7 ng/uL, and 10 ug/uL respectively. All standards should be stored at 4°C and protected from fluorescent light and should be freshly prepared once a week or sooner if check standards indicates a problem.

14.3.1.12 Analyze a constant volume (1-3 uL) of each calibration standard and tabulate the area responses of the primary characteristic ion of each standard against the mass injected. The results may be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<20% relative standard deviation, RSD), linearity through the origin may be assumed and the average ratio or calibration factor may be used in place of a calibration curve.

14.3.1.13 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±20%, the rest must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that compound.

14.3.2 Internal standard calibration procedure.

14.3.2.1 To use this approach, the analyst must

select one or more internal standards that are similar in analytical behavior to the compounds of interest. For analysis of B[a]P, the analyst should use perylene $-d_{12}$. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. The following internal standards are suggested at a concentration of 40 ng/uL for specific PAHs:

Perylene - d_{12}	Acenaphthene - d_{10}
Benzo(a)pyrene	Acenaphthene
Benzo(k)fluoranthene	Acenaphthylene
Benzo(g,h,i)perylene	Fluorene
Dibenzo(a,h)anthracene	
Indeno(1,2,3-cd)pyrene	Naphthalene - d_8
Chrysene - d_{12}	Naphthalene
Benzo(a)anthracene	Phenanthrene - d_{10}
Chrysene	
Pyrene	Anthracene
	Fluoranthene
	Phenanthrene

14.3.2.2 A mixture of the above deuterated compounds in the appropriate concentration range are commercially available (see Section 9.3.1.5).

14.3.2.3 Use the base peak ion as the primary ion for quantification of the standards. If interferences are noted, use the next two most intense ions as the secondary ions. The internal standard is added to all calibration standards and all sample extracts analyzed by GC/MS. Retention time standards, column performance standards, and a mass spectrometer tuning standard may be included in the internal standard solution used.

14.3.2.4 Prepare calibration standards at a minimum of three concentration level for each parameter of interest by adding appropriate volumes of one or more stock standards to a volumetric flask. To each calibration standard or standard mixture,

add a known constant amount of one or more of the internal deuterated standards to yield a resulting concentration of 40 ng/uL of internal standard and dilute to volume with methylene chloride. One of the calibration standards should be at a concentration near, but above, the minimum detection limit (MDL) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system.

14.3.2.5 Analyze constant amount (1-3 uL) of each calibration standard and tabulate the area of the primary characteristic ion against concentration for each compound and internal standard, and calculate the response factor (RF) for each analyte using the following equation:

$$RF = (A_s C_{is}) / (A_{is} C_s)$$

Where:

A_s = Area of the characteristic ion for the analyte to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

C_{is} = Concentration of the internal standard, (ng/uL).

C_s = Concentration of the analyte to be measured, (ng/uL).

If the RF value over the working range is a constant (<20% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF. Table 5.0 outlines key ions for selected internal deuterated standards.

14.3.2.6 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any

parameter varies from the predicted response by more than ±20%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared.

14.3.2.7 The relative retention times for each compound in each calibration run should agree within 0.06 relative retention time units.

14.4 Sample Analysis

14.4.1 It is highly recommended that the extract be screened on a GC/FID or GC/PID using the same type of capillary column as in the GC/MS procedure. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

14.4.2 Analyze the 1 mL extract (see Section 13.2) by GC/MS. The recommended GC/MS operating conditions to be used are specified in Section 14.2.

14.4.3 If the response for any quantitation ion exceeds the initial calibration curve range of the GC/MS system, extract dilution must take place. Additional internal standard must be added to the diluted extract to maintain the required 40 ng/uL of each internal standard in the extracted volume. The diluted extract must be reanalyzed.

14.4.4 Perform all qualitative and quantitative measurements as described in Section 14.3. The typical characteristic ions for selective PAHs are outlined in Table 6.0. Store the extracts at 4°C, protected from light in screw-cap vials equipped with unpierced Teflon®-lined, for future analysis.

14.4.5 For sample analysis, the comparison between the sample and references spectrum must illustrate:

(1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible substraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

14.4.6 Determine the concentration of each analyte in the sample according to Sections 17.1 and 17.2.2.

14.5 GC/MS Performance Tests

14.5.1 Daily DFTPP Tuning — At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see that acceptable performance criteria are achieved when challenged with a 1 uL injection volume containing 50 ng of decafluorotriphenylphosphine (DFTPP). The DFTPP key ions and ion abundance criteria that must be met are illustrated in Table 3.0. Analysis should not begin until all those criteria are met. Background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. The GC/MS tuning standard should be used to assess GC column performance and injection port inertness. Obtain a background correction mass spectra of DFTPP and check that all key ions criteria are met. If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks on standards are analyzed. If any key ion abundance observed for the daily DFTPP mass tuning check differs by more than 10% absolute abundance from that observed during the previous daily tuning, the instrument must be retuned or the sample and/or calibration solution reanalyzed until the above condition is met.

14.5.2 Daily 1-point Initial Calibration Check — At the beginning of each work day, a daily 1-point calibration check is performed by re-evaluating the midscale calibration standard. This is the same check that is applied during the initial calibration, but

one instead of five working standards are evaluated. Analyze the one working standards under the same conditions the initial calibration curve was evaluated. Analyze 1 uL of each of the mid-scale calibration standard and tabulate the area response of the primary characteristic ion against mass injected. Calculate the percent difference using the following equation:

$$\% \text{ Difference} = \frac{RF_c - \overline{RF_I}}{\overline{RF_I}} \times 100$$

Where:

RF_I = average response factor from initial calibration using mid-scale standard.

RF_c = response factor from current verification check using mid-scale standard.

If the percent difference for the mid-scale level is greater than 10%, the laboratory should consider this a warning limit. If the percent difference for the mid-scale standard is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (<20/5 difference), then corrective action MUST be taken. [Note: Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.] This check must be met before analysis begins. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration MUST be generated. This criterion MUST be met before sample analysis begins.

14.5.3 12-hour Calibration Verification — A calibration standard at mid-level concentration containing B[a]P or other PAHs must be performed every twelve continuous hours of analysis. Compare the standard every 12-hours with the average response factor from the initial calibration. If the % difference for the response factor (see Section 14.5.2) is less than 20%, then the GC/MS system is operative within initial calibration values. If the criteria is not met (>20% difference), then the source of the problem must be

determined and a new five-point curve MUST be generated.

14.5.4 Surrogate Recovery — Additional validation of the GC system performance is determined by the surrogate standard recovery. If the recovery of the surrogate standard deviates from 100% by not more than 20%, then the sample extraction, concentration, clean-up and analysis is certified. If it exceeds this value, then determine the cause of the problem and correct.

15. High Performance Liquid Chromatography (HPLC) Detection

15.1 Introduction

15.1.1 Detection of B[a]P by HPLC has also been a viable tool in recent years. The procedure outlined below has been written specifically for analysis of B[a]P by HPLC. However, by optimizing chromatographic conditions [(multiple detector fluorescence — excitation at 240 nm, emission at 425 nm; ultraviolet at 254 nm)] and varying the mobile phase composition through a gradient program, the following PAHs may also be quantified:

COMPOUND	DETECTOR[1]	COMPOUND	DETECTOR[1]
Acenaphthene	UV	Benzo(k)fluoranthene	FL
Acenaphthylene	UV	Dibenzo(a,h)anthracene	FL
Anthracene	UV	Fluoranthene	FL
Benzo(a)anthracene	FL	Fluorene	UV
Benzo(a)pyrene	FL	Indeno(1,2,3-cd)pyrene	FL
Benzo(b)fluoranthene	FL	Naphthalene	UV
Benzo[e]pyrene	FL	Phenanthrene	UV
Benzo(ghi)perylene	FL	Pyrene	FL

[1]UV = Ultraviolet
FL = Fluorescence

15.1.2 This method provides quantitative identification of the selected PAH's compounds listed above by high performance liquid chromatography. It is based on separating of compounds of a liquid mixture through a liquid chromatographic column and measuring the separated components with suitable detectors.

15.1.3 The method involves solvent exchange, with subsequent HPLC detection involving ultraviolet (UV) and fluorescence (FL) detection.

15.2 Solvent Exchange To Acetonitrile

15.2.1 To the extract in the concentrator tube, add 4 mL of acetonitrile and a new boiling chip; attach a micro-

snyder column to the apparatus.

15.2.2 Increase temperature of the hot water bath to 95 to 100°C.

15.2.3 Concentrate the solvent as in Section 12.3.

15.2.4 After cooling, remove the micro-Snyder column and rinse its lower sections into the concentration tube with approximately 0.2 mL acetonitrile.

15.2.5 Adjust its volume to 1.0 mL.

15.3 HPLC Assembly

15.3.1 The HPLC system is assembled, as illustrated in Figure 10.

15.3.2 The HPLC system is operated according to the following parameters:

HPLC Operating Parameters

Guard Column:	VYDAC 201 GCCIOYT	
Analytical Column:	VYDAC 201TP5415 C-18 RP (0.46 x 25 cm)	
Column Temperature:	27.0 ± 2°C	
Mobile Phase:	Solvent Composition	Time (Minutes)
	40% Acetonitrile/605 water	0
	100% Acetonitrile	25
	100% Acetonitrile	35
	40% Acetonitrile/605 water	45
	Linear gradient elution at 1.0 mL/min	
Detector:	Variable wavelength ultraviolet and fluorescence.	
Flow Rate:	1.0 mL/minute	

[Note: To prevent irreversible absorption due to "dirty" injections and premature loss of column efficiency, a guard column is installed between the injection and the analytical column. The guard column is generally packed with identical material as is found in the analytical column. The guard column is generally replaced with a fresh guard column after several injections (50) or when separation between compounds becomes difficult. The analytical column specified in this procedure has been laboratory evaluated. Other analytical columns may be used as long as they meet procedure and separation requirements. Table 7..0 outlines other columns uses to determine PAHs by HPLC.]

15.3.3 The mobile phases are placed in separate HPLC solvent reservoirs and the pumps are set to yield a total of 1.0 mL/minute and allowed to pump for 20-30 minutes before the first analysis. The detectors are switched on at least 30 minutes before the first analysis. UV detection at 254 nm is generally preferred. The fluorescence spectrometer excitation wavelengths range

from 250 to 800 nanometers. The excitation and emission slits are both set at 10 nanometers nominal bandpass.

15.4 HPLC Calibration

15.4.1 Prepare stock standard solutions at PAH concentrations or 1.00 ug/uL by dissolving 0.0100 grams of assayed material acetonitrile and diluting to volume in a 10 mL volumetric flask. [Note: Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 98% or greater, the weight can be used without correction to calculate the concentration of the stock standard.] Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

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

15.4.3 Stock standard solutions must be replaced after one year, or sooner, if comparison with check standards indicates a problem.

15.4.4 Prepare calibration standards at a minimum of five concentration levels ranging from 1 ng/uL to 10 ng/uL first diluting the stock standard 10:1 with acetonitrile, giving a daughter solution of 0.1 ug/uL. Accurately pipette 100 uL, 300 uL, 500 uL, 700 uL and 1000 uL of the daughter solution (0.1 ug/uL) into each 10 mL volumetric flask, respectively. Dilute to mark with acetonitrile. One of the concentration levels should be at a concentration near, but above, the method detection limit (MDL). The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the HPLC. [Note: Calibration standards must be replaced after six months, or sooner, if comparison with check standards indicates a problem.]

15.4.5 Analyze each calibration standard (at least five levels) three times. Tabulate area response vs. mass

injected. All calibration runs are performed as described for sample analysis in Section 15.5.1. Typical retention times for specific PAHs are illustrated in Table 8.0. Linear response is indicated where a correlation coefficient of at least 0.999 for a linear least-squares fit of the data (concentration versus area response) is obtained. The retention times for each analyte should agree within ±2%.

15.4.6 Once linear response has been documented, an intermediate concentration standard near the anticipated levels for each component, but at least 10 times the detection limit, should be chosen for a daily calibration check. The response for the various components should be within 15% day to day. If greater variability is observed, recalibration may be required or a new calibration curve must be developed from fresh standards.

15.4.7 The response for each component in the daily calibration standard is used to calculate a response factor according to the following equation:

$$RF_c = \frac{C_c \, x \, V_I}{R_c}$$

Where

RF_c = response factor (usually area counts) for th

C_c = concentration (mg/L) of analyte in the daily calibration standard.

V_I = volume (uL) of calibration standard injected.

R_c = response (area counts) for analyte in the calibration standard.

15.5 Sample Analysis

15.5.1 A 100 uL aliquot of the sample is drawn into a clean HPLC injection syringe. The sample injection loop (10 uL) is loaded and an injection is made. The data system, if available, is activated simultaneously with the injection and the point of injection is marked on the strip-chart recorded.

15.5.2 After approximately one minute, the injection valve is returned to the "load" position and the syringe and valve are flushed with water in preparation for the next sample analysis.

15.5.3 After elution of the last component of interest, concentrations are calculated as described in Section 16.2.3. [Note: Table 8.0 illustrates typical retention times associated with individual PAHs, while Figure 17 represents a typical chromatogram associated with fluorescence detection.]

15.5.4 After the last compound of interest has eluted, establish a stable baseline; the system can be now used for further sample analyses as described above.

15.5.5 If the concentration of analyte exceeds the linear range of the instrument, the sample should be diluted with mobile phase, or a smaller volume can be injected into the HPLC.

15.5.6 Calculate surrogate standard recovery on all samples, blanks and spikes. Calculate the percent difference by the following equation:

$$\% \text{ difference} = \frac{S_R - S_I}{S_I} \times 100$$

Where

S_I = surrogate injected, ng.

S_R = surrogate recovered, ng.

15.5.7 Once a minimum of thirty samples of the same matrix have been analyzed, calculate the average percent recovery (%R) and standard deviation of the percent recovery (SD) for the surrogate.

15.5.8 For a given matrix, calculate the upper and lower control limit for method performance for the surrogate standard. This should be done as follows:

Upper Control Limit (UCL) = (%R) + 3(SD)

Lower Control Limit (LCL) = (%R) - 3(SD)

The surrogate recovery must fall within the control limits. If recovery is not within limits, the following is required.

- Check to be sure there are no errors in calculations surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

15.5.9 Determine the concentration of each analyte in the

sample according to Section 17.1 and 17.2.3.

15.6 HPLC System Performance

15.6.1 The general appearance of the HPLC system should be similar to that illustrated in Figure 10.

15.6.2 HPLC system efficiency is calculated according to the following equation:

$$N = 5.54 \frac{t_r^2}{W_{1/2}}$$

Where:

N = column efficiency (theoretical plates).

t_r = retention time (seconds) of analyte.

$W_{1/2}$ = width of component peak at half height (seconds).

A column efficiency of >5,000 theoretical plates should be obtained.

15.6.3 Precision of response for replicate HPLC injections should be ±10% or less, day to day, for analyte calibration standards at 1 ug/mL or greater levels. At 0.5 ug/mL level and below, precision of replicate analyses could vary up to 25%. Precision of retention times should be ±2% on a given day.

15.6.4 From the calibration standards, area responses for each PAH compound can be used against the concentrations to establish working calibration curves. The calibration curve must be linear and have a correlation coefficient greater than 0.98 to be acceptable.

15.6.5 The working calibration curve should be checked daily with an analysis of one or more calibration standards. If the observed response (r_o) for any PAH varies by more than 15% from the predicted response (r_p), the test method must be repeated with new calibration standards. Alternately a new calibration curve must be prepared. [Note: If $\frac{r_o - r_p}{r_p}$ >15%, recalibration is necessary.]

15.7 HPLC Method Modification

15.7.1 The HPLC procedure has been automated by Acurex Corporation as part of their "Standard Operating Procedure for Polynuclear Aromatic Hydrocarbon Analysis by High Performance Liquid Chromatography Methods," as reported in Reference 9 of Section 18.

15.7.2 The system consists of a Spectra Physics 8100 Liquid Chromatograph, a micro-processor-controlled HPLC, a ternary gradient generator, and an autosampler (10 uL injection loop).

15.7.3 The chromatographic analysis involves an automated solvent program allowing unattended instrument operation. The solvent program consists of four timed segments using varying concentrations of acetonitrile in water with a constant flow rate, a constant column temperature, and a 10-minute equilibration time, as outlined below.

AUTOMATED HPLC WORKING PARAMETERS

Time	Solvent Composition	Temperature	Rate
10 minutes equilibration	40% Acetonitrile 60% Water	27.0 ± 2°C	1 mL/min
T=0	40% Acetonitrile 60% Water		
T=25	100% Acetonitrile		
T=35	100% Acetonitrile		
T=45	40% Acetonitrile 60% Water		

Table 9.0 outlines the associated PAHs with their minimum detection limits (MDL) which can be detected employing the automated HPLC methodology.

15.7.4 A Vydac or equivalent analytical column packed with a C_{18} bonded phase is used for PAH separation with a reverse phase guard column. The optical detection system consists of a Spectra Physics 8440 variable Ultraviolet (UV)/Visible (VIS) wavelength detector and a Perkin Elmer LS-4 Fluorescence Spectrometer. The UV/VIS detector, controlled by remote programmed commands, contains a Deuterium lamp with wavelength selection between 150 and 600 nanometers. It is set at 254 nanometers with the time constant (detector response) at 1.0 seconds.

15.7.5 The LS-4 Fluorescence Spectrometer contains separate excitation and emission monochromators which are positioned by separate microprocessor-controlled stepper motors. It contains a Xenon discharge lamp, side-on photomultiplier and a 3-microliter illuminated volume flow cell. It is equipped with a wavelength

programming facility to set the monochromators automatically to a given wavelength position. This greatly enhances selectivity by changing the fluorescence excitation and emission detection wavelengths during the chromatographic separation in order to optimize the detection of each PAH. The excitation wavelengths range from 230 to 720 nanometers; the emission wavelengths range from 250 to 800 nanometers. The excitation and emission slits are both set at 10 nanometers nominal bandpass.

15.7.6 The UV detector is used for determining naphthalene, acenaphthylene and acenaphthene, and the fluorescence detector is used for the remaining PAHs. Table 9 outlines the detection techniques and minimum detection limit (MDL) employing this HPLC system. A Dual Channel Spectra Physics (SP) 4200 computing integrator, with a Labnet power supply, provides data analysis and a chromatogram. An IBM PC XT with a 10-megabyte hard disk provides data storage and reporting. Both the SP4200 and the IBM PC XT can control all functions of the instruments in the series through the Labnet system except for the LS-4, whose wavelengths program is started with a signal from the High Performance Liquid Chromatograph autosampler when it injects. All data are transmitted to the XT and stored on the hard disk. Data files can later be transmitted to floppy disk storage.

16.0 Quality Assurance/Quality Control

16.1 General System QA/QC

16.1.1 Each laboratory that uses these methods is required to 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.

16.1.2 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.

16.1.3 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.

16.1.4 The experience of the analyst performing gas chromatography and high performance liquid chromatography 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?; Is 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.

16.2 Process, Field, and Solvent Blanks

16.2.1 One cartridge (XAD-2 or PUF) and filter from each batch of approximately twenty should be analyzed, without shipment to the field, for the compounds of interest per to serve as a process blank. A blank level of less than 10 ng per cartridge/filter assembly for single PAH component is considered to be acceptable.

16.2.2 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.

16.2.3 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 less than 10 ng/sample for single components to be acceptable.

16.2.4 Because the sampling configuration (filter and backup adsorbent) has been tested for targeted PAHs in the laboratory in relationship to collection efficiency and has been demonstrated to be greater than 95% for targeted PAHs, no field recovery evaluation will occur as part of the QA/QC program outlined in this section.

16.3 Gas Chromatography with Flame Ionization Detection

16.3.1 Under the calibration procedures (internal and external) the % RSD of the calibration factor should be <20% over the linear working range of a five point calibration curve (Sections 13.4.1.6 and 13.4.2.6).

16.3.2 Under the calibration procedures (internal and external), the daily working calibration curve for each analyte should not vary from the predicted response by more than ±20% (Sections 13.4.1.7 and 13.4.2.8).

16.3.3 For each analyte, the retention time window must be established (Section 13.5.1), verified on a daily basis (Section 13.6.3.2) and established for each analyte throughout the course of a 72-hour period (Section 13.5.3).

16.3.4 For each analyte, the mid-level standard must fall within the retention time window on a daily basis as a qualitative performance evaluation of the GC system

(Section 13.6.3.4).

16.3.5 The surrogate standard recovery must not deviate from 100% by no more than 20% (Section 13.6.3.5).

16.4 Gas Chromatography with Mass Spectroscopy Detection

16.4.1 Section 14.5.1 requires the mass spectrometer be tuned daily with DFTPP and meet relative ion abundance requirements outlined in Table 3.

16.4.2 Section 14.3.1.1 requires a minimum of five concentration levels of each analyte (plus deuterated internal standards) be prepared to establish a calibration factor to illustrate <20% variance over the linear working range of the calibration curve.

16.4.3 Section 14.3.1.13 requires the verification of the working curve each working day (if using the external standard technique) by the measurement of one or more calibration standards. The predicted response must not vary by more than ±20%.

16.4.4 Section 14.3.2.6 requires the initial calibration curve be verified each working day (if using the internal standard technique) by the measurement of one or more calibration standards. If the response varies by more than ±20% of predicted response, a fresh calibration curve (five point) must be established.

16.4.5 Section 14.4.5 requires that for sample analysis, the comparison between the sample and reference spectrum illustrates:

(1) Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible substraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

16.4.6 Section 14.5.3 requires that initial calibration curve be verified every twelve continuous hour of analysis by a mid-level calibration standard. The response must be less than 20% difference from the initial response.

16.4.7 The surrogate standard recovery must not deviate from 100% by no more than 20% (Section 14.5.4).

16.5 High Performance Liquid Chromatography

16.5.1 Section 15.4.4 requires the preparation of calibration standards at a minimum of five concentration levels to establish correlation coefficient of at least 0.999 for a linear least-squares fit of the data.

16.5.2 Section 15.4.5 requires that the retention time for each analyte should agree within ±2%.

16.5.3 A daily calibration check involving an intermediate standard of the initial five point calibration curve should be within ±15% from day to day.

16.5.4 Section 15.5.6 requires the calibration of percent difference of surrogate standard recovery in order to establish control limits:

Upper Control Limit (UCL) = (%R) + 3(SD)

Lower Control Limit (LCL) = (%R) - 3(SD)

The surrogate recovery must fall within the control limits.

17. Calculations

17.1 Sample Volume

17.1.1 The total sample volume should be corrected to standard temperature and pressure.

17.1.2 The total sample volume (V_m) is calculated from the periodic flow readings (Magnehelic readings taken in Section 11.3.13) using the following equation.

$$V_m = \frac{Q_1 + Q_2 \ldots Q_n}{N} \times \frac{T}{1000}$$

Where

V_m = total sample volume (m^3) at ambient conditions.

$Q_1, Q_2 \ldots Q_n$ = flow rates determined at the beginning, end, and intermediate points during sampling (m^3/minute).

N = number of data points.

T = elapsed sampling time (minutes).

17.1.3 The volume of air sampled can be converted to standard conditions (760 mm Hg pressure and 25°C) using the following equation:

Where

$$V_s = V_m \, x \, \frac{P_A}{760} \, x \, \frac{298}{273 + t_A}$$

V_s = total sample volume (m^3) at standard temperature and pressure (25°C and 760 mm Hg pressure).

V_m = total sample flow under ambient conditions (m^3).

P_A = ambient pressure (mm Hg).

t_A = ambient temperature (°C).

17.2 Sample Concentration

17.2.1 GC/FI Detection

17.2.1.1 The concentration of each analyte in the sample may be determined from the external standard technique by calculating the amount of standard injected, from the peak response, using the calibration curve or the calibration factor determined in Section 13.4.1.6.

17.2.1.2 The concentration of a specific analyte is calculated as follows:

$$\text{Concentration, ng/m}^3 = \frac{[(A_x)(V_t)(D)]}{[(CF)(V_i)(V_s)]}$$

Where:

CF = calibration factor for chromatographic system, peak height or area response per mass injected, Section 13.4.1.6.

A_x = Response for the analyte in the sample, area counts or peak height.

V_t = volume of total sample, uL.

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D=1, dimensionless.

V_i = volume of sample injected, uL.

V_s = total sample volume (m^3) at standard temperature and pressure (25°C and 760 mm Hg) Section 17.1.3.

17.2.2 GC/MS Detection

17.2.2.1 When an analyte has been identified, the quantification of that analyte will be based on the integrated abundance from the monitoring of the primary characteristic ion. Quantification will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte (see Section 14.3.2.1).

17.2.2.2 Calculate the concentration of each identified analyte in the sample as follows:

$$\text{Concentration, ng/m}^3 = \frac{[(A_x)(I_s)(V_t)(D)}{[(A_{is})(RF)(V_i)(V_s}$$

Where

A_x = area of characteristic ion(s) for analyte being measured.

I_s = amount of internal standard injected, ng.

V_t = volume of total sample, uL.

D = dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D=1, dimensionless.

A_{is} = area of characteristic ion(s) for internal standard.

V_i = volume of analyte injected, uL.

V_s = total sample volume (m^3) at standard temperature and pressure (25°C and 760 mm Hg), Section 17.1.3.

17.2.3 HPLC Detection

17.2.3.1 The concentration of each analyte in the sample may be determined from the external standard technique by calculating response factor and peak response using the calibration curve.

17.2.3.2 The concentration of a specific analyte is calculated as follows:

Where

$$\text{Concentration, ng/m}^3 = \frac{[(RF_c)(A_x)(V_t)(D}{[(V_i)(V_s)]}$$

RF_c = response factor (nanograms injected per area counts) calculated in Section 15.4.7.

A_x = response for the analyte in the sample, area counts or peak height.

V_t = volume of total sample, uL.

D = dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D=1, dimensionless.

V_i = volume of sample injected, uL.

V_s = total sample volume (m^3) at standard temperature and pressure (25°C and 760 mm Hg), Section 17.1.3.

17.3 Sample Concentration Conversion From ng/m^3 to ppbv

17.3.1 The concentrations calculated in Section 17.2 can be converted to ppbv for general reference.

17.3.2 The analyte concentration can be converted to ppbv using the following equation:

$$C_A\ (\text{ppbv}) = C_A\ (\text{ng/m}^3)\ x\ \frac{24.4}{MW_A}$$

Where

C_A = concentration of analyte, ng/m^3, calculated according to Sections 17.2.1 through 17.2.3.

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), 1/mole.

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TABLE 1.0 FORMULAE AND PHYSICAL PROPERTIES OF SELECTIVE PAHs

	FORMULA	MOLECULAR WEIGHT	MELTING POINT °C	BOILING POINT °C	CASE #
Acenaphthene	$C_{12}H_{10}$	154.21	96.2°	279	83-32-9
Acenaphthylene	$C_{12}H_8$	152.20	92-93	265-275	208-96-8
Anthracene	$C_{14}H_{10}$	178.22	218°	342	120-12-7
Benzo(a)anthracene	$C_{18}H_{12}$	228.29	158-159	-	56-55-3
Benzo(a)pyrene	$C_{20}H_{12}$	252.32	177°	310-312	50-32-8
Benzo(b)fluoranthene	$C_{20}H_{12}$	252.32	168	-	205-99-2
Benzo(e)pyrene	$C_{20}H_{12}$	252.32	178-179	-	192-92-2
Benzo(g,h,i)perylene	$C_{22}H_{12}$	276.34	273	-	191-24-2
Benzo(k)fluoranthene	$C_{20}H_{12}$	252.32	217	480	207-08-9
Chrysene	$C_{18}H_{12}$	228.29	255-256	-	218-01-9
Dibenzo(a,h)anthracene	$C_{22}H_{14}$	278.35	262	-	53-70-3
Fluoranthene	$C_{16}H_{10}$	202.26	110	-	206-44-0
Fluorene	$C_{13}H_{10}$	166.22	116-117	293-295	86-73-7
Indeno(1,2,3-cd)pyrene	$C_{22}H_{12}$	276.34	161.5-163	-	193-39-5
Naphthalene	$C_{10}H_8$	128.16	80.2	217.9	91-20-3
Phenanthrene	$C_{14}H_{10}$	178.22	100°	340	85-01-8
Pyrene	$C_{16}H_{10}$	202.26	156	399	129-00-0

*Many of these compounds sublime.

TABLE 2.0 RETENTION TIMES FOR SELECTIVE PAHs FOR PACKED AND CAPILLARY COLUMNS

Compound	Packed[1]	Capillary[2]
Acenaphthene	10.8	16.8
Acenaphthylene	10.4	15.9
Anthracene	15.9	20.7
Benzo(a)anthracene	20.6	29.1
Benzo(a)pyrene	29.4	36.2
Benzo(b)fluoranthene	28.0	34.2
Benzo(ghi)perylene	38.6	48.4
Benzo(k)fluoranthene	28.0	34.4
Chrysene	24.7	29.3
Dibenzo(a,h)anthracene	36.2	46.1
Fluoranthene	19.8	24.3
Fluorene	12.6	18.1
Indeno(1,2,3-cd)pyrene	36.2	45.6
Naphthalene	4.5	11.0
Phenanthrene	15.9	20.6
Pyrene	20.6	25.0

[1]GC conditions: Chromosorb W-AW-DMCS (100/120 mesh) coated with 3% OV-17, packed in a 1.8-m long x 2 mm ID glass column, with nitrogen carrier gas at a flow rate of 40 mL/min. Column temperature was held at 100°C for 4 min, then programmed at 8°/minute to a final hold at 280°C.

[2]Capillary GC conditions: 30 meter fused silica SPB-5 capillary column; flame ionization detector, splitless injection; oven temperature held at 80 degrees C for 2 minutes, increased at 8 degrees/min. to 280 degrees C.

TABLE 3.0 DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	Less than 2% of mass 69
70	Less than 2% of mass 69
127	40-60% of mass 198
197	Less than 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	Greater than 1% of mass 198
441	Present but less than mass 443
442	Greater than 40% of mass 198
443	17-23% of mass 442

TABLE 4.0 GC AND MS OPERATING CONDITIONS

Chromatography	
Column	Hewlett-Packard Ultra #2 crosslinked 5% phenyl methyl silicone (50 m x 0.25 mm, um film thickness) or equivalent
Carrier Gas	Helium velocity 20 cm^3/sec at 250°C
Injection Volume	Constant (1-3 uL)
Injection Mode	Splitless
Temperature Program	
Initial Column Temperature	45°C
Initial Hold Time	1 min
Program	45°C to 100°C in 5 min, then 100°C to 320°C at 8°C/min
Final Hold Time	15 min
Mass Spectrometer	
Detection Mode	Multiple ion detection, SIM mode

TABLE 5.0 CHARACTERISTIC IONS FROM GS/MS DETECTION FOR DEUTERATED INTERNAL STANDARDS AND SELECTED PAHs

Compound	M/Z
D_8-naphthalene	136
D_{10}-phenanthrene	188
Phenanthrene	178
Anthracene	178
Fluoranthene	202
D_{10}-pyrene	212
Pyrene	202
Cyclopenta[c,d]pyrene	226
Benzo[a]anthracene	228
D_{12}-chrysene	240
Benzo[e]pyrene	252
D_{12}-benzo[a]pyrene	264
Benzo[a]pyrene	252

TABLE 6.0 CHARACTERISTIC IONS FROM GC/MS DETECTION FOR SELECTED PAHs

Compound	Primary		Secondary
Acenaphthene	154	153	152
Acenaphthylene	152	151	153
Anthracene	178	179	176
Benzo(a)anthracene	228	229	226
Benzo(a)pyrene	252	253	125
Benzo(b)fluoranthene	252	253	125
Benzo(ghi)perylene	276	138	277
Benzo(k)fluoranthene	252	253	125
Chrysene	228	226	229
Dibenzo(a,h)anthracene	278	139	279
Fluoranthene	202	101	203
Fluorene	166	165	167
Indeno(1,2,3-cd)pyrene	276	138	227
Naphthalene	128	129	127
Phenanthrene	178	179	176
Pyrene	202	200	203

TABLE 7.0. COMMERCIAL AVAILABLE COLUMNS FOR PAH ANALYSIS USING HPLC

Company	Column Identification	Column Name
The Separation Group P.O. Box 867 Hesperia, California 92345	201-TP	VYDAC
Rainin Instrument Company Mack Road Wasurn, MA 01801-4626	Ultrasphere - ODS	ALEX
Supelco, Inc. Supelco Park Bellefonte, PA 16823-0048	LC-PAH	Supelcosil
DuPont Company Bictechnology Systems Barley Mill Plaza, P24 Wilmington, DE 19898	ODS	Zorbax
Perkin-Elmer Corp. Corporate Office Main Avenue Norwalk, CT 06856	HC-ODS	Sil-X
Waters Associates 34-T Maple St. Milford, MA 01757	u-Bondapak	NH_3 u-Bondapak

TABLE 8.0 TYPICAL RETENTION TIME FOR SELECTIVE PAHs BY HPLC SEPARATION AND DETECTION

Compound	Retention Times (minutes) HPLC Conditions			
	Condition A Fluorescence	UV	Condition B Fluorescence	UV
Acenaphthene		20.5		18.0
Acenaphthylene		18.5		15.8
Anthracene	23.4		21.0	21.0
Benzo(a)anthracene	28.5		26.3	26.3
Benzo(a)pyrene	33.9		31.1	31.1
Benzo(b)fluoranthene	31.6		29.3	29.3
Benzo(e)pyrene			31.1	
Benzo(ghi)perylene	36.3		33.9	33.9
Benzo(k)fluoranthene	32.9		30.2	30.2
Chrysene	29.3		26.7	
Dibenzo(a,h)anthracene	35.7		32.7	32.7
Fluoranthene	24.5		22.5	22.5
Fluorene		21.2	18.5	18.5
Indeno(1,2,3-cd)pyrene	37.4		34.6	34.6
Naphthalene		16.6		14.0
Phenanthrene	22.1		19.9	19.9
Pyrene	25.4		23.4	23.4

Condition A HPLC parameters: Reverse phase HC-ODS sil-X, 5 micron particle size, in a 250-mm x 2.6-mm I.D. stainless steel column. Isocratic elution for 5 min using acetonitrile/water (4:6)(v/v), then linear gradient elution to 100% acetonitrile over 25 min at 0.5 mL/min flow rate. If columns having other internal diameters are used, the flow rate should be adjusted to maintain a linear velocity of 2 mm/sec.

Condition B HPLC parameters: Reverse phase VYDAC 201 TP 5415, 5 micron particle size, in a .46 x 25 cm stainless steel column. Isocratic elution for 10 min using acetonitrile/water (4:6)(v/v), then linear gradient elution to 100% acetonitrile for 10 minutes then linear gradient to 40/60 acetonitrile for 10 minutes at 15 mL/min.

TABLE 9.0. RETENTION TIMES (RT) AND MINIMUM DETECTION LIMITS (MDLs) FOR SELECTED PAHs USING ULTRAVIOLET AND FLUORESCENCE DETECTION

	Ultraviolet Detector		Fluorescence Detector	
PAH	RT	MDL	RT	MDL
Naphthalene	14.0	250pg/uL		
Acenaphthylene	15.85	250pg/uL		
Acenaphthene	18.0	250pg/uL		
Fluorene	18.5	50pg/uL	18.5	5pg/uL
Phenanthrene	19.9	50pg/uL	19.9	10pg/uL
Anthracene	21.0	50pg/uL	21.0	50pg/uL
Fluoranthene	22.5	50pg/uL	22.5	10pg/uL
Pyrene	23.4	50pg/uL	23.4	5pg/uL
Benzo(a)anthracene	26.3	50pg/uL	26.3	5pg/uL
Chrysene	26.7	50pg/uL	26.7	5pg/uL
Benzo(b)fluoranthene	29.3	50pg/uL	29.3	10pg/uL
Benzo(k)fluoranthene	30.2	50pg/uL	30.2	5pg/uL
Benzo(a)pyrene	31.1	50pg/uL	31.1	5pg/uL
Dibenzo(a,h)anthracene	32.7	50pg/uL	32.7	5pg/uL
Benzo(ghi)perylene	33.9	50pg/uL	33.9	5pg/uL
Indeno(1,2,3-cd)pyrene	34.6	50pg/uL	34.6	50pg/uL

RT = Retention time in minutes
MDL = Minimum detection limit

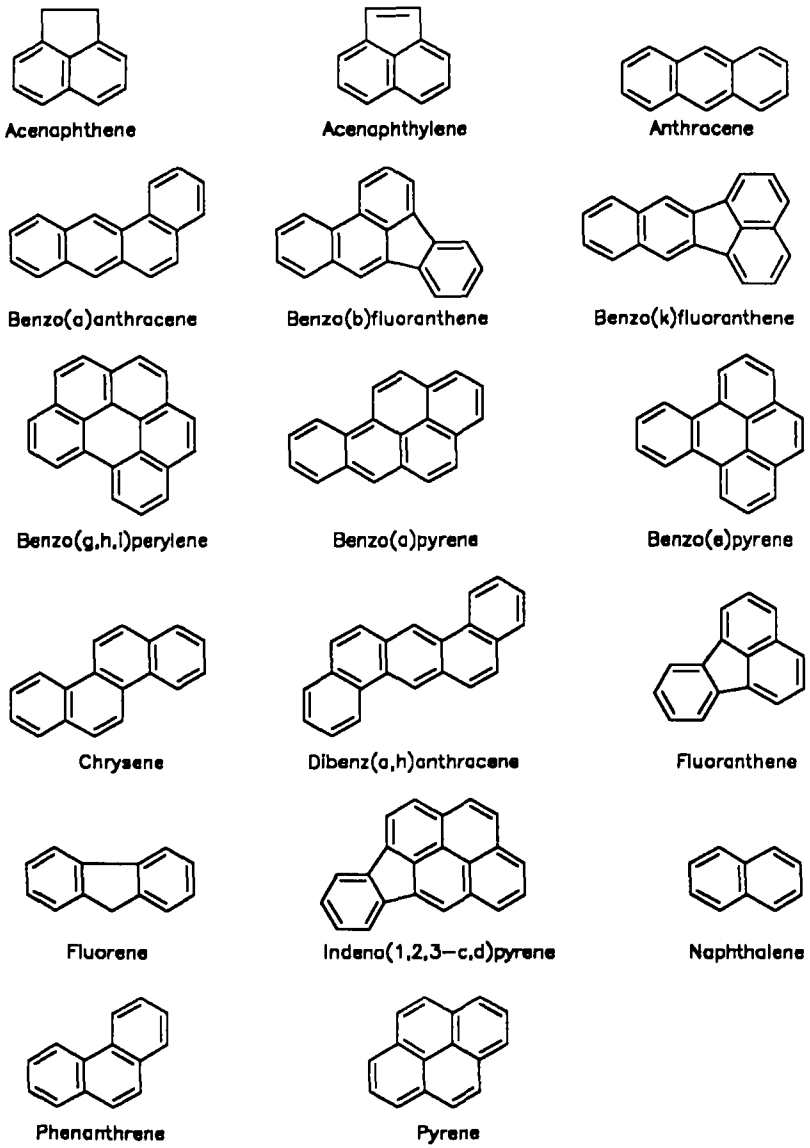


FIGURE 1.0. RING STRUCTURE OF SELECTIVE PAHs.

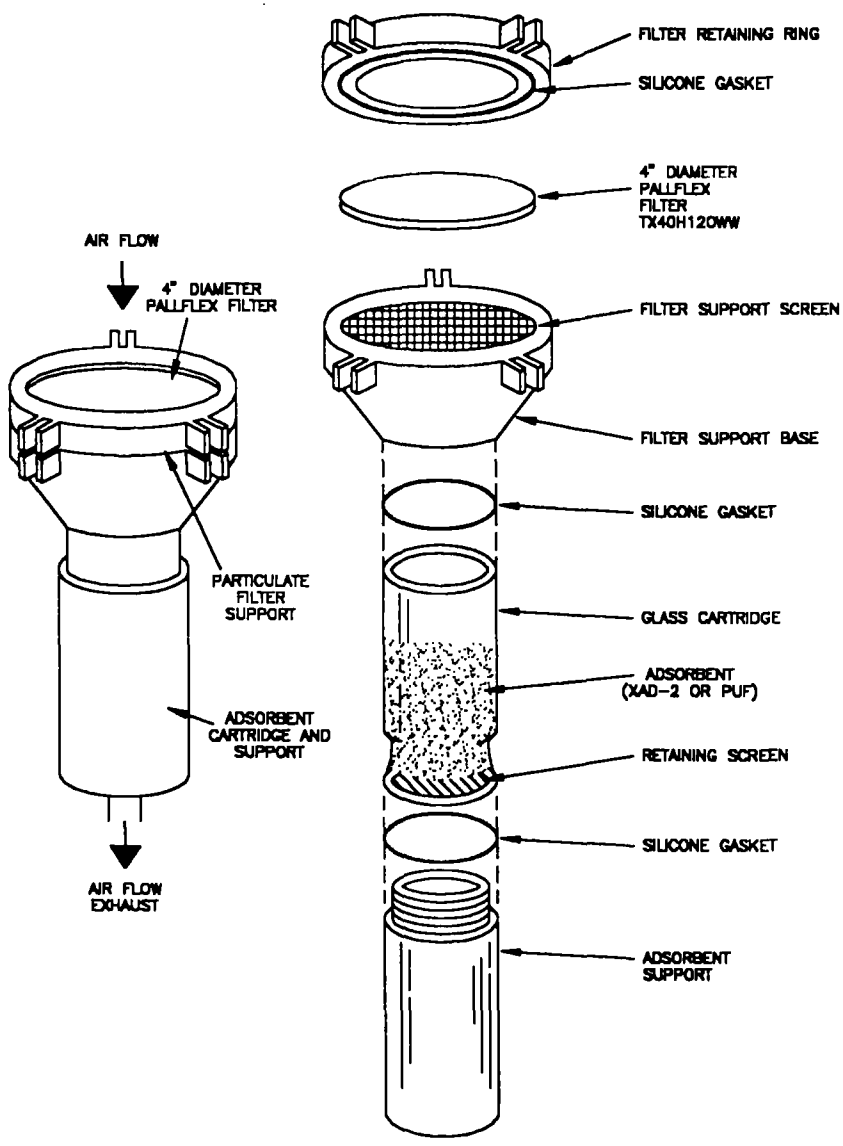
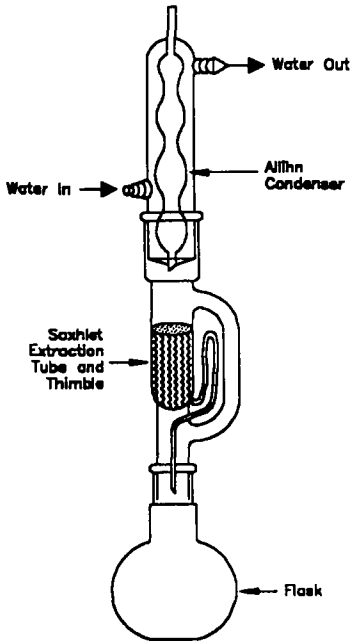
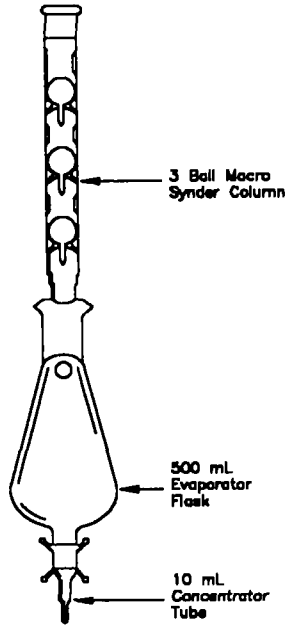


FIGURE 2.0. GENERAL METAL WORKS SAMPLING HEAD

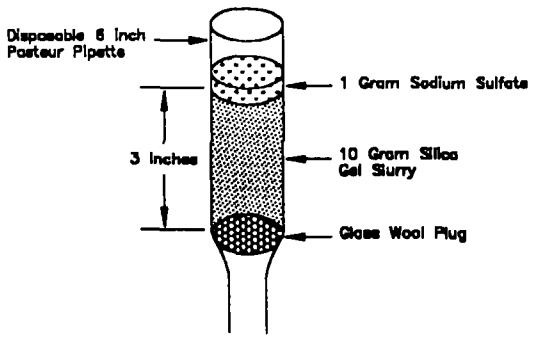
TO13-80



(a) Soxhlet Extraction Apparatus with Allihn Condenser



(b) Kuderna–Danish (K–D) Evaporator with Macro Synder Column



(c) Silica Gel Clean–up Column

FIGURE 3. APPARATUS USES IN SAMPLING ANALYSIS.

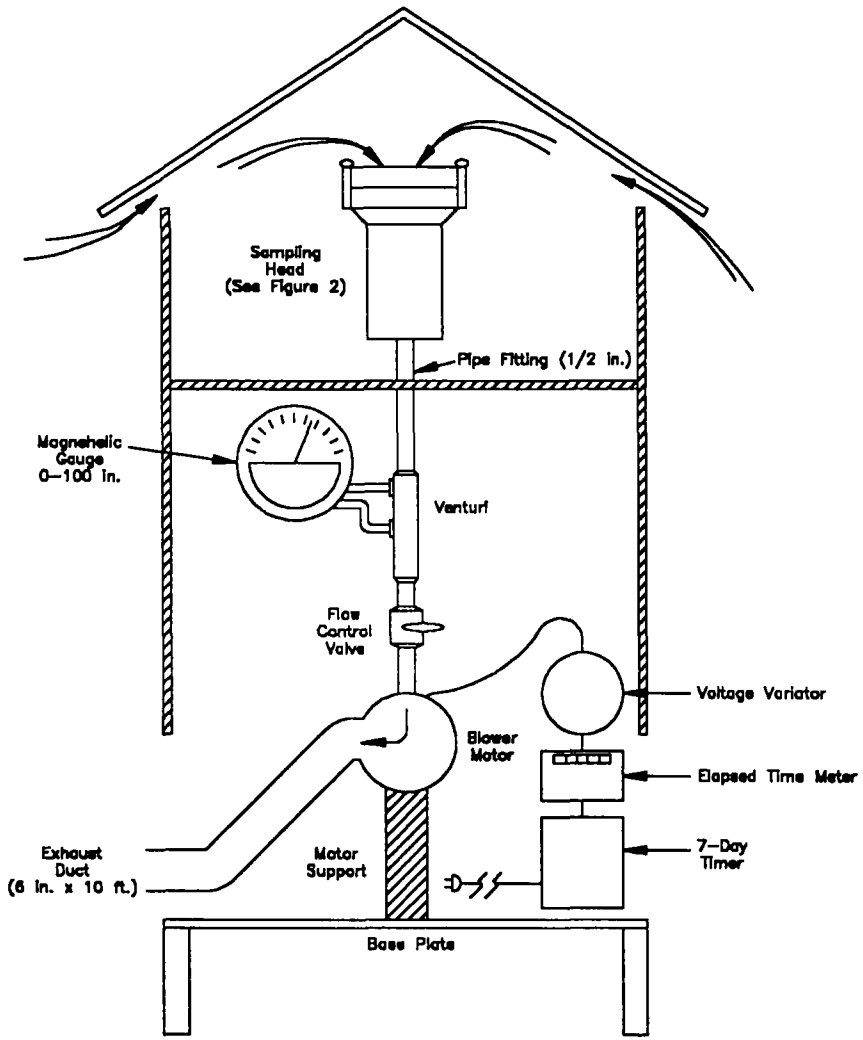


FIGURE 4. HIGH VOLUME AIR SAMPLER

GENERAL METAL WORKS MODEL PS–1 SAMPLER

T013-84

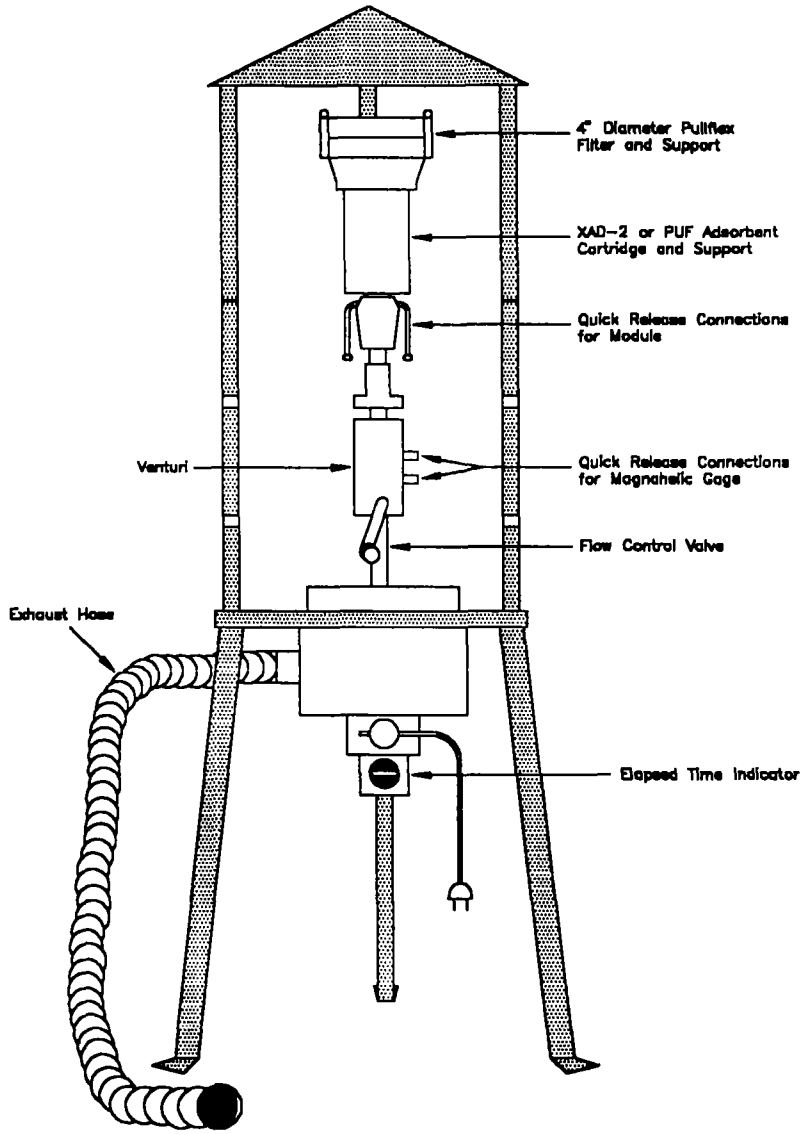


FIGURE 5. PORTABLE HIGH VOLUME AIR SAMPLER

T013-86

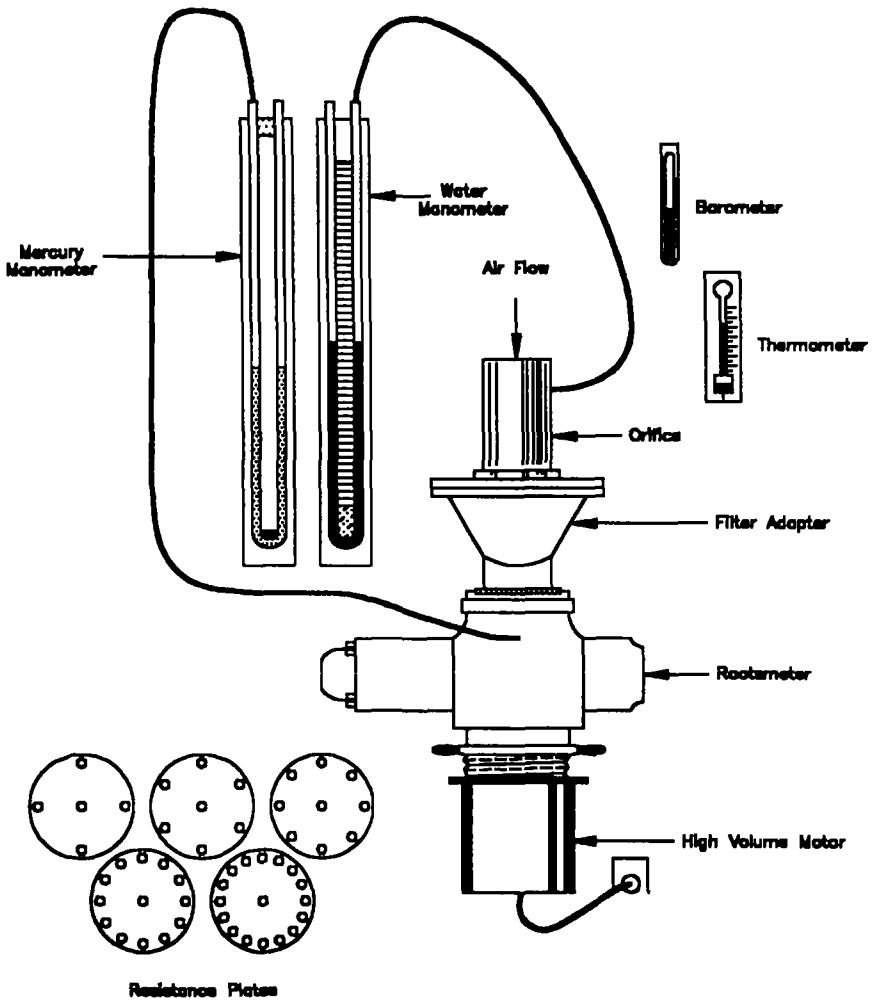


FIGURE 6. LABORATORY ORIFICE CALIBRATION SETUP

METHOD TO14

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

OUTLINE

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APPENDIX A - Availability of Audit Cylinders from U.S. Environmental Protection Agency (USEPA) to USEPA Program/Regional Offices, State/Local Agencies and Their Contractors

APPENDIX B - Operating Procedures for a Portable Gas Chromatograph Equipped With a Photoionization Detector

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METHOD TO14

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (VOCs) IN AMBIENT AIR USING SUMMA® PASSIVATED CANISTER SAMPLING AND GAS CHROMATOGRAPHIC ANALYSIS

1. Scope

1.1 This document describes a procedure for sampling and analysis of volatile organic compounds (VOCs) in ambient air. The method is based on collection of whole air samples in 6-liter, SUMMA® passivated stainless steel canisters. The VOCs are subsequently separated by gas chromatography and measured by multidetector techniques. This method presents procedures for sampling into canisters to final pressures both above and below atmospheric pressure (respectively referred to as pressurized and subatmospheric pressure sampling).

1.2 This method is applicable to VOCs that have been tested and determined to be stable when stored in pressurized and subatmospheric pressure canisters. Numerous compounds, many of which are chlorinated VOCs, have been successfully tested for storage stability in pressurized canisters (1,2). However, minimal documentation is currently available demonstrating stability of VOCs in subatmospheric pressure canisters.

1.3 The organic compounds that have been successfully collected in pressurized canisters by this method are listed in Table 1. These compounds have been successfully measured at the parts per billion by volume (ppbv) level.

2. Applicable Documents

2.1 ASTM Standards

D1356 - Definition of Terms Related to Atmospheric Sampling and Analysis
E260 - Recommended Practice for General Gas Chromatography Procedures
E355 - Practice for Gas Chromatography Terms and Relationships

2.2 Other Documents

U.S. Environmental Protection Agency Technical Assistance Document (3)
Laboratory and Ambient Air Studies (4-17)

3. Summary of Method

3.1 Both subatmospheric pressure and pressurized sampling modes use an initially evacuated canister and a pump-ventilated sample line during sample collection. Pressurized sampling requires an additional pump to provide positive pressure to the sample canister. A sample of ambient air is drawn through a sampling train comprised of components that regulate the rate and duration of sampling into a pre-evacuated SUMMA® passivated canister.

3.2 After the air sample is collected, the SUMMA® passivated canister valve is closed, an identification tag is attached to the canister, and the canister is transported to a predetermined laboratory for analysis.

3.3 Upon receipt at the laboratory, the canister tag data is recorded and the canister is attached to the analytical system. During analysis, water vapor is reduced in the gas stream by a Nafion® dryer (if applicable), then concentrated by collection in a cryogenically-cooled trap. The cryogen is then removed and the temperature of the trap is raised. The VOCs originally collected in the trap are revolatilized, separated on a GC column, then detected by one or more detectors for identification and quantitation.

3.4 The analytical strategy for Method TO14 involves using a high-resolution gas chromatograph (GC) coupled to an appropriate GC detector(s). Historically, detectors for a GC have been divided into two groups: non-specific detectors and specific detectors. The non-specific detectors include, but are not limited to, the nitrogen-phosphorus detector (NPD), the flame ionization detector (FID), the electron capture detector (ECD) and the photoionization detector (PID). The specific detectors include the mass spectrometer (MS) operating in either the select ion mode (SIM) or the SCAN mode, or use of the ion trap detector. The use of these detectors or a combination of these detectors as part of an analytical scheme is based upon the required specificity and sensitivity of the application. While the non-specific detectors are less expensive per analysis and in some cases more sensitive than the specific detector, they vary in specificity and sensitivity for a specific class of compounds. For instance, if multiple halogenated compounds are

targeted, an ECD is usually chosen; if only compounds containing nitrogen or phosphorus are of interest, a NPD can be used; or, if a variety of hydrocarbon compounds are sought, the broad response of the FID or PID is appropriate. In each of these cases, however, the specific identification of the compound within the class is determined only by its retention time, which can be subject to shifts or to interference from other nontargeted compounds. When misidentification occurs, the error is generally a result of a cluttered chromatogram, making peak assignment difficult. In particular, the more volatile organics (benzene, chloroethanes, ethyltoluenes, dichlorobenzenes, and various freons) exhibit less well defined chromatographic peaks, leading to misidentification using non-specific detectors. Quantitative comparisons indicate that the FID is more subject to error than the ECD because the ECD is a much more selective detector for a smaller class of compounds which exhibits a stronger response. Identification errors, however, can be reduced by: (a) employing simultaneous detection by different detectors or (b) correlating retention times from different GC columns for confirmation. In either case, interferences on the non-specific detectors can still cause error in identifying a complex sample. The non-specific detector system (GC-NPD-FID-ECD-PID), however, has been used for approximate quantitation of relatively clean samples. The non-specific detector system can provide a "snapshot" of the constituents in the sample, allowing determination of:

- Extent of misidentification due to overlapping peaks,
- Position of the VOCs within or not within the calibration range of anticipated further analysis by specific detectors (GC-MS-SCAN-SIM) (if not, the sample is further diluted), and
- Existence of unexpected peaks which need further identification by specific detectors.

On the other hand, the use of specific detectors (MS coupled to a GC) allows positive compound identification, thus lending itself to more specificity than the multidetector GC. Operating in the SIM mode, the MS can readily approach the same sensitivity as the

multidetector system, but its flexibility is limited. For SIM operation, the MS is programmed to acquire data for a limited number of targeted compounds while disregarding other acquired information. In the SCAN mode, however, the MS becomes a universal detector, often detecting compounds which are not detected by the multidetector approach. The GC-MS-SCAN will provide positive identification, while the GC-MS-SIM procedure provides quantitation of a restricted "target compound" list of VOCs.

The analyst often must decide whether to use specific or non-specific detectors by considering such factors as project objectives, desired detection limits, equipment availability, cost and personnel capability in developing an analytical strategy.

A list of some of the advantages and disadvantages associated with non-specific and specific detectors may assist the analyst in the decision-making process.

Non-Specific Multidetector Analytical System

Advantages	Disadvantages
o Less expensive than GC-MS	o Multiple detectors to calibrate
o Less sample volume required for analysis	o Compound identification not positive
o More sensitive - ECD may be 1000 times more sensitive than GC-MS	o Lengthy data interpretation (one hour each for analysis and data reduction)
	o Interference(s) from co-eluting compound(s)
	o Cannot identify unknown compounds - outside range of calibration - without standards
	o Does not differentiate targeted compounds from interfering compounds

Specific Detector Analytical System

GC-MS-SIM

Advantages	Disadvantages
o positive compound identification	o can't identify non-specified compounds (ions)
o greater sensitivity than GC-MS-SCAN	o greater cost than multi-detector GC
o less operator interpretation than for multidetector GC	o greater sample volume required than for multidetector GC
o can resolve co-eluting peaks	o universality of detector sacrificed to achieve enhancement in sensitivity
o more specific than the multi-detector GC	

GC-MS-SCAN

o positive compound identification	o lower sensitivity than GC-MS-SIM
o can identify all compounds	o greater sample volume required than for multidetector GC
o less operator interpretation	o greater cost than multi-detector GC
o can resolve co-eluting peaks	

The analytical finish for the measurement chosen by the analyst should provide a definitive identification and a precise quantitation of volatile organics. In a large part, the actual approach to these two objectives is subject to equipment availability. Figure 1 indicates some of the favorite options that are used as an analytical finish. The GC-MS-SCAN option uses a capillary column GC coupled to a MS operated in a scanning mode and supported by spectral library search routines. This option offers the nearest approximation to unambiguous identification and covers a wide range of compounds as defined by the completeness of the spectral library. GC-MS-SIM monitoring mode is limited to a set of target compounds which are user defined and is, however, more sensitive than GC-MS-SCAN by virtue of the longer dwell times at the restricted number of m/z values. Both these techniques, but especially the GC-MS-SIM option, can use a supplemental general non-specific detector to verify/identify the presence of VOCs. Finally, the option labelled GC-multidetector

system uses a combination of retention time and multiple general detector verification to identify compounds. However, interference due to nearly identical retention times can affect system quantitation when using this option.

Due to the low concentrations of VOCs encountered in urban air (typically less than 4 ppbv and the majority below 1 ppbv) along with their complicated chromatograms, Method TO14 uses the specific detectors (GC-MS-SCAN-SIM) for positive identification and for primary quantitation to ensure that high-quality ambient data is acquired.

For the experienced analyst who wishes to limit the analytical system to the non-specific detectors, Section 10.3 does provide guidelines and example chromatograms showing typical retention times and calibration response factors, and utilizing the non-specific detectors (GC-FID-ECD-PID) analytical system as the primary quantitative technique.

4. Significance

4.1 VOCs enter the atmosphere from a variety of sources, including petroleum refineries, synthetic organic chemical plants, natural gas processing plants, and automobile exhaust. Many of these VOCs are acutely toxic; therefore, their determination in ambient air is necessary to assess human health impacts.

4.2 Conventional methods for VOC determination use solid sorbent sampling techniques. The most widely used solid sorbent is Tenax®. An air sample is drawn through a Tenax®-filled cartridge where certain VOCs are trapped on the polymer. The sample cartridge is transferred to a laboratory and analyzed by GC-MS.

4.3 VOCs can also be successfully collected in stainless steel canisters. Collection of ambient air samples in canisters provides (1) convenient integration of ambient samples over a specific time period, (e.g., 24 hours); (2) remote sampling and central analysis; (3) ease of storing and shipping samples; (4) unattended sample collection; (5) analysis of samples from multiple sites with one analytical system; and (6) collection of sufficient sample volume to allow assessment of measurement precision and/or analysis of

samples by several analytical systems. However, care must be exercised in selecting, cleaning, and handling sample canisters and sampling apparatus to avoid losses or contamination of the samples. Contamination is a critical issue with canister-based sampling because the canister is the last element in the sampling train.

4.4 Canisters are treated internally by the SUMMA® passivated process in which a pure chrome-nickel oxide is formed on the interior surface. This type of vessel has been used in the past for sample collection and has demonstrated sample storage stability of many specific organic compounds.

4.5 This method can be applied to sampling and analysis of not only VOCs, but also some selected semivolatile organic compounds (SVOCs). The term "semivolatile organic compounds" is used to broadly describe organic compounds that are too volatile to be collected by filtration air sampling but not volatile enough for thermal desorption from solid sorbents. SVOCs can generally be classified as those with saturation vapor pressures at 25°C between 10^{-1} and 10^{-7} mm Hg. VOCs are generally classified as those organics having saturated vapor pressures at 25° greater than 10^{-1} mm Hg.

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 E355. All abbreviations and symbols within this method are defined at point of use.

5.1 Absolute canister pressure = Pg+Pa, where Pg = gauge pressure in the canister (kPa, psi) and Pa = standard atmospheric pressure (101 kPa, 14.7 psi). [Note: Pg+Pa gives absolute pressure in units; Pg/Pa gives absolute pressure in atmospheres.]

5.2 Absolute pressure - Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPa, mm Hg or psia.

5.3 Cryogen - A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid oxygen (bp -183.0°C) or liquid argon (bp -185.7°C).

5.4 Dynamic calibration - Calibration of an analytical system using calibration gas standard concentrations in a form identical or very similar to the samples to be analyzed into the inlet of the sampling or analytical system in a manner very similar to the normal sampling or analytical process.

5.5 Gauge pressure - Pressure measured above ambient atmospheric pressure (as opposed to absolute pressure). Zero gauge pressure is equal to ambient atmospheric pressure, which at standard conditions is 101 kPa (14.7 psia).

5.6 MS-SCAN - The GC is coupled to a MS programmed in the SCAN mode to scan all ions repeatedly during the GC run. As used in the current context, this procedure serves as a qualitative identification and characterization of the sample.

5.7 MS-SIM - The GC is coupled to a MS programmed to acquire data for only specified ions and to disregard all others. This is performed using SIM coupled to retention time discriminators. The GC-SIM analysis provides quantitative results for selected constituents of the sample gas as programmed by the user.

5.8 Megabore® column - Chromatographic column having an internal diameter (I.D.) of 0.31-0.55 mm.

5.9 Microbore® column - Chromatographic column having an I.D. of 0.31 mm or smaller.

5.10 Pressurized sampling - Collection of an air sample in a canister with a (final) canister pressure above atmospheric pressure, using a sample pump.

5.11 Qualitative accuracy - The ability of an analytical system to correctly identify compounds.

5.12 Quantitative accuracy - The ability of an analytical system to correctly measure the concentration of an identified compound.

5.13 Static calibration - Calibration of an analytical system using standards in a form different than the samples to be analyzed. An example of a static calibration would be injecting a small volume of a high concentration standard directly onto a GC column, bypassing the sample extraction and preconcentration portion of the analytical system.

5.14 Subatmospheric sampling - Collection of an air sample in an evacuated canister at a (final) canister pressure below atmospheric pressure, without the assistance of a sampling pump. The canister is filled as the internal canister pressure increases to ambient or near ambient pressure. An auxiliary vacuum pump may be used as part of the sampling system to flush the inlet tubing prior to or during sample collection.

6. Interferences and Limitations

6.1 Interferences can occur in sample analysis if moisture accumulates in the dryer (see Section 10.1.1.2). An automated cleanup procedure that periodically heats the dryer to about 100°C while purging with zero air eliminates any moisture buildup. This procedure does not degrade sample integrity.

6.2 Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) should be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples. Instructions for cleaning the canisters and certifying the field sampling system are described in Sections 12.1 and 12.2, respectively.

6.3 Because the GC-MS analytical system employs a Nafion® permeable membrane dryer to remove water vapor selectively from the sample stream, polar organic compounds may permeate concurrent with the moisture molecule. Consequently, the analyst should quantitate his or her system with the specific organic constituents under examination.

7. Apparatus

[Note: Subatmospheric pressure and pressurized canister sampling systems are commercially available and have been used as part of U.S. Environmental Protection Agency's Toxics Air Monitoring Stations (TAMS), Urban Toxic Air Pollutant Program (UTAP), and the non-methane organic compound (NMOC) sampling and analysis program.]

7.1 Sample Collection

7.1.1 Subatmospheric Pressure (Figure 2 Without Metal Bellows Type Pump)

7.1.1.1 Sampling inlet line - stainless steel tubing to connect the sampler to the sample inlet.

7.1.1.2 Sample canister - Leak-free stainless steel pressure vessels of desired volume (e.g., 6 L), with valve and SUMMA® passivated interior surfaces, (Scientific Instrumentation Specialists, Inc., P.O. Box 8941, Moscow, ID 83843, or Anderson Samplers, Inc., 4215-C Wendell Dr., Atlanta, GA, 30336), or equivalent.

7.1.1.3 Stainless steel vacuum/pressure gauge - capable of measuring vacuum (-100 to 0 kPa or 0 to 30 in Hg) and pressure (0-206 kPa or 0-30 psig) in the sampling system (Matheson, P.O. Box 136, Morrow, GA 30200, Model 63-3704), or equivalent. Gauges should be tested clean and leak tight.

7.1.1.4 Electronic mass flow controller - capable of maintaining a constant flow rate (± 10%) over a sampling period of up to 24 hours and under conditions of changing temperature (20-40°C) and humidity (Tylan Corp., 19220 S. Normandie Ave., Torrance, CA 90502, Model FC-260), or equivalent.

7.1.1.5 Particulate matter filter - 2 um sintered stainless steel in-line filter (Nupro Co., 4800 E. 345th St., Willoughby, OH 44094, Model SS-2F-K4-2), or equivalent.

7.1.1.6 Electronic timer - for unattended sample collection (Paragon Elect. Co., 606 Parkway Blvd., P.O. Box 23, Twin Rivers, WI 54201, Model 7008-00), or equivalent.

7.1.1.7 Solenoid valve - electrically-operated, bi-stable solenoid valve (Skinner Magnelatch Valve, New Britain, CT, Model V5RAM49710), or equivalent, with Viton® seat and o-rings.

7.1.1.8 Chromatographic grade stainless steel tubing and fittings - for interconnections (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #3125),

or equivalent. All such materials in contact with sample, analyte, and support gases prior to analysis should be chromatographic grade stainless steel.

7.1.1.9 Thermostatically controlled heater - to maintain temperature inside insulated sampler enclosure above ambient temperature (Watlow Co., Pfafftown, NC, Part 04010080), or equivalent.

7.1.1.10 Heater thermostat - automatically regulates heater temperature (Elmwood Sensors, Inc., 500 Narragansett Park Dr., Pawtucket RI 02861, Model 3455-RC-0100-0222), or equivalent.

7.1.1.11 Fan - for cooling sampling system (EG&G Rotron, Woodstock, NY, Model SUZAI), or equivalent.

7.1.1.12 Fan thermostat - automatically regulates fan operation (Elmwood Sensors, Inc., Pawtucket, RI, Model 3455-RC-0100-0244), or equivalent.

7.1.1.13 Maximum-minimum thermometer - records highest and lowest temperatures during sampling period (Thomas Scientific, Brooklyn Thermometer Co., Inc., P/N 9327H30), or equivalent.

7.1.1.14 Nupro stainless steel shut-off valves - leak free, for vacuum/pressure gauge.

7.1.1.15 Auxiliary vacuum pump - continuously draws ambient air to be sampled through the inlet manifold at 10 L/min. or higher flow rate. Sample is extracted from the manifold at a lower rate, and excess air is exhausted. [Note: The use of higher inlet flow rates dilutes any contamination present in the inlet and reduces the possibility of sample contamination as a result of contact with active adsorption sites on inlet walls.]

7.1.1.16 Elapsed time meter - measures duration of sampling (Conrac, Cramer Div., Old Saybrook, CT, Type 6364, P/N 10082), or equivalent.

7.1.1.17 Optional fixed orifice, capillary, or adjustable micrometering valve - may be used in lieu of the electronic flow controller for grab samples or short duration time-integrated samples. Usually appropriate only in situations where screening samples are taken to assess future sampling activity.

7.1.2 Pressurized (Figure 2 With Metal Bellows Type Pump and Figure

7.1.2.1 Sample pump - stainless steel, metal bellows type (Metal Bellows Corp., 1075 Providence Highway, Sharon, MA 02067, Model MB-151), or equivalent, capable of 2 atmospheres output pressure. Pump must be free of leaks, clean, and uncontaminated by oil or organic compounds. [Note: An alternative sampling system has been developed by Dr. R. Rasmussen, The Oregon Graduate Center (18-19) and is illustrated in Figure 3. This flow system uses, in order, a pump, a mechanical flow regulator, and a mechanical compensating flow restrictive device. In this configuration the pump is purged with a large sample flow, thereby eliminating the need for an auxiliary vacuum pump to flush the sample inlet. Interferences using this configuration have been minimal.]

7.1.2.2 Other supporting materials - all other components of the pressurized sampling system (Figure 2 with metal bellows type pump and Figure 3) are similar to components discussed in Sections 7.1.1.1 through 7.1.1.16.

7.2 Sample Analysis

7.2.1 GC-MS-SCAN Analytical System [Figure 4]

7.2.1.1 The GC-MS-SCAN analytical system must be capable of acquiring and processing data in the MS-SCAN mode.

7.2.1.2 Gas chromatograph (flame ionization detector optional) must be capable of sub-ambient temperature programming (Hewlett Packard, Rt. 41, Avondale, PA 19311, HP Model 5880A GC, Level 4 BASIC programming with oven temperature control), or equivalent. Convenient features such as a temperature-controlled oven and automatic control of the integrator, valve, and flow regulators are provided by this GC.

7.2.1.3 Chromatographic detector - mass selective detector (Hewlett Packard, 3000-T Hanover St., 9B, Palo Alto, CA 94304, Model HP-5970 MS), or equivalent, equipped with computer and appropriate software (Hewlett-Packard, 3000-T Hanover St., 9B, Palo Alto, CA 94304,

HP-216 Computer, Quicksilver MS software, Pascal 3.0, mass storage 9133 HP Winchester with 3.5 inch floppy disk), or equivalent. The GC-MS is set in the SCAN mode, where the MS screens the sample for identification and quantitation of VOC species.

7.2.1.4 Cryogenic trap with temperature control assembly - Refer to Section 10.1.1.3 for complete description of trap and temperature control assembly (Nutech Corporation, 2142 Geer St., Durham, NC, 27704, Model 320-01), or equivalent.

7.2.1.5 Electronic mass flow controllers (3) - maintain constant flow (for carrier gas and sample gas) and to provide analog output to monitor flow anomalies (Tylan Model 260, 0-100 cm^3/min), or equivalent.

7.2.1.6 Vacuum pump - general purpose laboratory pump, capable of drawing the desired sample volume through the cryogenic trap (Metal Bellows Corp., 1075 Providence Highway, MA 02067, Model MB-150), or equivalent.

7.2.1.7 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings - refer to Section 7.1.1.8 for description.

7.2.1.8 Chromatographic column - Microbore® OV-1 capillary column, 0.32 mm I.D., HP crosslinked methyl silicone (0.88 um film thickness), 50 m long (Hewlett-Packard, Rt. 41, Avondale, PA 19311), or equivalent.

7.2.1.9 Stainless steel vacuum/pressure gauge (optional) - capable of measuring vacuum (-101.3 to 0 kPa) and pressure (0-206 kPa) in the sampling system (Matheson, P.O. Box 136, Morrow, GA 30200, Model 63-3704, or equivalent). Gauges should be tested clean and leak tight.

7.2.1.10 Cylinder pressure stainless steel regulators - standard two-stage cylinder regulators with pressure gauges for helium, zero air and hydrogen gas cylinders.

7.2.1.11 Gas purifiers (3) - used to remove organic impurities and moisture from gas streams (Hewlett Packard, Rt. 41, Avondale, PA, 19311, P/N 19362 - 60500), or equivalent.

7.2.1.12 Low dead-volume tee (optional) - used to split the exit flow from the GC column (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #5839), or equivalent.

7.2.1.13 Nafion® dryer - consisting of Nafion tubing co-axially mounted within larger tubing (Perma Pure Products, 8 Executive Drive, Toms River, NJ, 08753, Model MD-125-48) or equivalent. Refer to Section 10.1.1.2 for description.

7.2.1.14 Six-port gas chromatographic valve - (Seismograph Service Corp, Tulsa, OK, Seiscor Model VIII), or equivalent.

7.2.1.15 Chart recorder (optional) - compatible with the detector output signals to record optional FID detector response to the sample.

7.2.1.16 Electronic integrator (optional) - compatible with the detector output signal of the FID and capable of integrating the area of one or more response peaks and calculating peak areas corrected for baseline drift.

7.2.2 GC-MS-SIM Analytical System [Figure 4]

7.2.2.1 The GC-MS-SIM analytical system must be capable of acquiring and processing data in the MS-SIM mode.

7.2.2.2 All components of the GC-MS-SIM system are identical to Sections 7.2.1.2 through 7.2.1.16.

7.2.3 GC-Multidetector Analytical System [Figure 5 and Figure 6]

7.2.3.1 Gas chromatograph with flame ionization, electron capture and photoionization detectors - must be capable of sub-ambient temperature programming and simultaneous flame ionization, electron capture and photoionization detection (Hewlett Packard, Rt. 41, Avondale, PA 19311, HP Model 5880A GC, Level 4 BASIC programming with oven temperature control, or equivalent). The HP Model 5880A - Level 4 GC provides convenient features such as temperature-controlled oven, automatic control of the integrator, valve and flow regulators.

7.2.3.2 Chart recorders - compatible with the detector output signals to record detector response to the sample.

7.2.3.3 Electronic integrator - compatible with the detector output signals and capable of integrating the area of one or more response peaks and calculating peak areas corrected for baseline drift.

7.2.3.4 Six-port gas chromatographic valve - (Seismograph Service Corp, Tulsa, OK, Seiscor Model VIII), or equivalent.

7.2.3.5 Cryogenic trap with temperature control assembly - Refer to Section 10.1.1.3 for complete description of trap and temperature control assembly (Nutech Corporation, 2142 Geer St., Durham, NC 27704, Model 320-01), or equivalent.

7.2.3.6 Electronic mass flow controllers (3) - maintain constant flow (for carrier gas, nitrogen make-up gas and sample gas) and to provide analog output to monitor flow anomalies (Tylan Model 260, 0-100 cm^3/min), or equivalent.

7.2.3.7 Vacuum pump - general purpose laboratory pump, capable of drawing the desired sample volume through the cryogenic trap (Metal Bellows Corp., 1075 Providence Highway, MA 02067, Model MB-150), or equivalent.

7.2.3.8 Chromatographic grade stainless steel tubing and stainless steel plumbing fittings - refer to Section 7.1.1.8 for description.

7.2.3.9 Chromatographic column - Megabore® OV-1 capillary column, 0.53 mm I.D., HP crosslinked methyl silicone (0.88 um film thickness), 30 m long (Hewlett-Packard, Rt. 41, Avondale, PA 19311) or equivalent. [Note: Other columns (e.g., DB-684) can be used as long as the system meets user needs. The Megabore® column, being wider than the Microbore® column, does not lend itself to plugging as a result of trapped water, thus eliminating the need for a Nafion® dryer in the analytical system. The Megabore® column has sample capacity approaching that of a packed column, while retaining much of the peak resolution traits of a Microbore® column.]

7.2.3.10 Vacuum/pressure gauges (3) - refer to Section 7.2.1.9 for description.

7.2.3.11 Cylinder pressure stainless steel regulators - standard, two-stage cylinder regulators with pressure gauges for helium, zero air, nitrogen, and hydrogen gas cylinders.

7.2.3.12 Gas purifiers (4) - used to remove organic impurities and moisture from gas streams (Hewlett-Packard, Rt. 41, Avondale, PA, 19311, P/N 19362 - 60500), or equivalent.

7.2.3.13 Low dead-volume tee - used to split (50/50) the exit flow from the GC column (Alltech Associates, 2051 Waukegan Rd., Deerfield, IL 60015, Cat. #5839), or equivalent.

7.3 Canister Cleaning System [Figure 7]

7.3.1 Vacuum pump - capable of evacuating sample canister(s) to an absolute pressure of <0.05 mm Hg.

7.3.2 Manifold - stainless steel manifold with connections for simultaneously cleaning several canisters.

7.3.3 Shut-off valve(s) - seven (7) on-off toggle valves.

7.3.4 Stainless steel vacuum gauge - capable of measuring vacuum in the manifold to an absolute pressure of <0.05 mm Hg or less.

7.3.5 Cryogenic trap (2 required) - stainless steel U-shaped open tubular trap cooled with liquid oxygen or argon to prevent contamination from back diffusion of oil from vacuum pump and to provide clean, zero air to sample canister(s).

7.3.6 Stainless steel pressure gauges (2) - 0-345 kPa (0-50 psig) to monitor zero air pressure.

7.3.7 Stainless steel flow control valve - to regulate flow of zero air into canister(s).

7.3.8 Humidifier - pressurizable water bubbler containing high performance liquid chromatography (HPLC) grade deionized water or other system capable of providing moisture to the zero air supply.

7.3.9 Isothermal oven (optional) for heating canisters (Fisher Scientific, Pittsburgh, PA, Model 349), or equivalent.

7.4 Calibration System and Manifold [Figure 8]

7.4.1 Calibration manifold - glass manifold, (1.25 cm I.D. x 66 cm) with sampling ports and internal baffles for flow disturbance to ensure proper mixing.

7.4.2 Humidifier - 500 mL impinger flask containing HPLC grade deionized water.

7.4.3 Electronic mass flow controllers - one (1) 0-to 5-L/min and one 0-to 50-cm^3/min, (Tylan Corporation, 23301-TS Wilmington Ave., Carson, CA, 90745, Model 2160 or equivalent).

7.4.4 Teflon® filter(s) - 47mm Teflon® filter for particulate control, best source.

8. Reagents and Materials

8.1 Gas cylinders of helium, hydrogen, nitrogen, and zero air - ultrahigh purity grade, best source.

8.2 Gas calibration standards - cylinder(s) containing approximately 10 ppmv of each of the following compounds of interest:

vinyl chloride
vinylidene chloride
1,1,2-trichloro-1,2,2-trifluoroethane
chloroform
1,2-dichloroethane
benzene
toluene
Freon 12
methyl chloride
1,2-dichloro-1,1,2,2-tetrafluoroethane
methyl bromide
ethyl chloride
Freon 11
dichloromethane
1,1-dichloroethane
cis-1,2-dichloroethylene
1,2-dichloropropane
1,1,2-trichloroethane
1,2-dibromoethane
tetrachloroethylene
chlorobenzene
benzyl chloride
hexachloro-1,3-butadiene
methyl chloroform
carbon tetrachloride
trichloroethylene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethylbenzene
o-xylene
m-xylene
p-xylene
styrene
1,1,2,2-tetrachloroethane
1,3,5-trimethylbenzene
1,2,4-trimethylbenzene
m-dichlorobenzene
o-dichlorobenzene
p-dichlorobenzene
1,2,4-trichlorobenzene

The cylinder(s) should be traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to a NBS/EPA approved Certified Reference Material (CRM). The components may be purchased in one cylinder or may be separated into different cylinders. Refer to manufacturer specification for guidance on purchasing and mixing VOCs in gas cylinders. Those compounds purchased should match one's own target list.

8.3 Cryogen - liquid oxygen (bp -183.0°C), or liquid argon (bp -185.7°C), best source.

8.4 Gas purifiers - connected in-line between hydrogen, nitrogen, and zero air gas cylinders and system inlet line, to remove moisture and organic impurities from gas streams (Alltech Associates, 2051 Waukegan Road, Deerfield, IL, 60015), or equivalent.

8.5 Deionized water - high performance liquid chromatography (HPLC) grade, ultrahigh purity (for humidifier), best source.

8.6 4-bromofluorobenzene - used for tuning GC-MS, best source.

8.7 Hexane - for cleaning sampling system components, reagent grade, best source.

8.8 Methanol - for cleaning sampling system components, reagent grade, best source.

9. Sampling System

9.1 System Description

9.1.1 Subatmospheric Pressure Sampling [Figure 2 (without metal bellows type pump)]

9.1.1.1 In preparation for subatmospheric sample collection in a canister, the canister is evacuated to 0.05 mm Hg. When opened to the atmosphere containing the VOCs to be sampled, the differential pressure causes the sample to flow into the canister. This technique may be used to collect grab samples (duration of 10 to 30 seconds) or time-integrated samples (duration of 12 to 24 hours) taken through a flow-restrictive inlet (e.g., mass flow controller, critical orifice).

9.1.1.2 With a critical orifice flow restrictor, there will be a decrease in the flow rate as the pressure approaches atmospheric. However, with a mass flow controller, the subatmospheric sampling system can maintain a constant flow rate from full vacuum to within about 7 kPa (1.0 psi) or less below ambient pressure.

9.1.2 Pressurized Sampling [Figure 2 (with metal bellow type pump)]

9.1.2.1 Pressurized sampling is used when longer-term integrated samples or higher volume samples are required. The sample is collected in a canister using a pump and flow control arrangement to achieve a typical 103-206 kPa (15-30 psig) final canister pressure. For example, a 6-liter evacuated canister can be filled at 10 cm^3/min for 24 hours to achieve a final pressure of about 144 kPa (21 psig).

9.1.2.2 In pressurized canister sampling, a metal bellows type pump draws in ambient air from the sampling manifold to fill and pressurize the sample canister.

9.1.3 All Samplers

9.1.3.1 A flow control device is chosen to maintain a constant flow into the canister over the desired sample period. This flow rate is determined so the canister is filled (to about 88.1 kPa for subatmospheric pressure sampling or to about one atmosphere above ambient pressure for pressurized sampling) over the desired sample period. The flow rate can be calculated by

$$F = \frac{P \times V}{T \times 60}$$

where:

F = flow rate (cm3/min).

P = final canister pressure, atmospheres absolute. P is approximately equal to

$$\frac{\text{kPa}_{\text{gauge}}}{101.2} + 1$$

V = volume of the canister (cm^3).

T = sample period (hours).

For example, if a 6-L canister is to be filled to 202 kPa (2 atmospheres) absolute pressure in 24 hours, the flow rate can be calculated by

$$F = \frac{2 \times 6000}{24 \times 60} = 8.3 \text{ cm}^3/\text{min}$$

9.1.3.2 For automatic operation, the timer is wired to start and stop the pump at appropriate times for the desired sample period. The timer must also control the solenoid valve, to open the valve when starting the pump and close the valve when stopping the pump.

9.1.3.3 The use of the Skinner Magnelatch valve avoids any substantial temperature rise that would occur with a conventional, normally closed solenoid valve that would have to be energized during the entire sample period. The temperature rise in the valve could cause outgassing of organic compounds from the Viton valve seat material. The Skinner Magnelatch valve requires only a brief electrical pulse to open or close at the appropriate start and stop times and therefore experiences no temperature increase. The pulses may be obtained either with an electronic timer that can be programmed for short (5 to 60 seconds) ON periods, or with a conventional mechanical timer and a special pulse circuit. A simple electrical pulse circuit for operating the Skinner Magnelatch solenoid valve with a conventional mechanical timer is illustrated in Figure 9(a). However, with this simple circuit, the valve may operate unreliably during brief power interruptions or if the timer is manually switched on and off too fast. A better circuit incorporating a time-delay relay to provide more reliable valve operation is shown in Figure 9(b).

9.1.3.4 The connecting lines between the sample inlet and the canister should be as short as possible to minimize their volume. The flow rate into the canister should remain relatively constant over the entire sampling period. If a critical orifice is used, some drop in the flow rate may occur near the end of the sample period as the canister pressure approaches the final calculated pressure.

9.1.3.5 As an option, a second electronic timer (see Section 7.1.1.6) may be used to start the auxiliary pump several hours prior to the sampling period to flush and condition the inlet line.

9.1.3.6 Prior to field use, each sampling system must pass a humid zero air certification (see Section 12.2.2). All plumbing should be checked carefully for leaks. The canisters must also pass a humid zero air certification before use (see Section 12.1).

9.2 Sampling Procedure

9.2.1 The sample canister should be cleaned and tested according to the procedure in Section 12.1.

9.2.2 A sample collection system is assembled as shown in Figure 2 (and Figure 3) and must meet certification requirements as outlined in Section 12.2.3. [Note: The sampling system should be contained in an appropriate enclosure.]

9.2.3 Prior to locating the sampling system, the user may want to perform "screening analyses" using a portable GC system, as outlined in Appendix B, to determine potential volatile organics present and potential "hot spots." The information gathered from the portable GC screening analysis would be used in developing a monitoring protocol, which includes the sampling system location, based upon the "screening analysis" results.

9.2.4 After "screening analysis," the sampling system is located. Temperatures of ambient air and sampler box interior are recorded on canister sampling field data sheet (Figure 10). [Note: The following discussion is related to Figure 2.]

9.2.5 To verify correct sample flow, a "practice" (evacuated) canister is used in the sampling system. [Note: For a subatmospheric sampler, the flow meter and practice canister are needed. For the pump-driven system, the practice canister is not needed, as the flow can be measured at the outlet of the system.] A certified mass flow meter is attached to the inlet line of the manifold, just in front of the filter. The canister is opened. The sampler is turned on and the reading of the certified mass flow meter is compared to the sampler mass flow controller. The values should agree within ±10%. If not, the sampler mass flow meter needs to be recalibrated or there is a leak in the system. This should be investigated and corrected. [Note: Mass flow meter readings may drift. Check the zero reading carefully and add or subtract the zero reading when reading or adjusting the sampler flow rate, to compensate for any zero drift.] After two minutes, the desired canister flow rate is adjusted to the proper value (as indicated by the certified mass flow meter) by the sampler flow control unit controller (e.g., 3.5 cm^3/min for 24 hr, 7.0 cm^3/min for 12 hr). Record final flow under "CANISTER FLOW RATE," Figure 10.

9.2.6 The sampler is turned off and the elapsed time meter is reset to 000.0. Note: Any time the sampler is turned off, wait at least 30 seconds to turn the sampler back on.

9.2.7 The "practice" canister and certified mass flow meter are disconnected and a clean certified (see Section 12.1) canister is attached to the system.

9.2.8 The canister valve and vacuum/pressure gauge valve are opened.

9.2.9 Pressure/vacuum in the canister is recorded on the canister sampling field data sheet (Figure 10) as indicated by the sampler vacuum/pressure gauge.

9.2.10 The vacuum/pressure gauge valve is closed and the maximum-minimum thermometer is reset to current temperature. Time of day and elapsed time meter readings are recorded on the canister sampling field data sheet.

9.2.11 The electronic timer is set to begin and stop the sampling period at the appropriate times. Sampling commences and stops by the programmed electronic timer.

9.2.12 After the desired sampling period, the maximum, minimum, current interior temperature and current ambient temperature are recorded on the sampling field data sheet. The current reading from the flow controller is recorded.

9.2.13 At the end of the sampling period, the vacuum/pressure gauge valve on the sampler is briefly opened and closed and the pressure/vacuum is recorded on the sampling field data sheet. Pressure should be close to desired pressure. [Note: For a subatmospheric sampling system, if the canister is at atmospheric pressure when the field final pressure check is performed, the sampling period may be suspect. This information should be noted on the sampling field data sheet.] Time of day and elapsed time meter readings are also recorded.

9.2.14 The canister valve is closed. The sampling line is disconnected from the canister and the canister is removed from the system. For a subatmospheric system, a certified mass flow meter is once again connected to the inlet manifold in front of the in-line filter and a "practice" canister is attached to the Magnelatch valve of the sampling system. The final flow rate is recorded on the canister sampling field data sheet (see Figure 10). [Note: For a pressurized system, the final flow may be measured directly.] The sampler is turned off.

9.2.15 An identification tag is attached to the canister. Canister serial number, sample number, location, and date are recorded on the tag.

10. Analytical Systems [See Figures 4, 5 and 6]

10.1 System Description

10.1.1 GC-MS-SCAN System

10.1.1.1 The analytical system is comprised of a GC equipped with a mass selective detector set in the SCAN mode [see Figure 4]. All ions are scanned by the MS repeatedly during the

GC run. The system includes a computer and appropriate software for data acquisition, data reduction, and data reporting. A 400 cm^3 air sample is collected from the canister into the analytical system. The sample air is first passed through a Nafion® dryer, through the 6-port chromatographic valve, then routed into a cryogenic trap. [Note: While the GC-multidetector analytical system does not employ a Nafion® dryer for drying the sample gas stream, it is used here because the GC-MS system utilizes a larger sample volume and is far more sensitive to excessive moisture than the GC-multidetector analytical system. Moisture can adversely affect detector precision. The Nafion® dryer also prevents freezing of moisture on the Microbore® column, which may cause column blockage and possible breakage.] The trap is heated (-160°C to 120°C in 60 sec) and the analyte is injected onto the Microbore® OV-1 GC capillary column. [Note: Rapid heating of the trap provides efficient transfer of the sample components onto the gas chromatographic column.] Upon sample injection onto the column, the MS computer is signaled by the GC computer to begin detection of compounds which elute from the column. The gas stream from the GC is scanned within a preselected range of atomic mass units (amu). For detection of compounds in Table 1, the range should be 18 to 250 amu, resulting in a 1.5 Hz repetition rate. Six (6) scans per eluting chromatographic peak are provided at this rate. The 10-15 largest peaks are chosen by an automated data reduction program, the three scans nearest the peak apex are averaged, and a background subtraction is performed. A library search is then performed and the top ten best matches for each peak are listed. A qualitative characterization

of the sample is provided by this procedure. A typical chromatogram of VOCs determined by GC-MS SCAN is illustrated in Figure 11(a).

10.1.1.2 A Nafion® permeable membrane dryer is used to remove water vapor selectively from the sample stream. The permeable membrane consists of Nafion® tubing (a copolymer of tetrafluoroethylene and fluorosulfonyl monomer) that is coaxially mounted within larger tubing. The sample stream is passed through the interior of the Nafion® tubing, allowing water (and other light, polar compounds) to permeate through the walls into a dry air purge stream flowing through the annular space between the Nafion® and outer tubing. [Note: To prevent excessive moisture build-up and any memory effects in the dryer, a clean-up procedure involving periodic heating of the dryer (100°C for 20 minutes) while purging with dry zero air (500 cm^3/min) should be implemented as part of the user's SOP manual. The clean-up procedure is repeated during each analysis (see Section 15, notation 7). Recent studies have indicated no substantial loss of targeted VOCs utilizing the above clean-up procedure (7). This cleanup procedure is particularly useful when employing cryogenic preconcentration of VOCs with subsequent GC analysis using a Microbore® column because excess accumulated water can cause trap and column blockage and also adversely affect detector precision. In addition, the improvement in water removal from the sampling stream will allow analyses of much larger volumes of sample air in the event that greater system sensitivity is required for targeted compounds.]

10.1.1.3 The packed metal tubing used for reduced temperature trapping of VOCs is shown in Figure 12. The cooling unit is comprised of a 0.32 cm outside diameter (O.D.) nickel tubing loop packed with 60-80 mesh Pyrex® beads (Nutech Model 320-01, or equivalent). The nickel tubing loop is wound onto a cylindrically formed tube heater (250 watt). A cartridge heater (25 watt) is sandwiched between pieces of aluminum plate at the trap inlet and outlet to provide additional heat to eliminate cold spots in the transfer tubing. During operation, the trap is inside a two-section stainless steel shell which is well insulated. Rapid heating (-150 to +100°C in 55 s) is accomplished by direct thermal contact between the heater and the trap tubing. Cooling is achieved by vaporization of the cryogen (liquified nitrogen). In the shell, efficient cooling (+120 to -150°C in 225 s) is facilitated by confining the vaporized cryogen to the small open volume surrounding the trap assembly. The trap assembly and chromatographic valve are mounted on a baseplate fitted into the injection and auxiliary zones of the GC on an insulated pad directly above the column oven when used with the Hewlett-Packard 5880 GC. [Note: Alternative trap assembly and connection to the GC may be used depending upon user's requirements.] The carrier gas line is connected to the injection end of the analytical column with a zero-dead-volume fitting that is usually held in the heated zone above the GC oven. A 15 cm x 15 cm x 24 cm aluminum box is fitted over the sample handling elements to complete the package. Vaporized cryogen is vented through the top of the box.

10.1.1.4 As an option, the analyst may wish to split the gas stream exiting the column with a low dead-volume tee, passing one-third of the sample gas (1.0 mL/min) to the mass selective detector and the remaining two-thirds (~2.0 mL/min) through a flame ionization detector, as illustrated as an option in Figure 4. The use of the specific detector (MS-SCAN) coupled with the non-specific detector (FID) enables enhancement of data acquired from a single analysis. In particular, the FID provides the user:

- o Semi-real time picture of the progress of the analytical scheme;
- o Confirmation by the concurrent MS analysis of other labs that can provide only FID results; and
- o Ability to compare GC-FID with other analytical laboratories with only GC-FID capability.

10.1.2 GC-MS-SIM System

10.1.2.1 The analytical system is comprised of a GC equipped with a Microbore® OV-1 capillary column and a mass selective detector set in the SIM mode [see Figure 4]. The GC-MS is set up for automatic, repetitive analysis. The system is programmed to acquire data for only the target compounds and to disregard all others. The sensitivity is 0.1 ppbv for a 250 cm^3 air sample with analytical precision of about 5% relative standard deviation. Concentration of compounds based upon a previously installed calibration table is reported by an automated data reduction program. A Nafion® dryer is also employed by this analytical system prior to cryogenic preconcentration; therefore, many polar compounds are not identified by this procedure.

10.1.2.2 SIM analysis is based on a combination of retention times and relative abundances of selected ions (see Table 2). These qualifiers are stored on the hard disk of the GC-MS 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, except for vinyl chloride and methylene chloride, which is determined to be ± 25%. Three ions are measured for most of the forty compounds. When compound identification is made by the computer, any peak that fails any of the qualifying tests is flagged (e.g., with an *). All 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. While this 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. A typical chromatogram of VOCs determined by GC-MS-SIM mode is illustrated in Figure 11(b).

10.1.3 GC-Multidetector (GC-FID-ECD) System with Optional PID

10.1.3.1 The analytical system (see Figure 5) is comprised of a gas chromatograph equipped with a capillary column and electron capture and flame ionization detectors (see Figure 5). In typical operation, sample air is vented past the inlet to the analytical system from the canister at a flow rate of 75 cm^3/min. For analysis, only 35 cm^3/min of sample gas is

used, while excess is vented to the atmosphere. The sample gas stream is routed through a six port chromatographic valve and into the cryogenic trap for a total sample volume of 490 cm^3. [Note: This represents a 14 minute sampling period at a rate of 35 cm^3/min.] The trap (see Section 10.1.1.3) is cooled to -150°C by controlled release of a cryogen. VOCs and SVOCs are condensed on the trap surface while N_2, O_2, and other sample components are passed to the pump. After the organic compounds are concentrated, the valve is switched and the trap is heated. The revolatilized compounds are transported by helium carrier gas at a rate of 4 cm^3/min to the head of the Megabore® OV-1 capillary column (0.53 mm x 30 m). Since the column initial temperature is at -50°C, the VOCs and SVOCs are cryofocussed on the head of the column. Then, the oven temperature is programmed to increase and the VOCs/SVOC in the carrier gas are chromatographically separated. The carrier gas containing the separated VOCs/SVOC is then directed to two parallel detectors at a flow rate of 2 cm^3/min each. The detectors sense the presence of the speciated VOCs/SVOC, and the response is recorded by either a strip chart recorder or a data processing unit.

10.1.3.2 Typical chromatograms of VOCs determined by the GC-FID-ECD analytical system are illustrated in Figures 11 (c) and 11(d) respectively.

10.1.3.3 Helium is used as the carrier gas (4 cm^3/min) to purge residual air from the trap at the end of the sampling phase and to carry the revolatilized VOCs through the Megabore® GC column. Moisture and organic impurities are removed from the helium gas stream by a chemical purifier installed in the GC (see

Section 7.2.1.11). After exiting the OV-1 Megabore® column, the carrier gas stream is split to the two detectors at rates of 2 cm^3/min each.

10.1.3.4 Gas scrubbers containing Drierite® or silica gel and 5A molecular sieve are used to remove moisture and organic impurities from the zero air, hydrogen, and nitrogen gas streams. [Note: Purity of gas purifiers is checked prior to use by passing humid zero-air through the gas purifier and analyzing according to Section 12.2.2.]

10.1.3.5 All lines should be kept as short as practical. All tubing used for the system should be chromatographic grade stainless steel connected with stainless steel fittings. After assembly, the system should be checked for leaks according to manufacturer's specifications.

10.1.3.6 The FID burner air, hydrogen, nitrogen (make-up), and helium (carrier) flow rates should be set according to the manufacturer's instructions to obtain an optimal FID response while maintaining a stable flame throughout the analysis. Typical flow rates are: burner air, 450 cm^3/min; hydrogen, 30 cm^3/min; nitrogen, 30 cm^3/min; helium, 2 cm^3/min.

10.1.3.7 The ECD nitrogen make-up gas and helium carrier flow rates should be set according to manufacturer's instructions to obtain an optimal ECD response. Typical flow rates are: nitrogen, 76 cm^3/min and helium, 2 cm^3/min.

10.1.3.8 The GC-FID-ECD could be modified to include a PID (see Figure 6) for increased sensitivity (20). In the photoionization process, a molecule is ionized by ultraviolet light as follows: R + hv --> R + e-, where R+ is the ionized species and a photon is represented by hv, with energy less than or equal to the ionization potential c

the molecule. Generally all species with an ionization potential less than the ionization energy of the lamp are detected. Because the ionization potential of all major components of air (O_2, N_2, CO, CO_2, and H_2O) is greater than the ionization energy of lamps in general use, they are not detected. The sensor is comprised of an argon-filled, ultraviolet (UV) light source where a portion of the organic vapors are ionized in the gas stream. A pair of electrodes are contained in a chamber adjacent to the sensor. When a positive potential is applied to the electrodes, any ions formed by the absorption of UV light are driven by the created electronic field to the cathode, and the current (proportional to the organic vapor concentration) is measured. The PID is generally used for compounds having ionization potentials less than the ratings of the ultraviolet lamps. This detector is used for determination of most chlorinated and oxygenated hydrocarbons, aromatic compounds, and high molecular weight aliphatic compounds. Because the PID is insensitive to methane, ethane, carbon monoxide, carbon dioxide, and water vapor, it is an excellent detector. The electron volt rating is applied specifically to the wavelength of the most intense emission line of the lamp's output spectrum. Some compounds with ionization potentials above the lamp rating can still be detected due to the presence of small quantities of more intense light. A typical system configuration associated with the GC-FID-ECD-PID is illustrated in Figure 6. This system is currently being used in EPA's FY-88 Urban Air Toxics Monitoring Program.

10.2 GC-MS-SCAN-SIM System Performance Criteria

10.2.1 GC-MS System Operation

10.2.1.1 Prior to analysis, the GC-MS system is assembled and checked according to manufacturer's instructions.

10.2.1.2 Table 3.0 outlines general operating conditions for the GC-MS-SCAN-SIM system with optional FID.

10.2.1.3 The GC-MS system is first challenged with humid zero air (see Section 11.2.2).

10.2.1.4 The GC-MS and optional FID system is acceptable if it contains less than 0.2 ppbv of targeted VOCs.

10.2.2 Daily GC-MS Tuning (See Figure 13)

10.2.2.1 At the beginning of each day or prior to a calibration, the GC-MS system must be tuned to verify that acceptable performance criteria are achieved.

10.2.2.2 For tuning the GC-MS, a cylinder containing 4-bromofluorobenzene is introduced via a sample loop valve injection system. [Note: Some systems allow auto-tuning to facilitate this process.] The key ions and ion abundance criteria that must be met are illustrated in Table 4.0. Analysis should not begin until all those criteria are met.

10.2.2.3 The GC-MS tuning standard could also be used to assess GC column performance (chromatographic check) and as an internal standard. Obtain a background correction mass spectra of 4-bromofluorobenzene and check that all key ions criteria are met. If the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

10.2.2.4 The performance criteria must be achieved before any samples, blanks or standards are analyzed. If any key ion abundance observed for the daily 4-bromofluorobenzene mass tuning check differs by more than 10% absolute abundance from that observed during the previous daily tuning, the instrument must be retuned or the sample and/or calibration gases reanalyzed until the above condition is met.

10.2.3 GC-MS Calibration (see Figure 13)

[Note: Initial and routine calibration procedures are illustrated in Figure 13.]

10.2.3.1 Initial Calibration - Initially, a multipoint dynamic calibration (three levels plus humid zero air) is performed on the GC-MS system, before sample analysis, with the assistance of a calibration system (see Figure 8). The calibration system uses NBS traceable standards or NBS/EPA CRMs in pressurized cylinders [containing a mixture of the targeted VOCs at nominal concentrations of 10 ppmv in nitrogen (Section 8.2)] as working standards to be diluted with humid zero air. The contents of the working standard cylinder(s) are metered (2 cm^3/min) into the heated mixing chamber where they are mixed with a 2-L/min humidified zero air gas stream to achieve a nominal 10 ppbv per compound calibration mixture (see Figure 3). This nominal 10 ppbv standard mixture is allowed to flow and equilibrate for a minimum of 30 minutes. After the equilibration period, the gas standard mixture is sampled and analyzed by the real-time GC-MS system [see Figure 8(a) and Section 7.2.1]. The results of the analyses are averaged, flow audits are performed on the mass flow meters and the calculated concentration compared to generated values. After the GC-MS is calibrated at three concentration levels, a second humid zero air sample is passed through the system and analyzed. The second humid zero air test is used to verify that the GC-MS system is certified clean (less than 0.2 ppbv of target compounds).

10.2.3.2 As an alternative, a multipoint humid static calibration (three levels plus zero humid air) can be performed on the GC-MS system. During the humid static calibration analyses, three (3) SUMMA® passivated canisters are filled each at a different concentration between 1-20 ppbv from the calibration manifold using a pump and mass flow control arrangement [see Figure 8(c)]. The canisters are then delivered to the GC-MS to serve as calibration standards. The canisters are analyzed by the MS in the SIM mode, each analyzed twice. The expected retention time and ion abundance (see Table 2 and Table 5) are used to verify proper operation of the GC-MS system. A calibration response factor is determined for each analyte, as illustrated in Table 5, and the computer calibration table is updated with this information, as illustrated in Table 6.

10.2.3.3 Routine Calibration - The GC-MS system is calibrated daily (and before sample analysis) with a one-point calibration. The GC-MS system is calibrated either with the dynamic calibration procedure [see Figure 8(a)] or with a 6-L SUMMA® passivated canister filled with humid calibration standards from the calibration manifold (see Section 10.2.3.2). After the single point calibration the GC-MS analytical system is challenged with a humidified zero gas stream to insure the analytical system returns to specification (less than 0.2 ppbv of selective organics).

10.3 GC-FID-ECD System Performance Criteria (with optional PID system) (see Figure 14)

10.3.1 Humid Zero Air Certification

10.3.1.1 Before system calibration and sample analysis, the GC-FID-ECD analytical system is assembled and checked according to manufacturer's instructions.

10.3.1.2 The GC-FID-ECD system is first challenged with humid zero air (see Section 12.2.2) and monitored.

10.3.1.3 Analytical systems contaminated with less than 0.2 ppbv of targeted VOCs are acceptable.

10.3.2 GC Retention Time Windows Determination (see Table 7)

10.3.2.1 Before analysis can be performed, the retention time windows must be established for each analyte.

10.3.2.2 Make sure the GC system is within optimum operating conditions.

10.3.2.3 Make three injections of the standard containing all compounds for retention time window determination. [Note: The retention time window must be established for each analyte every 72 hours during continuous operation.]

10.3.2.4 Calculate the standard deviation of the three absolute retention times for each single component standard. The retention window is defined as the mean plus or minus three times the standard deviation of the individual retention times for each standard. In those cases where the standard deviation for a particular standard is zero, the laboratory must substitute the standard deviation of a closely - eluting, similar compound to develop a valid retention time window.

10.3.2.5 The laboratory must calculate retention time windows for each standard (see Table 7) on each GC column, whenever a new GC column is installed or when major components of the GC are changed. The data must be noted and retained in a notebook by the laboratory as part of the user SOP and as a quality assurance check of the analytical system.

10.3.3 GC Calibration

[Note: Initial and routine calibration procedures are illustrated in Figure 14.]

10.3.3.1 Initial Calibration - Initially, a multipoint dynamic calibration (three levels plus humid zero air) is performed on the GC-FID-ECD system, before sample analysis, with the assistance of a calibration system (see Figure 8). The calibration system uses NBS traceable standards or NBS/EPA CRMs in pressurized cylinders [containing a mixture of the targeted VOCs at nominal nominal concentrations of 10 ppmv in nitrogen (Section 8.2)] as working standards to be diluted with humid zero air. The contents of the working standard cylinders are metered (2 cm^3/min) into the heated mixing chamber where they are mixed with a 2-L/min humidified zero air stream to achieve a nominal 10 ppbv per compound calibration mixture (see Figure 8). This nominal 10 ppbv standard mixture is allowed to flow and equilibrate for a minimum of 30 minutes. After the equilibration period, the gas standard mixture is sampled and analyzed by the GC-MS system [see Figure 8(a)]. The results of the analyses are averaged, flow audits are performed on the mass flow controllers used to generate the standards and the appropriate response factors (concentration/area counts) are calculated for each compound, as illustrated in Table 5.0. [Note: GC-FIDs are linear in the 1-20 ppbv range and may not require repeated multipoint calibrations; whereas, the GC-ECD will require frequent linearity evaluation.] Table 5.0 outlines typical calibration response factors

and retention times for 40 VOCs. After the GC-FID-ECD is calibrated at the three concentration levels, a second humid zero air sample is passed through the system and analyzed. The second humid zero air test is used to verify that the GC-FID-ECD system is certified clean (less than 0.2 ppbv of target compounds).

10.3.3.2 Routine Calibration - A one-point calibration is performed daily on the analytical system to verify the initial multi-point calibration (see Section 10.3.3.1). The analyzers (GC-FID-ECD) are calibrated (before sample analysis) using the static calibration procedures (see Section 10.2.3.2) involving pressurized gas cylinders containing low concentrations of the targeted VOCs (10 ppbv) in nitrogen. After calibration, humid zero air is once again passed through the analytical system to verify residual VOCs are not present.

10.3.4 GC-FID-ECD-PID System Performance Criteria

10.3.4.1 As an option, the user may wish to include a photoionization detector (PID) to assist in peak identification and increase sensitivity.

10.3.4.2 This analytical system is presently being used in U.S. Environmental Protection Agency's Urban Toxic Air Pollutant Program (UTAP).

10.3.4.3 Preparation of the GC-FID-ECD-PID analytical system is identical to the GC-FID-ECD system (see Section 10.3).

10.3.4.4 Table 8.0 outlines typical retention time (minutes) for selected organics using the GC-FID-ECD-PID analytical system.

10.3.4.5 The analytical system used to generate Table 8.0 was a Varian® 3700 GC equipped with a J&W Megabore® DB 624 capillary column (30 m x 0.53 I.D. mm) using helium carrier gas.

10.4 Analytical Procedures

10.4.1 Canister Receipt

10.4.1.1 The overall condition of each sample canister is observed. Each canister should be received with an attached sample identification tag.

10.4.1.2 Each canister is recorded in the dedicated laboratory logbook. Also noted on the identification tag are date received and initials of recipient.

10.4.1.3 The pressure of the canister is checked by attaching a pressure gauge to the canister inlet. The canister valve is opened briefly and the pressure (kPa, psig) is recorded. [Note: If pressure is <83 kPa (<12 psig) the user may wish to pressurize the canisters, as an option, with zero grade humidified nitrogen up to 137 kPa (20 psig) to ensure that enough sample is available for analysis. However, pressurizing the canister can introduce additional error, increase the minimum detection limit (MDL), and is time consuming. The user should weigh these limitations as part of his program objectives before pressurizing.] Final cylinder pressure is recorded on canister sampling field data sheet (see Figure 10).

10.4.1.4 If the canister pressure is increased, a dilution factor (DF) is calculated and recorded on the sampling data sheet.

$$DF = \frac{Y_a}{X_a}$$

where:

X_a = canister pressure (kPa, psia) absolute before dilution.

Y_a = canister pressure (kPa, psia) absolute after dilution.

After sample analysis, detected VOC concentrations are multiplied by the dilution factor to determine concentration in the sampled air.

10.4.2 GC-MS-SCAN Analysis (With Optional FID System)

10.4.2.1 The analytical system should be properly assembled, humid zero air certified (see Section 12.3), operated (see Table 3), and calibrated for accurate VOC determination.

10.4.2.2 The mass flow controllers are checked and adjusted to provide correct flow rates for the system.

10.4.2.3 The sample canister is connected to the inlet of the GC-MS-SCAN (with optional FID) analytical system. A mass flow controller is placed on the canister and the canister valve is opened and the canister flow is vented past a tee inlet to the analytical system at a flow of 75 cm^3/min so that 40 cm^3/min is pulled through the Nafion® dryer to the six-port chromatographic valve. [Note: Flow rate is not as important as acquiring sample volume of 400 cm^3.]

10.4.2.4 The GC oven and cryogenic trap (inject position) are cooled to their set points of -50°C and -160°C, respectively.

10.4.2.5 As soon as the cryogenic trap reaches its lower set point of -160°C, the six-port chromatographic valve is turned to its fill position to initiate sample collection.

10.4.2.6 A ten minute collection period of canister sample is utilized [Note: 40 cm^3/min x 10 min = 400 cm^3 sampled canister contents.]

10.4.2.7 After the sample is preconcentrated in the cryogenic trap, the GC sampling valve is cycled to the inject position and the cryogenic trap is heated. The trapped analytes are thermally desorbed onto the head of the OV-1 Microbore® capillary column (0.31 mm I.D. x 50 m length). The GC oven is programmed to start at -50°C and after 2 min to heat to 150°C at a rate of 8°C per minute.

10.4.2.8 Upon sample injection onto the column, the MS is signaled by the computer to scan the eluting carrier gas from 18 to 250 amu, resulting in a 1.5 Hz repetition rate. This corresponds to about 6 scans per eluting chromatographic peak.

10.4.2.9 Primary identification is based upon retention time and relative abundance of eluting ions as compared to the spectral library stored on the hard disk of the GC-MS data computer.

10.4.2.10 The concentration (ppbv) is calculated using the previously established response factors (see Section 10.2.3.2), as illustrated in Table 5.0. [Note: If the canister is diluted before analysis, an appropriate multiplier is applied to correct for the volume dilution of the canister (Section 10.4.1.4).]

10.4.2.11 The optional FID trace allows the analyst to record the progress of the analysis.

10.4.3 GC-MS-SIM Analysis (With Optional FID system)

10.4.3.1 When the MS is placed in the SIM mode of operation, the MS monitors only preselected ions, rather than scanning all masses continuously between two mass limits.

10.4.3.2 As a result, increased sensitivity and improved quantitative analysis can be achieved.

10.4.3.3 Similar to the GC-MS-SCAN configuration, the GC-MS-SIM analysis is based on a combination of retention times and relative abundances of selected ions (see Table 2.0 and Table 5.0). These qualifiers are stored on the hard disk of the GC-MS computer and are applied for identification of each chromatographic peak. Once the GC-MS-SIM has identified the peak, a calibration response factor is used to determine the analyte's concentration.

10.4.3.4 The individual analyses are handled in three phases: data acquisition, data reduction, and data reporting. The data acquisition software is set in the SIM mode, where specific compound fragments are monitored by the MS at specific times in the analytical run. Data reduction is coordinated by the postprocessing macro program that is automatically accessed after data acquisition is completed at the end of the GC run. Resulting ion profiles are extracted, peaks are identified and integrated, and an internal integration report is generated by the program. A reconstructed ion chromatogram for hardcopy reference is prepared by the program and various parameters of interest such as time, date, and integration constants are printed. At the completion of the macro program, the data reporting software is accessed. The appropriate calibration table (see Table 9) is retrieved by the data reporting program from the computer's hard disk storage and the proper retention time and response factor parameters are applied to the macro program's integration file. With reference to certain pre-set acceptance criteria, peaks are automatically identified and quantified and a final summary report is prepared, as illustrated in Table 10.

10.4.4 GC-FID-ECD Analysis with Optional PID

10.4.4.1 The analytical system should be properly assembled, humid zero air certified (see Section 12.2) and calibrated through a dynamic standard calibration procedure (see Section 10.3.2). The FID detector is lit and allowed to stabilize.

10.4.4.2 Sixty-four minutes are required for each sample analysis - 15 min for system initialization, 14 min. for sample collection, 30 min for analysis, and 5 min. for post-time, during which a report is printed. [Note: This may vary depending upon system configuration and programming.]

10.4.4.3 The helium and sample mass flow controllers are checked and adjusted to provide correct flow rates for the system. Helium is used to purge residual air from the trap at the end of the sampling phase and to carry the revolatilized VOCs from the trap onto the GC column and into the FID-ECD. The hydrogen, burner air, and nitrogen flow rates should also be checked. The cryogenic trap is connected and verified to be operating properly while flowing cryogen through the system.

10.4.4.4 The sample canister is connected to the inlet of the GC-FID-ECD analytical system. The canister valve is opened and the canister flow is vented past a tee inlet to the analytical system at 75 cm^3/min using a 0-500 cm^3/min Tylan mass flow controller. During analysis, 40 cm^3/min of sample gas is pulled through the six-port chromatographic valve and routed through the trap at the appropriate time while the extra sample is vented. The volatile VOCs are condensed in the trap while the excess flow is exhausted through an exhaust vent, which assures that the sample air flowing through the trap is at atmospheric pressure.

10.4.4.5 The six-port valve is switched to the inject position and the canister valve is closed.

10.4.4.6 The electronic integrator is started.

10.4.4.7 After the sample is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -50°C, the VOCs are cryofocused on the column. Then, the oven temperature (programmed) increases and the VOCs elute from the column to the parallel FID-ECD assembly.

10.4.4.8 The peaks eluting from the detectors are identified by retention time (see Table 7.0 and Table 8.0), while peak areas are recorded in area

counts. Figures 15 and 16 illustrate typical response of the FID and ECD, respectively, for the forty (40) targeted VOCs. [Note: Refer to Table 7 for peak number and identification.]

10.4.4.9 The response factors (see Section 10.3.3.1) are multiplied by the area counts for each peak to calculate ppbv estimates for the unknown sample. If the canister is diluted before analysis, an appropriate dilution multiplier (DF) is applied to correct for the volume dilution of the canister (see Section 10.4.1.4).

10.4.4.10 Each canister is analyzed twice, and the final concentrations for each analyte are the averages of the two analyses.

10.4.4.11 However, if the GC-FID-ECD analytical system discovers unexpected peaks which need further identification and attention or overlapping peaks are discovered, eliminating possible quantitation, the sample should then be subjected to a GC-MS-SCAN for positive identification and quantitation.

11. Cleaning and Certification Program

11.1 Canister Cleaning and Certification

11.1.1 All canisters must be clean and free of any contaminants before sample collection.

11.1.2 All canisters are leak tested by pressurizing them to approximately 206 kPa (30 psig) with zero air. [Note: The canister cleaning system in Figure 7 can be used for this task.] The initial pressure is measured, the canister valve is closed, and the final pressure is checked after 24 hours. If leak tight, the pressure should not vary more than ± 13.8 kPa (± 2 psig) over the 24 hour period.

11.1.3 A canister cleaning system may be assembled as illustrated in Figure 7. Cryogen is added to both the vacuum pump and zero air supply traps. The canister(s)

are connected to the manifold. The vent shut-off valve and the canister valve(s) are opened to release any remaining pressure in the canister(s). The vacuum pump is started and the vent shut-off valve is then closed and the vacuum shut-off valve is opened. The canister(s) are evacuated to < 0.05 mm Hg (for at least one hour). [Note: On a daily basis or more often if necessary, the cryogenic traps should be purged with zero air to remove any trapped water from previous canister cleaning cycles.]

11.1.4 The vacuum and vacuum/pressure gauge shut-off valves are closed and the zero air shut-off valve is opened to pressurize the canister(s) with humid zero air to approximately 206 kPa (30 psig). If a zero gas generator system is used, the flow rate may need to be limited to maintain the zero air quality.

11.1.5 The zero shut-off valve is closed and the canister(s) is allowed to vent down to atmospheric pressure through the vent shut-off valve. The vent shut-off valve is closed. Steps 11.1.3 through 11.1.5 are repeated two additional times for a total of three (3) evacuation/pressurization cycles for each set of canisters.

11.1.6 At the end of the evacuation/pressurization cycle, the canister is pressurized to 206.7 kPa (30 psig) with humid zero air. The canister is then analyzed by a GC-MS or GC-FID-ECD analytical system. Any canister that has not tested clean (compared to direct analysis of humidified zero air of less than 0.2 ppbv of targeted VOCs) should not be used. As a "blank" check of the canister(s) and cleanup procedure, the final humid zero air fill of 100% of the canisters is analyzed until the cleanup system and canisters are proven reliable (less than 0.2 ppbv of targets VOCs). The check can then be reduced to a lower percentage of canisters.

11.1.7 The canister is reattached to the cleaning manifold and is then reevacuated to <0.05 mm Hg and remains in this condition until used. The canister valve is closed. The canister is removed from the cleaning system and the canister connection is capped with a stainless steel fitting.

The canister is now ready for collection of an air sample. An identification tag is attached to the neck of each canister for field notes and chain-of-custody purposes.

11.1.8 As an option to the humid zero air cleaning procedures, the canisters could be heated in an isothermal oven to 100°C during Section 11.1.3 to ensure that lower molecular weight compounds (C_2-C_8) are not retained on the walls of the canister. [Note: For sampling heavier, more complex VOC mixtures, the canisters should be heated to 250°C during Section 11.1.3.7.] Once heated, the canisters are evacuated to 0.05 mm Hg. At the end of the heated/evacuated cycle, the canisters are pressurized with humid zero air and analyzed by the GC-FID-ECD system. Any canister that has not tested clean (less than 0.2 ppbv of targeted compounds) should not be used. Once tested clean, the canisters are reevacuated to 0.05 mm Hg and remain in the evacuated state until used.

11.2 Sampling System Cleaning and Certification

11.2.1 Cleaning Sampling System Components

11.2.1.1 Sample components are disassembled and cleaned before the sampler is assembled. Nonmetallic parts are rinsed with HPLC grade deionized water and dried in a vacuum oven at 50°C. Typically, stainless steel parts and fittings are cleaned by placing them in a beaker of methanol in an ultrasonic bath for 15 minutes. This procedure is repeated with hexane as the solvent.

11.2.1.2 The parts are then rinsed with HPLC grade deionized water and dried in a vacuum oven at 100°C for 12 to 24 hours.

11.2.1.3 Once the sampler is assembled, the entire system is purged with humid zero air for 24 hours.

11.2.2 Humid Zero Air Certification

[Note: In the following sections, "certification" is defined as evaluating the sampling system with humid

zero air and humid calibration gases that pass through all active components of the sampling system. The system is "certified" if no significant additions or deletions (less than 0.2 ppbv of targeted compounds) have occurred when challenged with the test gas stream.]

11.2.2.1 The cleanliness of the sampling system is tested by testing the sampler with humid zero air without an evacuated gas cylinder, as follows.

11.2.2.2 The calibration system and manifold is assembled, as illustrated in Figure 8. The sampler (without an evacuated gas cylinder) is connected to the manifold and the zero air cylinder activated to generate a humid gas stream (2 L/min) to the calibration manifold [see Figure 8(b)].

11.2.2.3 The humid zero gas stream passes through the calibration manifold, through the sampling system (without an evacuated canister) to a GC-FID-ECD analytical system at 75 cm^3/min so that 35 cm^3/min is pulled through the six-port valve and routed through the cryogenic trap (see Section 10.2.2.1) at the appropriate time while the extra sample is vented. [Note: The exit of the sampling system (without the canister) replaces the canister in Figure 4.] After the sample (400 mL) is preconcentrated on the trap, the trap is heated and the VOCs are thermally desorbed onto the head of the capillary column. Since the column is at -50°C, the VOCs are cryofocused on the column. Then, the oven temperature (programmed) increases and the VOCs begin to elute and are detected by a GC-MS (see Section 10.2) or the GC-FID-ECD (see Section 10.3) detector. The analytical system should not detect greater than 0.2 ppbv of targeted VOCs in order for the sampling system to pass the humid zero air certification

test. Chromatograms of a certified sampler and contaminated sampler are illustrated in Figures 17(a) and (b), respectively. If the sampler passes the humid zero air test, it is then tested with humid calibration gas standards containing selected VOCs at concentration levels expected in field sampling (e.g., 0.5 to 2 ppbv) as outlined in Section 11.2.3.

11.2.3 Sampler System Certification with Humid Calibration Gas Standards

11.2.3.1 Assemble the dynamic calibration system and manifold as illustrated in Figure 8.

11.2.3.2 Verify that the calibration system is clean (less than 0.2 ppbv of targeted compounds) by sampling a humidified gas stream, <u>without</u> gas calibration standards, with a previously certified clean canister (see Section 12.1).

11.2.3.3 The assembled dynamic calibration system is certified clean if less than 0.2 ppbv of targeted compounds are found.

11.2.3.4 For generating the humidified calibration standards, the calibration gas cylinder(s) (see Section 8.2) containing nominal concentrations of 10 ppmv in nitrogen, of selected VOCs, are attached to the calibration system, as outlined in Section 10.2.3.1. The gas cylinders are opened and the gas mixtures are passed through 0 to 10 cm^3/min certified mass flow controllers to generate ppb levels of calibration standards.

11.2.3.5 After the 30-minute equilibrium period, attach the sampling system (containing a certified evacuated canister) to the manifold, as illustrated in Figure 8(a).

11.2.3.6 Sample the dynamic calibration gas stream with the sampling system according to Section 9.2.1. [Note: To conserve generated calibration gas, bypass the canister sampling system manifold and attach the sampling system to the calibration gas stream at the inlet of the in-line filter of the sampling system so the flow will be less than 500 cm^3/ min.]

11.2.3.7 Concurrent with the sampling system operation, realtime monitoring of the calibration gas stream is accomplished by the on-line GC-MS or GC-multi-detector analytical system [Figure 8(b)] to provide reference concentrations of generated VOCs.

11.2.3.8 At the end of the sampling period (normally same time period used for anticipated sampling) the sampling system canister is analyzed and compared to the reference GC-MS or GC-multi-detector analytical system to determine if the concentration of the targeted VOCs were increased or decreased by the sampling system.

11.2.3.9 A recovery of between 90% and 110% is expected for all targeted VOCs.

12. Performance Criteria and Quality Assurance

12.1 Standard Operating Procedures (SOPs)

12.1.1 SOPs should be generated in each laboratory describing and documenting the following activities: (1) assembly, calibration, leak check, and operation of specific sampling systems and equipment used; (2) preparation, storage, shipment, and handling of samples; (3) assembly, leak-check, calibration, and operation of the analytical system, addressing the specific equipment used; (4) canister storage and cleaning; and (5) all aspects of data recording and processing, including lists of computer hardware and software used.

12.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the laboratory personnel conducting the work.

12.2 Method Relative Accuracy and Linearity

12.2.1 Accuracy can be determined by injecting VOC standards (see Section 8.2) from an audit cylinder into a sampler. The contents are then analyzed for the components contained in the audit canister. Percent relative accuracy is calculated:

$$\% \text{ Relative Accuracy} = \frac{X - Y}{X} \times 100$$

Where: Y = Concentration of the targeted compound recovered from sampler.

X = Concentration of VOC targeted compound in the NBS-SRM or EPA-CRM audit cylinders.

12.2.2 If the relative accuracy does not fall between 90 and and 110 percent, the field sampler should be removed from use, cleaned, and recertified according to initial certification procedures outlined in Section 11.2.2 and Section 11.2.2. Historically, concentrations of carbon tetrachloride, tetrachloroethylene, and hexachlorobutadiene have sometimes been detected at lower concentrations when using parallel ECD and FID detectors. When these three compounds are present at concentrations close to calibration levels, both detectors usually agree on the reported concentrations. At concentrations below 4 ppbv, there is a problem with nonlinearity of the ECD. Plots of concentration versus peak area for calibration compounds detected by the ECD have shown that the curves are nonlinear for carbon tetrachloride, tetrachloroethylene, and hexachlorobutadiene, as illustrated in Figures 18(a) through 18(c). Other targeted ECD and FID compounds scaled linearly for the range 0 to 8 ppbv, as shown for chloroform in Figure 18(d). For compounds that are not linear over the calibration

range, area counts generally roll off between 3 and 4 ppbv. To correct for the nonlinearity of these compounds, an additional calibration step is performed. An evacuated stainless steel canister is pressurized with calibration gas at a nominal concentration of 8 ppbv. The sample is then diluted to approximately 3.5 ppbv with zero air and analyzed. The instrument response factor (ppbv/area) of the ECD for each of the three compounds is calculated for the 3.5 ppbv sample. Then, both the 3.5 ppbv and the 8 ppbv response factors are entered into the ECD calibration table. The software for the Hewlett-Packard 5880 level 4 GC is designed to accommodate multilevel calibration entries, so the correct response factors are automatically calculated for concentrations in this range.

12.3 Method Modification

12.3.1 Sampling

12.3.1.1 The sampling system for pressurized canister sampling could be modified to use a lighter, more compact pump. The pump currently being used weighs about 16 kilograms (35 lbs). Commercially available pumps that could be used as alternatives to the prescribed sampler pump are described below. Metal Bellows MB-41 pump: These pumps are cleaned at the factory; however, some precaution should be taken with the circular (4.8 cm diameter) Teflon and stainless steel part directly under the flange. It is often dirty when received and should be cleaned before use. This part is cleaned by removing it from the pump, manually cleaning with deionized water, and placing in a vacuum oven at 100°C for at least 12 hours. Exposed parts of the pump head are also cleaned with swabs and allowed to air dry. These pumps have

proven to be very reliable; however, they are only useful up to an outlet pressure of about 137 kPa (20 psig). Neuberger Pump: Viton gaskets or seals must be specified with this pump. The "factory direct" pump is received contaminated and leaky. The pump is cleaned by disassembling the pump head (which consists of three stainless steel parts and two gaskets), cleaning the gaskets with deionized water and drying in a vacuum oven, and remachining (or manually lapping) the sealing surfaces of the stainless steel parts. The stainless steel parts are then cleaned with methanol, hexane, deionized water and heated in a vacuum oven. The cause for most of the problems with this pump has been scratches on the metal parts of the pump head. Once this rework procedure is performed, the pump is considered clean and can be used up to about 240 kPa (35 psig) output pressure. This pump is utilized in the sampling system illustrated in Figure 3.

12.3.1.2 The subatmospheric sampling system described in this method has been modified and designed specifically for use in USEPA's Urban Toxic Air Pollutant Program (UTAP), a joint project of USEPA's Office of Air Quality Planning and Standards, the Environmental Monitoring Systems Laboratory, and the participating state air pollution control agencies. The purpose of UTAP is to provide analytical support to the states in their assessment of potential health risks from certain toxic organic compounds that may be present in urban atmospheres.

The sampler is based on the collection of whole air samples in a 6-liter, SUMMA® passivated stainless steel canisters. The sampler features electronic timer for ease, accuracy and flexibility of sample period programming, an independently setable pre-sample warm-up and ambient air purge period, protection from loss of sample due to power interruptions, and a self-contained configuration housed in an all-metal portable case, as illustrated in Figure 19. The design of the sampler is pumpless, using an evacuated canister to draw the ambient sample air into itself at a fixed flow rate (3-5 cm^3/min) controlled by an electronic mass flow controller. Because of the relatively low sample flow rates necessary for the integration periods, auxiliary flushing of the sample inlet line is provided by a small, general-purpose vacuum pump (not in contact with the sample air stream). Further, experience has shown that inlet lines and surfaces sometimes build up or accumulate substantial concentrations of organic materials under stagnant (zero flow rate) conditions. Therefore such lines and surfaces need to be purged and equilibrated to the sample air for some time prior to the beginning of the actual sample collection period. For this reason, the sampler includes dual timers, one of which is set to start the pump several hours prior to the specified start of the sample period to purge the inlet lines and surfaces. As illustrated in Figure 20, sample air drawn into the canister passes through only four components: the heated inlet line, a 2-micron particulate filter, the electron flow controller, and the latching solenoid valve.

In operation, timer 1 is set to start the pump about 6 hours before the scheduled sample period. The pump draws sample air in through the sample inlet and particulate filter to purge and equilibrate these components, at a flow rate limited by the capillary to approximately 100 cm^3/min. Timer 1 also energizes the heated inlet line to allow it to come up to its controlled temperature of 65 to 70 degrees C, and turns on the flow controller to allow it to stabilize. The pump draws additional sample air through the flow controller by way of the normally open port of the 3-way solenoid valve. This flow purges the flow controller and allows it to achieve a stable controlled flow at the specified sample flow rate prior to the sample period. At the scheduled start of the sample period, timer 2 is set to activate both solenoid valves. When activated, the 3-way solenoid valve closes its normally open port to stop the flow controller purge flow and opens its normally closed port to start flow through the aldehyde sample cartridges. Simultaneously, the latching solenoid valve opens to start sample flow into the canister. At the end of the sample period, timer 2 closes the latching solenoid valve to stop the sample flow and seal the sample in the canister and also de-energizes the pump, flow controller, 3-way solenoid, and heated inlet line. During operation, the pump and sampler are located external to the sampler. The 2.4 meter (8 foot) heated inlet line is installed through the outside wall, with most of its length outside and terminated externally with an inverted glass funnel to exclude

precipitation. The indoor end is terminated in a stainless steel cross fitting to provide connections for the canister sample and the two optional formaldehyde cartridge samples (see Figure 19). A detailed description of installation and operation procedures for the UTAP sampler is provided in Appendix C.

12.3.2 Analysis

12.3.2.1 Inlet tubing from the calibration manifold could be heated to 50°C (same temperature as the calibration manifold) to prevent condensation on the internal walls of the system.

12.3.2.2 The analytical strategy for Method TO14 involves positive identification and quantitation by GC-MS-SCAN-SIM mode of operation with optional FID. This is a highly specific and sensitive detection technique. Because a specific detector system (GC-MS-SCAN-SIM) is more complicated and expensive than the use of nonspecific detectors (GC-FID-ECD-PID), the analyst may want to perform a screening analysis and preliminary quantitation of VOC species in the sample, including any polar compounds, by utilizing the GC-multidetector (GC-FID-ECD-PID) analytical system prior to GC-MS analysis. This system can be used for approximate quantitation. The GC-FID-ECD-PID provides a "snapshot" of the constituents in the sample, allowing the analyst to determine:

- Extent of misidentification due to overlapping peaks,

- Whether the constituents are within the calibration range of the anticipated GC-MS-SCAN-SIM analysis or does the sample require further dilution, and

- Are there unexpected peaks which need further identification through GC-MS-SCAN or are there peaks of interest needing attention?

If unusual peaks are observed from the GC-FID-ECD-PID system, the analyst then performs a GC-MS-SCAN analysis. The GC-MS-SCAN will provide positive identification of suspect peaks from the GC-FID-ECD-PID system. If no unusual peaks are identified and only a select number of VOCs are of concern, the analyst can then proceed to GC-MS-SIM. The GC-MS-SIM is used for final quantitation of selected VOCs. Polar compounds, however, cannot be identified by the GC-MS-SIM due to the use of a Nafion® dryer to remove water from the sample prior to analysis. The dryer removes polar compounds along with the water.

The analyst often has to make this decision incorporating project objectives, detection limits, equipment availability, cost and personnel capability in developing an analytical strategy. Figure 21 outlines the use of the GC-FID-ECD-PID as a "screening" approach, with the GC-MS-SCAN-SIM for final identification and quantitation.

12.4 Method Safety

This procedure may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the user's responsibility to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to the implementation of this procedure. This should be part of the user's SOP manual.

12.5 Quality Assurance [see Figure 22]

12.5.1 Sampling System

12.5.1.1 Section 9.2 suggests that a portable GC system be used as a "screening analysis" prior to locating fixed-site samplers (pressurized or subatmospheric

12.5.1.2 Section 9.2 requires pre-and post-sampling measurements with a certified mass flow controller for flow verification of sampling system.

12.5.1.3 Section 11.1 requires all canisters to be pressure tested to 207 kPa ± 14 kPa (30 psig ± 2psig) over a period of 24 hours.

12.5.1.4 Section 11.1 requires that all canisters be certified clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.

12.5.1.5 Section 11.2.2 requires all field sampling systems to be certified initially clean (containing less than 0.2 ppbv of targeted VOCs) through a humid zero air certification program.

12.5.1.6 Section 11.2.3 requires all field sampling systems to pass an initial humidified calibration gas certification [at VOC concentration levels expected in the field (e.g., 0.5 to 2 ppbv)] with a percent recovery of greater than 90.

12.5.2 GC-MS-SCAN-SIM System Performance Criteria

12.5.2.1 Section 10.2.1 requires the GC-MS analytical system to be certified clean (less than 0.2 ppbv of targeted VOCs) prior to sample analysis, through a humid zero air certification.

12.5.2.2 Section 10.2.2 requires the daily tuning of the GC-MS with 4-bromofluorobenzene (4-BFB) and that it meet the key ions and ion abundance critera (10%) outlined in Table 5.0.

12.5.2.3 Section 10.2.3 requires both an initial multipoint humid static calibration (three levels plus humid zero air) and a daily calibration (one point) of the GC-MS analytical system.

12.5.3 GC-Multidetector System Performance Criteria

12.5.3.1 Section 10.3.1 requires the GC-FID-ECD analytical system, prior to analysis, to be certified clean (less than 0.2 ppbv of targeted VOCs) through a humid zero air certification.

12.5.3.2 Section 10.3.2 requires that the GC-FID-ECD analytical system establish retention time windows for each analyte prior to sample analysis, when a new GC column is installed, or major components of the GC system altered since the previous determination.

12.5.3.3 Section 8.2 requires that all calibration gases be traceable to a National Bureau of Standards (NBS) Standard Reference Material (SRM) or to a NBS/EPA approved Certified Reference Material (CRM).

12.5.3.4 Section 10.3.2 requires that the retention time window be established throughout the course of a 72-hr analytical period.

13.5.3.5 Section 10.3.3 requires both an initial multipoint calibration (three levels plus humid zero air) and a daily calibration (one point) of the GC-FID-ECD analytical system with zero gas dilution of NBS traceable or NBS/EPA CRMs gases. [Note: Gas cylinders of VOCs at the ppm and ppb level are available for audits from the USEPA, Environmental Monitoring Systems Laboratory, Quality Assurance Division, MD-77B, Research Triangle Park, NC 27711, (919)541-4531. Appendix A outlines five groups of audit gas cylinders available from USEPA.]

13. Acknowledgements

The determination of volatile and some semi-volatile organic compounds in ambient air is a complex task, primarily because of the wide variety of compounds of interest and the lack of standardized sampling and analytical procedures. While there are numerous procedures for sampling and analyzing VOCs/SVOCs in ambient air, this method draws upon the best aspects of each one and combines them into a standardized methodology. To that end, the following individuals contributed to the research, documentation and peer review of this manuscript.

Topic	Contact	Address	Telephone No.
Sampling System	Mr. Frank McElroy Mr. Vince Thompson	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77 Research Triangle Park, N.C. 27711	919-541-2622 919-541-3791
	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Mr. Tom Merrifield	Anderson Samplers, Inc. 4215-C Wendell Drive Atlanta, GA 30336	1-800-241-6898
	Mr. Joseph P. Krasnec	Scientific Instrumentation Specialists, Inc. P.O. Box 8941 Moscow, Idaho, 83843	208-882-3860
Analytical System			
GC-FID	Mr. Vince Thompson	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77 Research Triangle Park, N.C. 27711	919-541-3791
GC-FID-ECD	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Ms. Karen D. Oliver	Northrop Services, Inc. Environmental Sciences P.O. Box 12313 Research Triangle Park, N.C. 27709	919-549-0611
GC-FID-ECD-PID	Dave-Paul Dayton JoAnn Rice	Radian Corporation P.O. Box 13000 Progress Center Research Triangle Park, N.C. 27709	919-481-0212

Topic	Contact	Address	Telephone No.
GC-MS-SCAN-SIM	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Mr. John V. Hawkins	Research Triangle Laboratories, Inc. P.O. Box 12507 Research Triangle Park, N.C. 27709	919-544-5775
Canister Cleaning Certification and VOC Canister Storage Stability	Mr. Vince Thompson	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77 Research Triangle Park, N.C. 27711	919-541-3791
	Dr. Bill McClenny Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-3158 919-541-4680
	Dave-Paul Dayton JoAnn Rice	Radian Corporation P.O. Box 13000 Progress Center Research Triangle Park, N.C. 27709	919-481-0212
	Dr. R.K.M. Jayanty	Research Triangle Institute P.O. Box 12194 Research Triangle Park, N.C. 27709	919-541-6000
Cryogenic Sampling Unit	Mr. Lou Ballard Mr. Pete Watson	NuTech Corporation 2806 Cheek Road Durham, N.C., 27704	919-682-0402
	Mr. Joachim Pleil	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-44 Research Triangle Park, N.C. 27711	919-541-4680
U.S. EPA Audit Gas Standards	Mr. Bob Lampe	U.S. Environmental Protection Agency Environmental Monitoring Systems Laboratory MD-77B Research Triangle Park, N.C.27711	919-541-4531

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TABLE 1 VOLATILE ORGANIC COMPOUND DATA SHEET

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT (°C)	MELTING POINT (°C)	CAS NUMBER
Freon 12 (Dichlorodifluoromethane)	Cl_2CF_2	120.91	-29.8	-158.0	
Methyl chloride (Chloromethane)	CH_3Cl	50.49	-24.2	-97.1	74-87-3
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	$ClCF_2CClF_2$	170.93	4.1	-94.0	
Vinyl chloride (Chloroethylene)	$CH_2{=}CHCl$	62.50	-13.4	-1538.0	75-01-4
Methyl bromide (Bromomethane)	CH_3Br	94.94	3.6	-93.6	74-83-9
Ethyl chloride (Chloroethane)	CH_3CH_2Cl	64.52	12.3	-136.4	75-00-3
Freon 11 (Trichlorofluoromethane)	CCl_3F	137.38	23.7	-111.0	
Vinylidene chloride (1,1-Dichloroethene)	$C_2H_2Cl_2$	96.95	31.7	-122.5	75-35-4
Dichloromethane (Methylene chloride)	CH_2Cl_2	84.94	39.8	-95.1	75-09-2
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	CF_2ClCCl_2F	187.38	47.7	-36.4	
1,1-Dichloroethane (Ethylidene chloride)	CH_3CHCl_2	98.96	57.3	-97.0	74-34-3
cis-1,2-Dichloroethylene	$CHCl{=}CHCl$	96.94	60.3	-80.5	
Chloroform (Trichloromethane)	$CHCl_3$	119.38	61.7	-63.5	67-66-3
1,2-Dichloroethane (Ethylene dichloride)	$ClCH_2CH_2Cl$	98.96	83.5	-35.3	107-06-2
Methyl chloroform (1,1,1-Trichloroethane)	CH_3CCl_3	133.41	74.1	-30.4	71-55-6
Benzene (Cyclohexatriene)	C_6H_6	78.12	80.1	5.5	71-43-2
Carbon tetrachloride (Tetrachloromethane)	CCl_4	153.82	76.5	-23.0	56-23-5
1,2-Dichloropropane (Propylene dichloride)	$CH_3CHClCH_2Cl$	112.99	96.4	-100.4	78-87-5
Trichloroethylene (Trichloroethene)	$ClCH{=}CCl_2$	131.29	87	-73.0	79-01-6
cis-1,3-Dichloropropene (cis-1,3-dichloropropylene)	$CH_3CCl{=}CHCl$	110.97	76		

TABLE 1 VOLATILE ORGANIC COMPOUND DATA SHEET (cont'd)

COMPOUND (SYNONYM)	FORMULA	MOLECULAR WEIGHT	BOILING POINT(°C)	MELTING POINT (°C)	CAS NUMBER
trans-1,3-Dichloropropene (cis-1,3-Dichloropropylene)	$ClCH_2CH{=}CHCl$	110.97	112.0		
1,1,2-Trichloroethane (Vinyl trichloride)	$CH_2ClCHCl_2$	133.41	113.8	-36.5	79-00-5
Toluene (Methyl benzene)	$C_6H_5CH_3$	92.15	110.6	-95.0	108-88-3
1,2-Dibromoethane (Ethylene dibromide)	$BrCH_2CH_2Br$	187.88	131.3	9.8	106-93-4
Tetrachloroethylene (Perchloroethylene)	$Cl_2C{=}CCl_2$	165.83	121.1	-19.0	127-18-4
Chlorobenzene (Phenyl chloride)	C_6H_5Cl	112.56	132.0	-45.6	108-90-7
Ethylbenzene	$C_6H_5C_2H_5$	106.17	136.2	-95.0	100-41-4
m-Xylene (1,3-Dimethylbenzene)	$1,3\text{-}(CH_3)_2C_6H_4$	106.17	139.1	-47.9	
p-Xylene (1,4-Dimethylxylene)	$1,4\text{-}(CH_3)_2C_6H_4$	106.17	138.3	13.3	
Styrene (Vinyl benzene)	$C_6H_5CH{=}CH_2$	104.16	145.2	-30.6	100-42-5
1,1,2,2-Tetrachloroethane	$CHCl_2CHCl_2$	167.85	146.2	-36.0	79-34-5
o-Xylene (1,2-Dimethylbenzene)	$1,2\text{-}(CH_3)_2C_6H_4$	106.17	144.4	-25.2	
1,3,5-Trimethylbenzene (Mesitylene)	$1,3,5\text{-}(CH_3)_3C_6H_6$	120.20	164.7	-44.7	108-67-8
1,2,4-Trimethylbenzene (Pseudocumene)	$1,2,4\text{-}(CH_3)_3C_6H_6$	120.20	169.3	-43.8	95-63-6
m-Dichlorobenzene (1,3-Dichlorobenzene)	$1,3\text{-}Cl_2C_6H_4$	147.01	173.0	-24.7	541-73-1
Benzyl chloride (α-Chlorotoluene)	$C_6H_5CH_2Cl$	126.59	179.3	-39.0	100-44-7
o-Dichlorobenzene (1,2-Dichlorobenzene)	$1,2\text{-}Cl_2C_6H_4$	147.01	180.5	-17.0	95-50-1
p-Dichlorobenzene (1,4-Dichlorobenzene)	$1,4\text{-}Cl_2C_6H_4$	147.01	174.0	53.1	106-46-7
1,2,4-Trichlorobenzene	$1,2,4\text{-}Cl_3C_6H_3$	181.45	213.5	17.0	120-82-1
Hexachlorobutadiene (1,1,2,3,4,4-Hexachloro-1,3-butadiene)					

TABLE 2 ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM

Compound	Ion/Abundance (amu/% base peak)	Expected Retent Time (min)
Freon 12 (Dichlorodifluoromethane)	85/100 87/ 31	5.01
Methyl chloride (Chloromethane)	50/100 52/ 34	5.69
Freon 114 (1,2-Dichloro-1,1,2,2-tetrafluoroethane)	85/100 135/ 56 87/ 33	6.55
Vinyl chloride (Chloroethene)	62/100 27/125 64/ 32	6.71
Methyl bromide (Bromomethane)	94/100 96/ 85	7.83
Ethyl chloride (Chloroethane)	64/100 29/140 27/140	8.43
Freon 11 (Trichlorofluoromethane)	101/100 103/ 67	9.97
Vinylidene chloride (1,1-Dichloroethylene)	61/100 96/ 55 63/ 31	10.93
Dichloromethane (Methylene chloride)	49/100 84/ 65 86/ 45	11.21
Freon 113 (1,1,2-Trichloro-1,2,2-trifluoroethane)	151/100 101/140 103/ 90	11.60
1,1-Dichloroethane (Ethylidene dichloride)	63/100 27/ 64 65/ 33	12.50
cis-1,2-Dichloroethylene	61/100 96/ 60 98/ 44	13.40
Chloroform (Trichloromethane)	83/100 85/ 65 47/ 35	13.75
1,2-Dichloroethane (Ethylene dichloride)	62/100 27/ 70 64/ 31	14.39
Methyl chloroform (1,1,1-Trichloroethane)	97/100 99/ 64 61/ 61	14.62
Benzene (Cyclohexatriene)	78/100 77/ 25 50/ 35	15.04
Carbon tetrachloride (Tetrachloromethane)	117/100 119/ 97	15.13

(continued)

TABLE 2 ION/ABUNDANCE AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM (cont'd)

Compound	Ion/Abundance (amu/% base peak)	Estimated Retention Time (min)
1,2-Dichloropropane (Propylene dichloride)	63/100 41/ 90 62/ 70	15.83
Trichloroethylene (Trichloroethene)	130/100 132/ 92 95/ 87	16.10
cis-1,3-Dichloropropene	75/100 39/ 70 77/ 30	16.96
trans-1,3-Dichloropropene (1,3 dichloro-1-propene)	75/100 39/ 70 77/ 30	17.49
1,1,2-Trichloroethane (Vinyl trichloride)	97/100 83/ 90 61/ 82	17.61
Toluene (Methyl benzene)	91/100 92/ 57	17.86
1,2-Dibromoethane (Ethylene dibromide)	107/100 109/ 96 27/115	18.48
Tetrachloroethylene (Perchloroethylene)	166/100 164/ 74 131/ 60	19.01
Chlorobenzene (Benzene chloride)	112/100 77/ 62 114/ 32	19.73
Ethylbenzene	91/100 106/ 28	20.20
m,p-Xylene(1,3/1,4-dimethylbenzene)	91/100 106/ 40	20.41
Styrene (Vinyl benzene)	104/100 78/ 60 103/ 49	20.81
1,1,2,2-Tetrachloroethane (Tetrachloroethane)	83/100 85/ 64	20.92
o-Xylene (1,2-Dimethylbenzene)	91/100 106/ 40	20.92
4-Ethyltoluene	105/100 120/ 29	22.53
1,3,5-Trimethylbenzene (Mesitylene)	105/100 120/ 42	22.65
1,2,4-Trimethylbenzene (Pseudocumene)	105/100 120/ 42	23.13
m-Dichlorobenzene (1,3-Dichlorobenzene)	146/100 148/ 65 111/ 40	23.31

(continued)

TABLE 2 ION/ABUNDANCES AND EXPECTED RETENTION TIME FOR SELECTED VOCs ANALYZED BY GC-MS-SIM (cont'd)

Compound	Ion/Abundance (amu/% base peak)	Expected Retention Time (min)
Benzyl chloride (α-Chlorotoluene)	91/100 126/ 26	23.32
p-Dichlorobenzene (1,4-Dichlorobenzene)	146/100 148/ 65 111/ 40	23.41
o-Dichlorobenzene (1,2-Dichlorobenzene)	146/100 148/ 65 111/ 40	23.88
1,2,4-Trichlorobenzene	180/100 182/ 98 184/ 30	26.71
Hexachlorobutadiene (1,1,2,3,4,4 Hexachloro-1,3-butadiene)	225/100 227/ 66 223/ 60	27.68

TABLE 3 GENERAL GC AND MS OPERATING CONDITIONS

Chromatography	
Column	Hewlett-Packard OV-1 crosslinked methyl silicone (50 m x 0.31-mm I.D., 17 um film thickness), or equivalent
Carrier Gas	Helium (20 cm^3/sec at 250°C)
Injection Volume	Constant (1-3 uL)
Injection Mode	Splitless
Temperature Program	
Initial Column Temperature	-50°C
Initial Hold Time	2 min
Program	8°C/min to 150°C
Final Hold Time	15 min
Mass Spectrometer	
Mass Range	18 to 250 amu
Scan Time	1 sec/scan
EI Condition	70 eV
Mass Scan	Follow manufacturer's instruction for selecting mass selective detector (MS) and selected ion monitoring (SIM) mode
Detector Mode	Multiple ion detection
FID System (Optional)	
Hydrogen Flow	30 cm^3/minute
Carrier Flow	30 cm^3/minute
Burner Air	400 cm^3/minute

TABLE 4 4-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base Peak, 100% Relative Abundance
95	5 to 9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but< 101% of mass 174
177	5 to 9% of mass 176

TABLE 5 RESPONSE FACTORS (ppbv/area count) AND EXPECTED RETENTION TIME FOR GC-MS-SIM ANALYTICAL CONFIGURATION

Compounds	Response Factor (ppbv/area count)	Expected Retention Time (minutes)
Freon 12	0.6705	5.01
Methyl chloride	4.093	5.64
Freon 114	0.4928	6.55
Vinyl chloride	2.343	6.71
Methyl bromide	2.647	7.83
Ethyl chloride	2.954	8.43
Freon 11	0.5145	9.87
Vinylidene chloride	1.037	10.93
Dichloromethane	2.255	11.21
Trichlorotrifluoroethane	0.9031	11.60
1,1-Dichloroethane	1.273	12.50
cis-1,2-Dichloroethylene	1.363	13.40
Chloroform	0.7911	13.75
1,2-Dichloroethane	1.017	14.39
Methyl chloroform	0.7078	14.62
Benzene	1.236	15.04
Carbon tetrachloride	0.5880	15.18
1,2-Dichloropropane	2.400	15.83
Trichloroethylene	1.383	16.10
cis-1,3-Dichloropropene	1.877	16.96
trans-1,3-Dichloropropene	1.338	17.49
1,1,2-Trichloroethane	1.891	17.61
Toluene	0.9406	17.86
1,2-Dibromoethane (EDB)	0.8662	18.48
Tetrachloroethylene	0.7357	19.01
Chlorobenzene	0.8558	19.73
Ethylbenzene	0.6243	20.20
m,p-Xylene	0.7367	20.41
Styrene	1.888	20.80
1,1,2,2-Tetrachloroethane	1.035	20.92
o-Xylene	0.7498	20.92
4-Ethyltoluene	0.6181	22.53
1,3,5-Trimethylbenzene	0.7088	22.65
1,2,4-Trimethylbenzene	0.7536	23.18
m-Dichlorobenzene	0.9643	23.31
Benzyl chloride	1.420	23.32
p-Dichlorobenzene	0.8912	23.41
o-Dichlorobenzene	1.004	23.88
1,2,4-Trichlorobenzene	2.150	26.71
Hexachlorobutadiene	0.4117	27.68

TABLE 6 GC-MS-SIM CALIBRATION TABLE

*** External Standard ***

Operator: JDF 8 Jan 87 10:[illegible] am
Sample Info : SYR 1
Misc Info:
Integration File Name : DATA:SYR2A02A.I
Sequence Index: 1 Bottle Number : 2

Last Update: 8 Jan 87 8:13 am
Reference Peak Window: 5.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.667

Peak Num	Peak Type	Int Type	Ret Time	Signal Description			Compound Name	Area	Amount
1		1	PP	5.020	Mass	85.00 amu	FREON 12	12893	4011 ppbv
2		1	PP	5.654	Mass	50.00 amu	METHYLCHLORI	4445	2586 ppbv
3		1	BP	6.525	Mass	85.00 amu	FREON 114	7067	1215 ppbv
4		1	PB	6.650	Mass	62.00 amu	VINYLCHLORID	2892	1929 ppbv
5		1	BP	7.816	Mass	94.00 amu	METHYLBROMID	2401	1729 ppbv
6		1	BB	8.421	Mass	64.00 amu	ETHYLCHLORID	2134	2769 ppbv
7		1	BV	9.940	Mass	101.00 amu	FREON 11	25069	6460 ppbv
8		1	BP	10.869	Mass	61.00 amu	VINDENECHLOR	5034	1700 ppbv
9		1	BP	11.187	Mass	49.00 amu	DICHLOROMETH	4803	2348 ppbv
10		1	PP	11.225	Mass	41.00 amu	ALLYLCHLORID	761	8247 ppbv
11		1	BP	11.578	Mass	151.00 amu	3CHL3FLUETHA	5477	1672 ppbv
12		1	BP	12.492	Mass	63.00 amu	1,1DICHLOETH	5052	1738 ppbv
13		1	VP	13.394	Mass	61.00 amu	c-1,2DICHLET	4761	1970 ppbv
14		1	PH	13.713	Mass	83.00 amu	CHLOROFORM	5327	1673 ppbv
15		1	BP	14.378	Mass	62.00 amu	1,2DICHLETHA	5009	2263 ppbv
16		1	PB	14.594	Mass	97.00 amu	METHCHLOROFO	6656	2334 ppbv
17		1	VP	15.009	Mass	78.00 amu	BENZENE	8352	2167 ppbv
18		1	VP	15.154	Mass	117.00 amu	CARBONTETRAC	5888	1915 ppbv
19		1	BB	15.821	Mass	63.00 amu	1,2DICHLPROP	3283	1799 ppbv
20		1	BB	16.067	Mass	130.00 amu	TRICHLETHENE	4386	2109 ppbv
21		1	PB	16.941	Mass	75.00 amu	c-1,3DICHLPR	2229	987.3 ppbv
22		1	BP	17.475	Mass	75.00 amu	t-1,3DICHLPR	1626	689.2 ppbv
23		1	BB	17.594	Mass	97.00 amu	1,1,2CHLETHA	2721	1772 ppbv
24		1	BV	17.844	Mass	91.00 amu	TOLUENE	14417	2733 ppbv
25		1	PB	18.463	Mass	107.00 amu	EDB	4070	1365 ppbv
26		1	PH	18.989	Mass	166.00 amu	TETRACHLETHE	6874	2065 ppbv
27		1	PB	19.705	Mass	112.00 amu	CHLOROBENZEN	5648	1524 ppbv
28		1	BP	20.168	Mass	91.00 amu	ETHYLBENZENE	11084	1842 ppbv
29		1	PB	20.372	Mass	91.00 amu	m,p-XYLENE	17989	3750 ppbv
30		1	BV	20.778	Mass	104.00 amu	STYRENE	3145	1695 ppbv
31		1	BH	20.887	Mass	83.00 amu	TETRACHLETHA	4531	1376 ppbv
32		1	BP	20.892	Mass	91.00 amu	o-XYLENE	9798	2010 ppbv
33		1	VV	22.488	Mass	105.00 amu	4-ETHYLTOLUE	7694	1481 ppbv
34		1	VB	22.609	Mass	105.00 amu	1,3,5METHBEN	6731	1705 ppbv
35		1	BB	23.144	Mass	105.00 amu	1,2,4METHBEN	7892	2095 ppbv
36		1	BV	23.272	Mass	146.00 amu	m-DICHLBENZE	3046	1119 ppbv
37		1	VV	23.279	Mass	91.00 amu	BENZYLCHLORI	3880	1006 ppbv
38		1	VB	23.378	Mass	146.00 amu	p-DICHLBENZE	6090	2164 ppbv
39		1	BP	23.850	Mass	146.00 amu	o-DICHLBENZE	2896	1249 ppbv
40		1	BB	26.673	Mass	180.00 amu	1,2,4CHLBENZ	562	767.1 ppbv
41		1	BB	27.627	Mass	225.00 amu	HEXACHLBUTAD	6309	1789 ppbv

TABLE 7 TYPICAL RETENTION TIME (MIN) AND CALIBRATION RESPONSE FACTORS (ppbv/area count) FOR TARGETED VOCs ASSOCIATED WITH FID AND ECD ANALYTICAL SYSTEM

Peak Number[1]	Compound	FID		ECD
		Retention Time (RT), minutes	Response Factor (RF) (ppbv/area counts)	Response Factor (ppbv/area count x 10^{-5})
1	Freon 12	3.65	3.465	13.89
2	Methyl chloride	4.30	0.693	
3	Freon 114	5.13	0.578	22.32
4	Vinyl chloride	5.28	0.406	
5	Methyl bromide	6.44		26.34
6	Ethyl chloride	7.06	0.413	
7	Freon 11	8.60	6.367	1.367
8	Vinylidene chloride	9.51	0.347	
9	Dichloromethane	9.84	0.903	
10	Trichlorotrifluoroethane	10.22	0.374	3.955
11	1,1-Dichloroethane	11.10	0.359	
12	cis-1,2-Dichloroethylene	11.99	0.368	
13	Chloroform	12.30	1.059	11.14
14	1,2-Dichloroethane	12.92	0.409	
15	Methyl chloroform	13.12	0.325	3.258
16	Benzene	13.51	0.117	
17	Carbon tetrachloride	13.64	1.451	1.077
18	1,2-Dichloropropane	14.26	0.214	
19	Trichloroethylene	14.50	0.327	8.910
20	cis-1,3-Dichloropropene	15.31		
21	trans-1,3-Dichloropropene	15.83		
22	1,1,2-Trichloroethane	15.93	0.336	
23	Toluene	16.17	0.092	
24	1,2-Dibromoethane (EDB)	16.78	0.366	5.137
25	Tetrachloroethylene	17.31	0.324	1.449
26	Chlorobenzene	18.03	0.120	
27	Ethylbenzene	18.51	0.092	
28	m,p-Xylene	18.72	0.095	
29	Styrene	19.12	0.143	
30	1,1,2,2-Tetrachloroethane	19.20		9.856
31	o-Xylene	19.23		
32	4-Ethyltoluene	20.82	0.100	
33	1,3,5-Trimethylbenzene	20.94	0.109	
34	1,2,4-Trimethylbenzene	21.46	0.111	
35	m-Dichlorobenzene	21.50		
36	Benzyl chloride	21.56		
37	p-Dichlorobenzene	21.67	0.188	
38	o-Dichlorobenzene	22.12	0.188	
39	1,2,4-Trichlorobenzene	24.88	0.667	
40	Hexachlorobutadiene	25.82	0.305	1.055

[1] Refer to Figures 15 and 16 for peak location

TABLE 8 TYPICAL RETENTION TIME (minutes) FOR SELECTED ORGANICS USING GC-FID-ECD-PID* ANALYTICAL SYSTEM

Compound	Retention Time (minutes)		
	FID	ECD	PID
Acetylene	2.984	--	--
1,3-Butadiene	3.599	--	3.594
Vinyl chloride	3.790	--	3.781
Chloromethane	5.137	--	--
Chloroethane	5.738	--	--
Bromoethane	8.154	--	--
Methylene Chloride	9.232	--	9.218
trans-1,2-Dichloroethylene	10.077	--	10.065
1,1-Dichloroethane	11.190	--	--
Chloroprene	11.502	--	11.491
Perfluorobenzene	13.077	13.078	13.069
Bromochloromethane	13.397	13.396	13.403
Chloroform	13.768	13.767	13.771
1,1,1-Trichloroethane	14.151	14.153	14.158
Carbon Tetrachloride	14.642	14.667	14.686
Benzene/1,2-Dichloroethane	15.128	--	15.114
Perfluorotoluene	15.420	15.425	15.412
Trichloroethylene	17.022	17.024	17.014
1,2-Dichloropropene	17.491	17.805	17.522
Bromodichloromethane	18.369	--	--
trans-1,3-Dichloropropylene	19.694	19.693	19.688
Toluene	20.658	--	20.653
cis-1,3-Dichloropropylene	21.461	21.357	21.357
1,1,2-Trichloroethane	21.823	--	--
Tetrachloroethylene	22.340	22.346	22.335
Dibromochloromethane	22.955	22.959	22.952
Chlorobenzene	24.866	--	24.861
m/p-Xylene	25.763	--	25.757
Styrene/o-Xylene	27.036	--	27.030
Bromofluorobenzene	28.665	28.663	28.660
1,1,2,2-Tetrachloroethane	29.225	29.227	29.228
m-Dichlorobenzene	32.347	32.345	32.342
p-Dichlorobenzene	32.671	32.669	32.666
o-Dichlorobenzene	33.885	33.883	33.880

* Varian® 3700 GC equipped with J & W Megabore® DB 624 Capillary Column (30 m X 0.53 I.D. mm) using helium carrier gas.

TABLE 9 GC-MS-SIM CALIBRATION TABLE

Last Update: 18 Dec 86 7:54 am
Reference Peak Window: 5.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.000

Ret Time	Pk#	Signal	Descr		Amt pptv	Lvl	[Area]	Pk-Type	Partial Name
5.008	1	Mass	85.00	amu	13620	1	72974	1	FREON 12
5.690	2	Mass	50.00	amu	12720	1	36447	1	METHYLCHLORID
6.552	3	Mass	85.00	amu	8380	1	81251	1	FREON 114
6.709	4	Mass	62.00	amu	8050	1	20118	1	VINYLCHLORIDE
7.831	5	Mass	94.00	amu	12210	1	28265	1	METHYLBROMIDE
8.431	6	Mass	64.00	amu	12574	1	16149	1	ETHYLCHLORIDE
9.970	7	Mass	101.00	amu	12380	1	80088	1	FREON 11
10.927	8	Mass	61.00	amu	7890	1	38954	1	VINDENECHLORI
11.209	9	Mass	49.00	amu	12760	1	43507	1	DICHLOROMETHA
11.331	10	Mass	41.00	amu	12650	1	1945	1	ALLYLCHLORIDE
11.595	11	Mass	151.00	amu	7420	1	40530	1	3CHL3FLUETHAN
12.502	12	Mass	63.00	amu	12710	1	61595	1	1,1DICHLOETHA
13.403	13	Mass	61.00	amu	12630	1	50900	1	c-1,2DICHLETH
13.747	14	Mass	83.00	amu	7670	1	40565	1	CHLOROFORM
14.387	15	Mass	62.00	amu	9040	1	33356	1	1,2DICHLETHAN
14.623	16	Mass	97.00	amu	8100	1	38503	1	METHCHLOFOFOR
15.038	17	Mass	78.00	amu	10760	1	69119	1	BENZENE
15.183	18	Mass	117.00	amu	8340	1	42737	1	CARBONTETRACH
15.829	19	Mass	63.00	amu	12780	1	38875	1	1,2DICHLPROPA
16.096	20	Mass	130.00	amu	8750	1	30331	1	TRICHLETHENE
16.956	21	Mass	75.00	amu	4540	1	17078	1	c-1,3DICHLPRO
17.492	22	Mass	75.00	amu	3380	1	13294	1	t-1,3DICHLPRO
17.610	23	Mass	97.00	amu	12690	1	32480	1	1,1,2CHLETHAN
17.862	24	Mass	91.00	amu	10010	1	88036	1	TOLUENE
18.465	25	Mass	107.00	amu	6710	1	33350	1	EDB
19.012	26	Mass	166.00	amu	7830	1	43454	1	TETRACHLETHEN
19.729	27	Mass	112.00	amu	7150	1	44224	1	CHLOROBENZENE
20.195	28	Mass	91.00	amu	12740	1	127767	1	ETHYLBENZENE
20.407	29	Mass	91.00	amu	25400	1	200973	1	m,p-XYLENE
20.806	30	Mass	104.00	amu	12390	1	38332	1	STYRENE
20.916	31	Mass	83.00	amu	11690	1	64162	1	TETRACHLETHAN
20.921	32	Mass	91.00	amu	11065	1	90096	1	o-XYLENE
22.528	33	Mass	105.00	amu	12560	1	108747	1	4-ETHYLTOLUEN
22.648	34	Mass	105.00	amu	12620	1	83666	1	1,3,5METHBENZ
23.179	35	Mass	105.00	amu	12710	1	79833	1	1,2,4METHBENZ
23.307	36	Mass	146.00	amu	12650	1	57409	1	m-DICHLBENZEN
23.317	37	Mass	91.00	amu	7900	1	50774	1	BENZYLCHLORID
23.413	38	Mass	146.00	amu	12390	1	58127	1	p-DICHLBENZEN
23.885	39	Mass	146.00	amu	13510	1	52233	1	o-DICHLBENZEN
26.714	40	Mass	180.00	amu	15520	1	18967	1	1,2,4CHLBENZE
27.680	41	Mass	225.00	amu	7470	1	43920	1	HEXACHLBUTADI

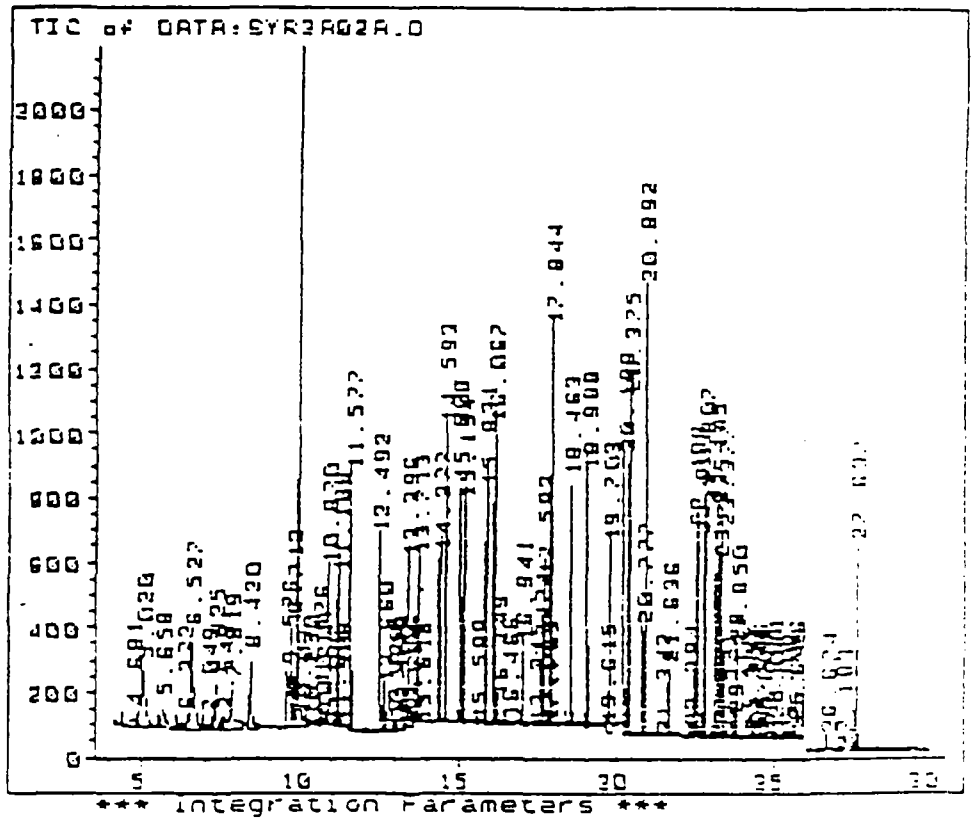
TABLE 10 EXAMPLE OF HARD-COPY OF GC-MS-SIM ANALYSIS

Data file: DATA:SYR2A02A.D
File type: GC / MS DATA FILE

Name Info: SYR 1
Misc Info:
Operator : JDP

Date : 8 Jan 87 10:02 am
Instrment: MS_5970
Inlet : GC

Sequence index : 1
Als bottle num : 2
Replicate num : 1



*** Integration Parameters ***

FALSE : Shoulder Detection Enabled
0.020 : Expected Peak Width (Min)
11 : Initial Peak Detection Threshold

4.000 THRESHOLD 5.000
4.000 PEAK_WIDTH 0.200
9.300 PEAK_WIDTH 0.060

TABLE 10 EXAMPLE OF HARD-COPY OF GC-MS-SIM ANALYSIS (cont.)

Operator: JDF 8 Jan 87 10:00 am
Sample Info : SYR 1
Misc Info:
Integration File Name : DATA:SYR2A02A.I
Sequence Index: 1 Bottle Number : 2

Last Update: 8 Jan 87 8:13 am
Reference Peak Window: 5.00 Absolute Minutes
Non-Reference Peak Window: 0.40 Absolute Minutes
Sample Amount: 0.000 Uncalibrated Peak RF: 0.000 Multiplier: 1.667

Peak Num	Type	Int Type	Ret Time	Signal Description			Compound Name	Area	Amount
1		1 FP	5.020	Mass	85.00	amu	FREON 12	12393	4011 ppbv
2		1 FP	5.654	Mass	50.00	amu	METHYLCHLORI	4445	2586 ppbv
3		1 BP	6.525	Mass	85.00	amu	FREON 114	7067	1215 ppbv
4		1 PB	6.650	Mass	62.00	amu	VINYLCHLORID	2392	1329 ppbv
5		1 BP	7.816	Mass	94.00	amu	METHYLBROMID	2401	1729 ppbv
6		1 BB	8.421	Mass	64.00	amu	ETHYLCHLORID	2174	2769 ppbv
7		1 BV	9.940	Mass	101.00	amu	FREON 11	25069	6460 ppbv
8		1 BP	10.869	Mass	61.00	amu	VINDENECHLOR	5034	1700 ppbv
9		1 BP	11.167	Mass	49.00	amu	DICHLOROMETH	4607	2048 ppbv
10		1 PP	11.225	Mass	41.00	amu	ALLYLCHLORID	761	8247 ppbv
11		1 BP	11.578	Mass	151.00	amu	3CHL3FLUETHA	5477	1672 ppbv
12		1 BP	12.492	Mass	63.00	amu	1,1DICHLOETH	5052	1738 ppbv
13		1 VP	13.394	Mass	61.00	amu	c-1,2DICHLET	4761	1970 ppbv
14		1 PH	13.713	Mass	83.00	amu	CHLOROFORM	5327	1673 ppbv
15		1 BP	14.378	Mass	62.00	amu	1,2DICHLETHA	5009	2263 ppbv
16		1 PB	14.594	Mass	97.00	amu	METHCHLOROFO	6656	2334 ppbv
17		1 VP	15.009	Mass	78.00	amu	BENZENE	8352	2167 ppbv
18		1 VP	15.154	Mass	117.00	amu	CARBONTETRAC	5883	1915 ppbv
19		1 BB	15.821	Mass	63.00	amu	1,2DICHLPROP	3293	1797 ppbv
20		1 BB	16.067	Mass	130.00	amu	TRICHLETHENE	4336	2109 ppbv
21		1 PB	16.941	Mass	75.00	amu	c-1,3DICHLPR	2228	987.3 ppbv
22		1 BP	17.475	Mass	75.00	amu	t-1,3DICHLPR	1626	689.2 ppbv
23		1 BB	17.594	Mass	97.00	amu	1,1,2CHLETHA	2721	1772 ppbv
24		1 BV	17.844	Mass	91.00	amu	TOLUENE	14417	2722 ppbv
25		1 PB	18.463	Mass	107.00	amu	EDB	4070	1365 ppbv
26		1 PH	18.989	Mass	166.00	amu	TETRACHLETHE	6874	2065 ppbv
27		1 PB	19.705	Mass	112.00	amu	CHLOROBENZEN	5648	1524 ppbv
28		1 BP	20.168	Mass	91.00	amu	ETHYLBENZENE	11084	1842 ppbv
29		1 PB	20.372	Mass	91.00	amu	m,p-XYLENE	17989	2790 ppbv
30		1 BV	20.776	Mass	104.00	amu	STYRENE	3145	1695 ppbv
31		1 BH	20.887	Mass	83.00	amu	TETRACHLETHA	4531	1376 ppbv
32		1 BP	20.892	Mass	91.00	amu	o-XYLENE	9798	2010 ppbv
33		1 VV	22.488	Mass	105.00	amu	4-ETHYLTOLUE	7694	1481 ppbv
34		1 VB	22.609	Mass	105.00	amu	1,3,5METHBEN	6731	1705 ppbv
35		1 BB	23.144	Mass	105.00	amu	1,2,4METHBEN	7892	2095 ppbv
36		1 BV	23.273	Mass	146.00	amu	m-DICHLBENZE	3046	1119 ppbv
37		1 VV	23.279	Mass	91.00	amu	BENZYLCHLORI	3880	1006 ppbv
38		1 VB	23.378	Mass	146.00	amu	p-DICHLBENZE	6090	2164 ppbv
39		1 BP	23.850	Mass	146.00	amu	o-DICHLBENZE	2396	1249 ppbv
40		1 BB	26.673	Mass	180.00	amu	1,2,4CHLBENZ	562	767.1 ppbv
41		1 BB	27.637	Mass	225.00	amu	HEXACHLBUTAD	6309	1739 ppbv

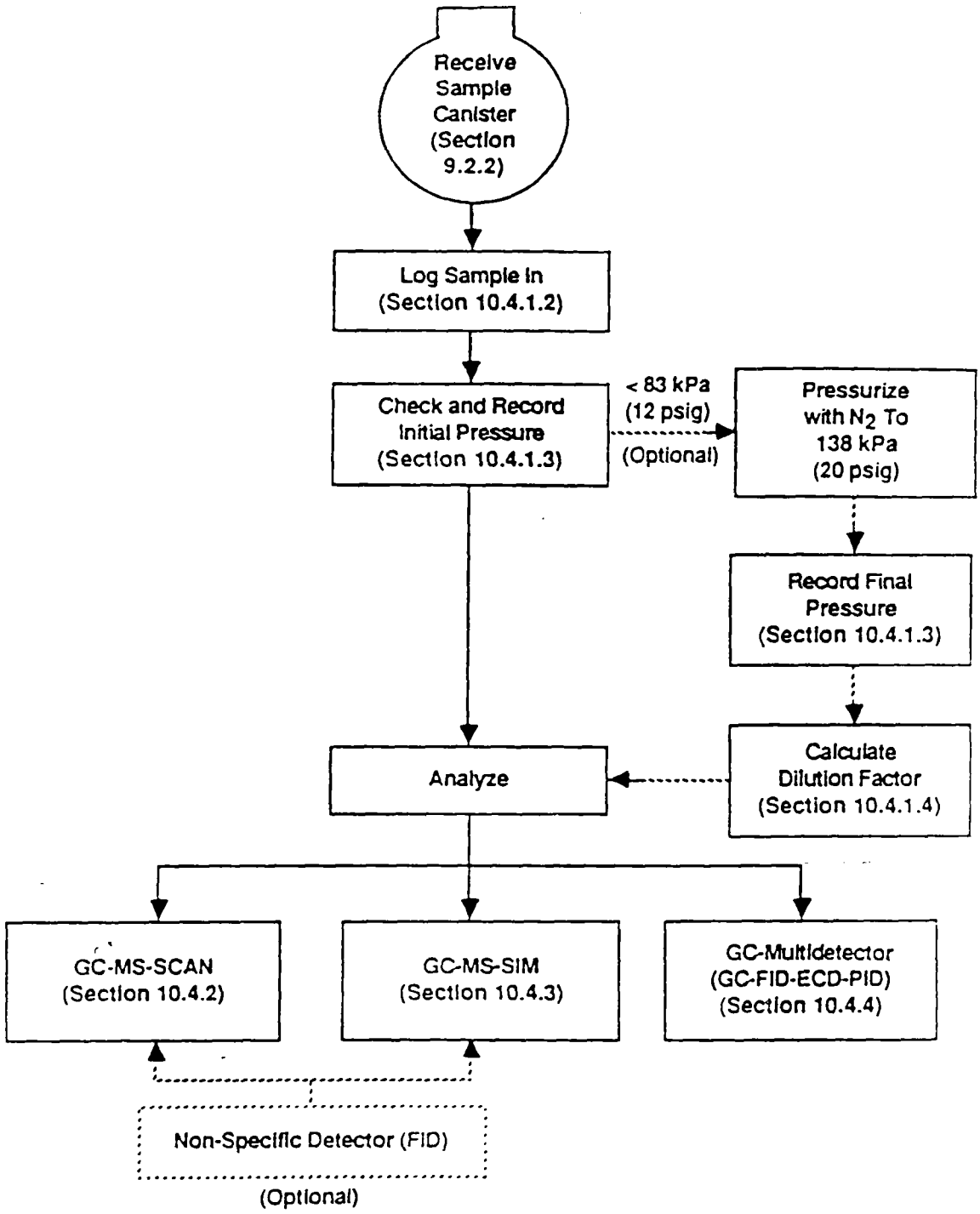


FIGURE 1. ANALYTICAL SYSTEMS AVAILABLE FOR CANISTER VOC IDENTIFICATION AND QUANTITATION

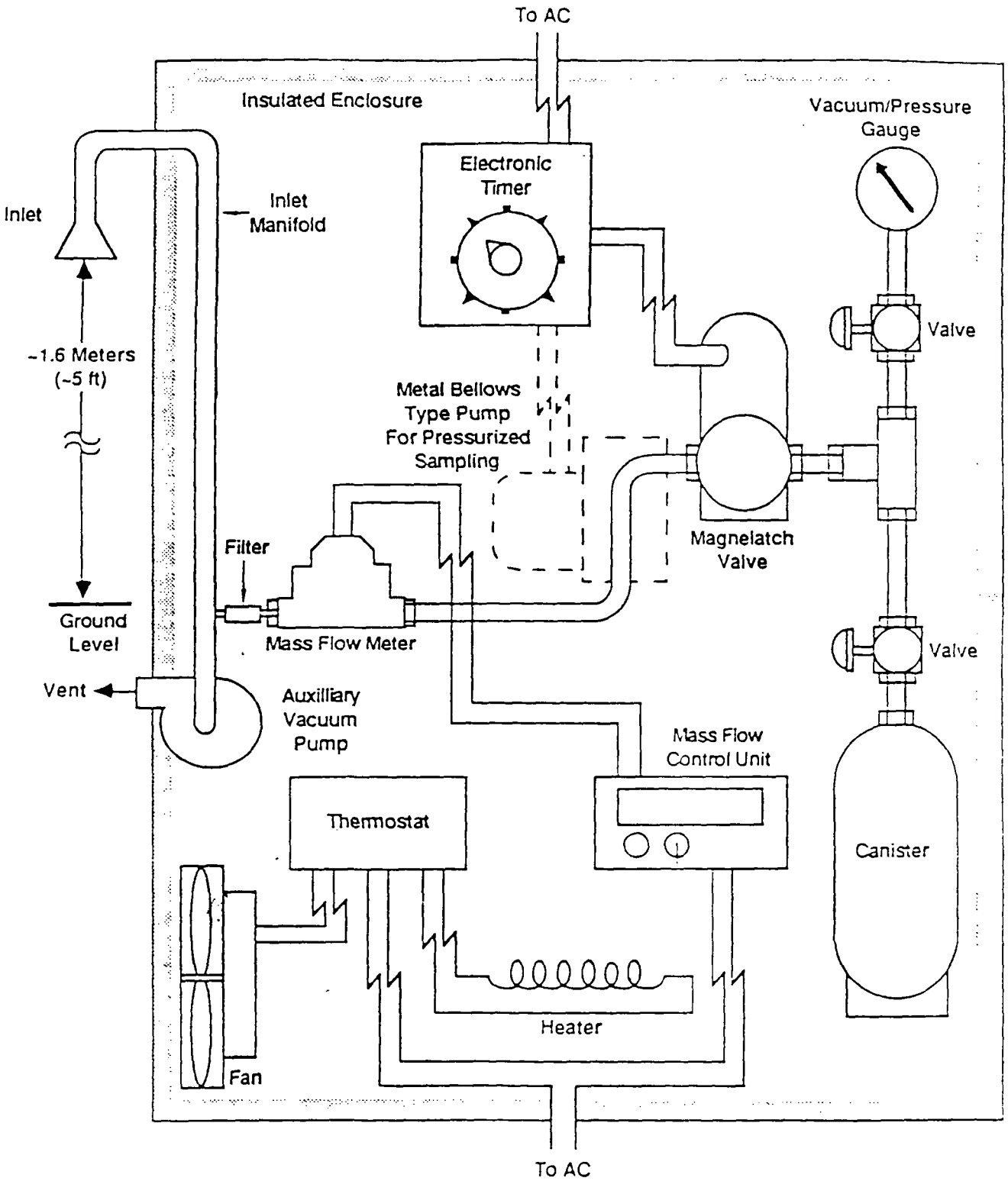


FIGURE 2. SAMPLER CONFIGURATION FOR SUBATMOSPHERIC PRESSURE OR PRESSURIZED CANISTER SAMPLING

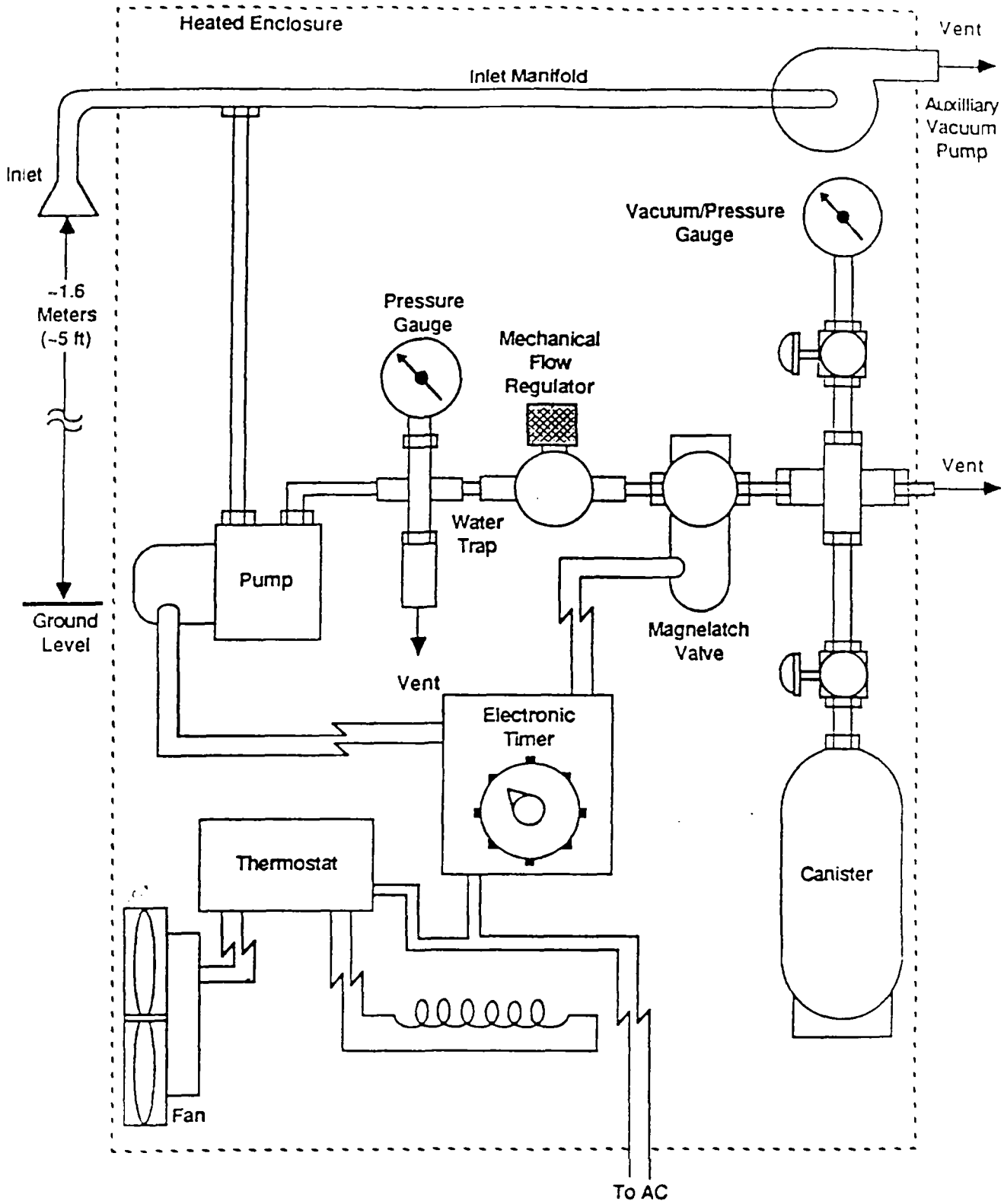


FIGURE 3. ALTERNATIVE SAMPLER CONFIGURATION FOR PRESSURIZED CANISTER SAMPLING

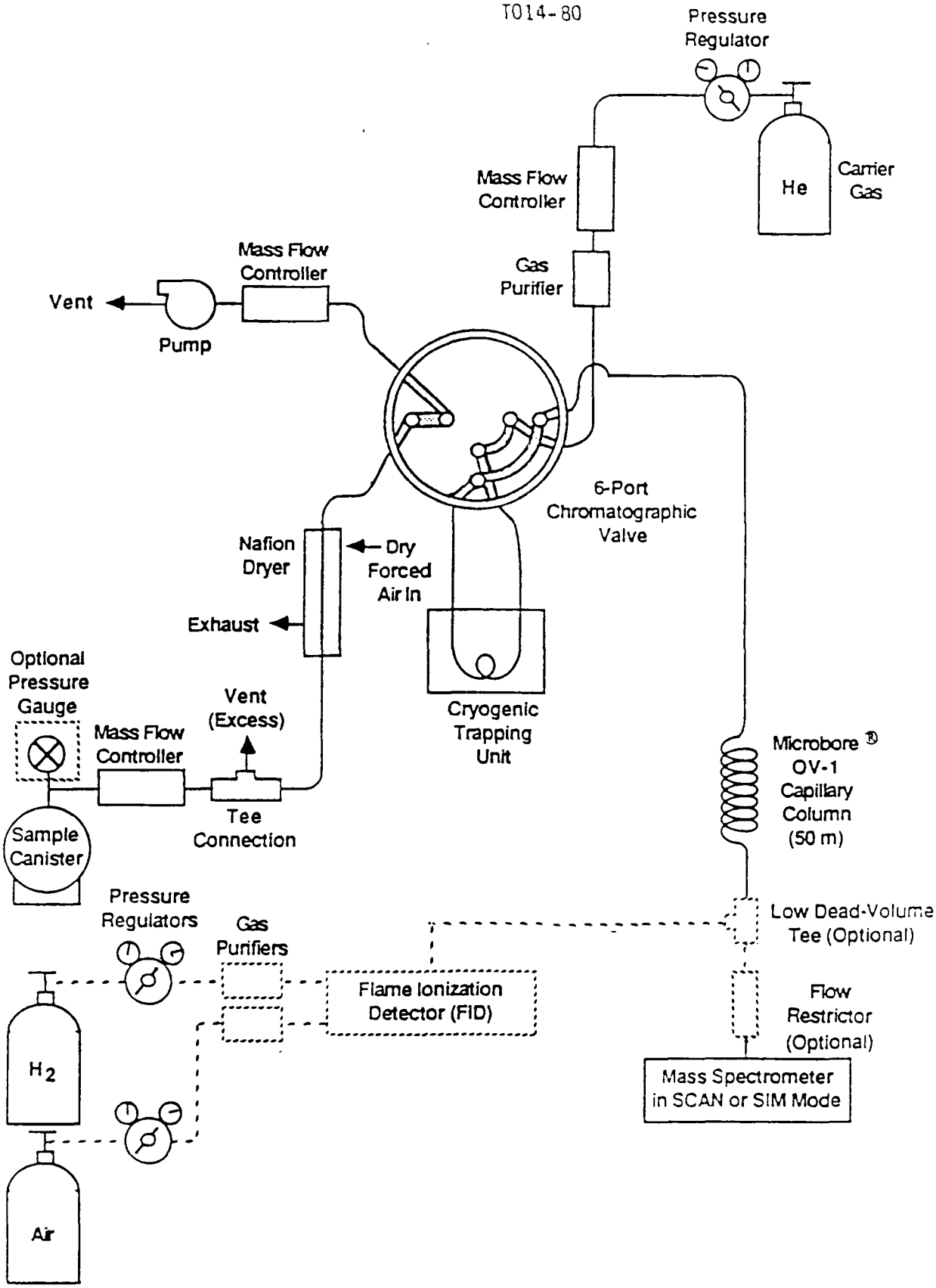


FIGURE 4. CANISTER ANALYSIS UTILIZING GC-MS-SCAN-SIM ANALYTICAL SYSTEM WITH OPTIONAL FLAME IONIZATION DETECTOR

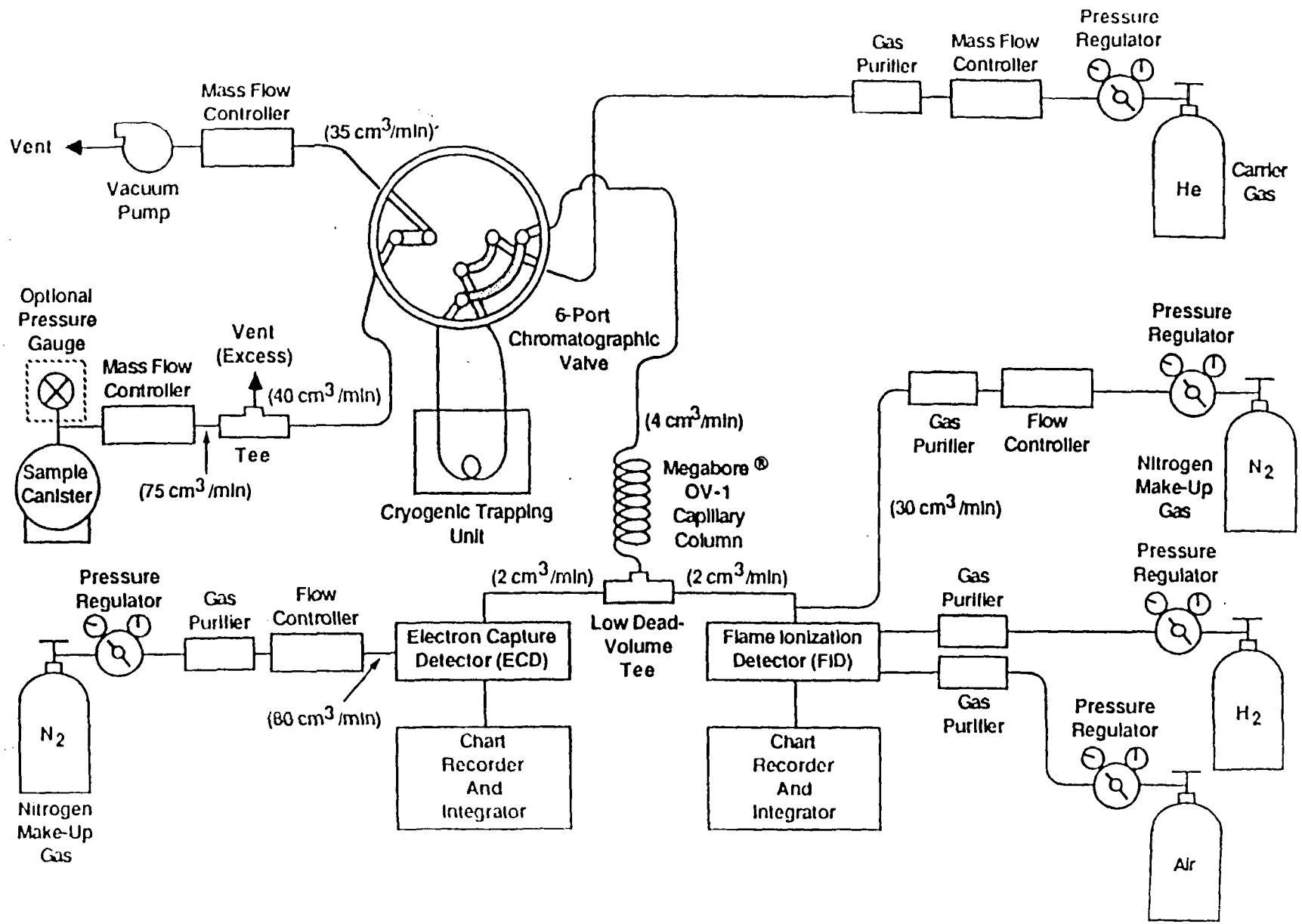


FIGURE 5. GC-FID-ECD ANALYTICAL SYSTEM WITH THE 6-PORT CHROMATOGRAPHIC VALVE IN THE SAMPLE DESORPTION MODE.

T014-81

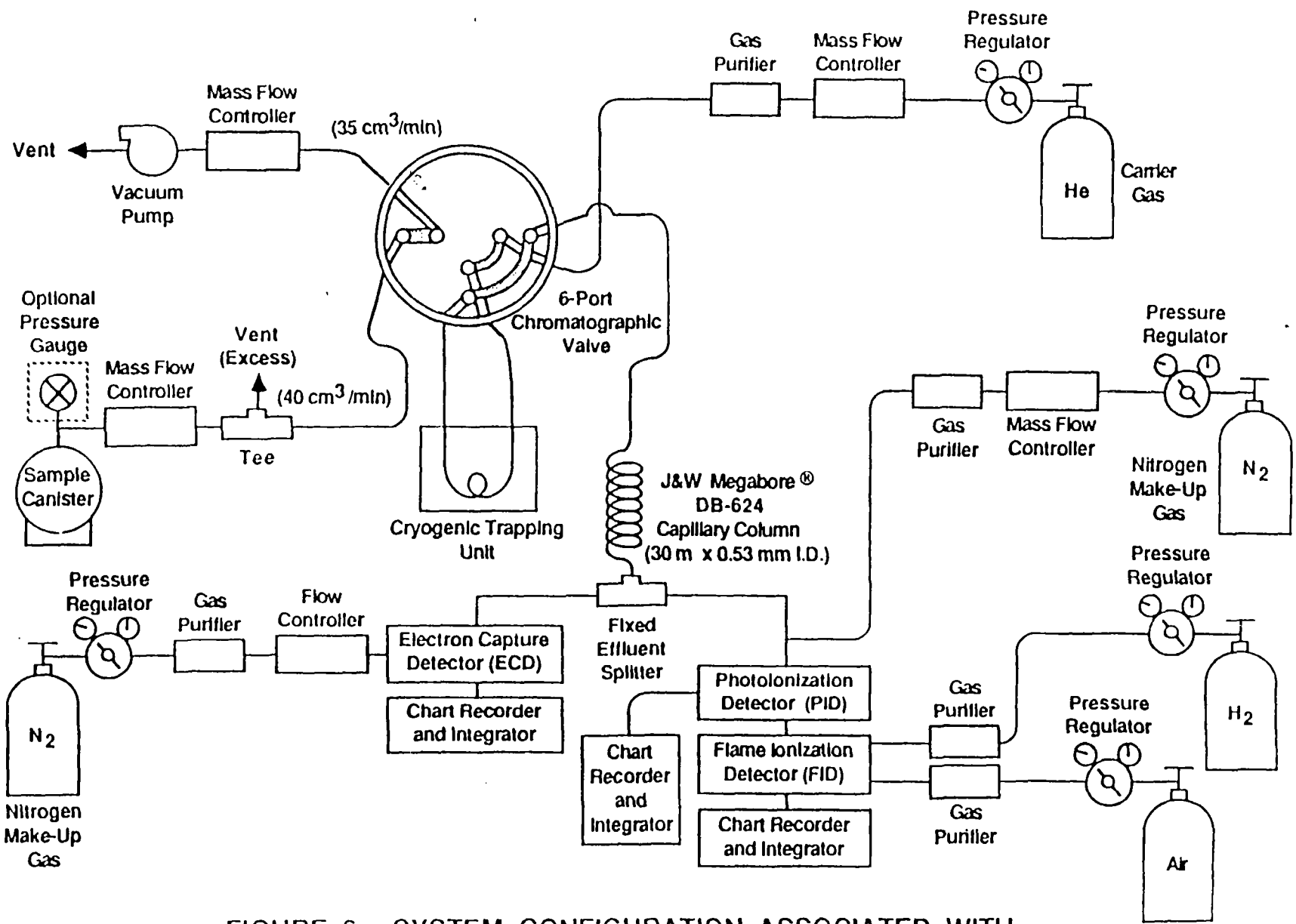


FIGURE 6. SYSTEM CONFIGURATION ASSOCIATED WITH THE GC-FID-ECD-PID ANALYTICAL SYSTEM

T014-82

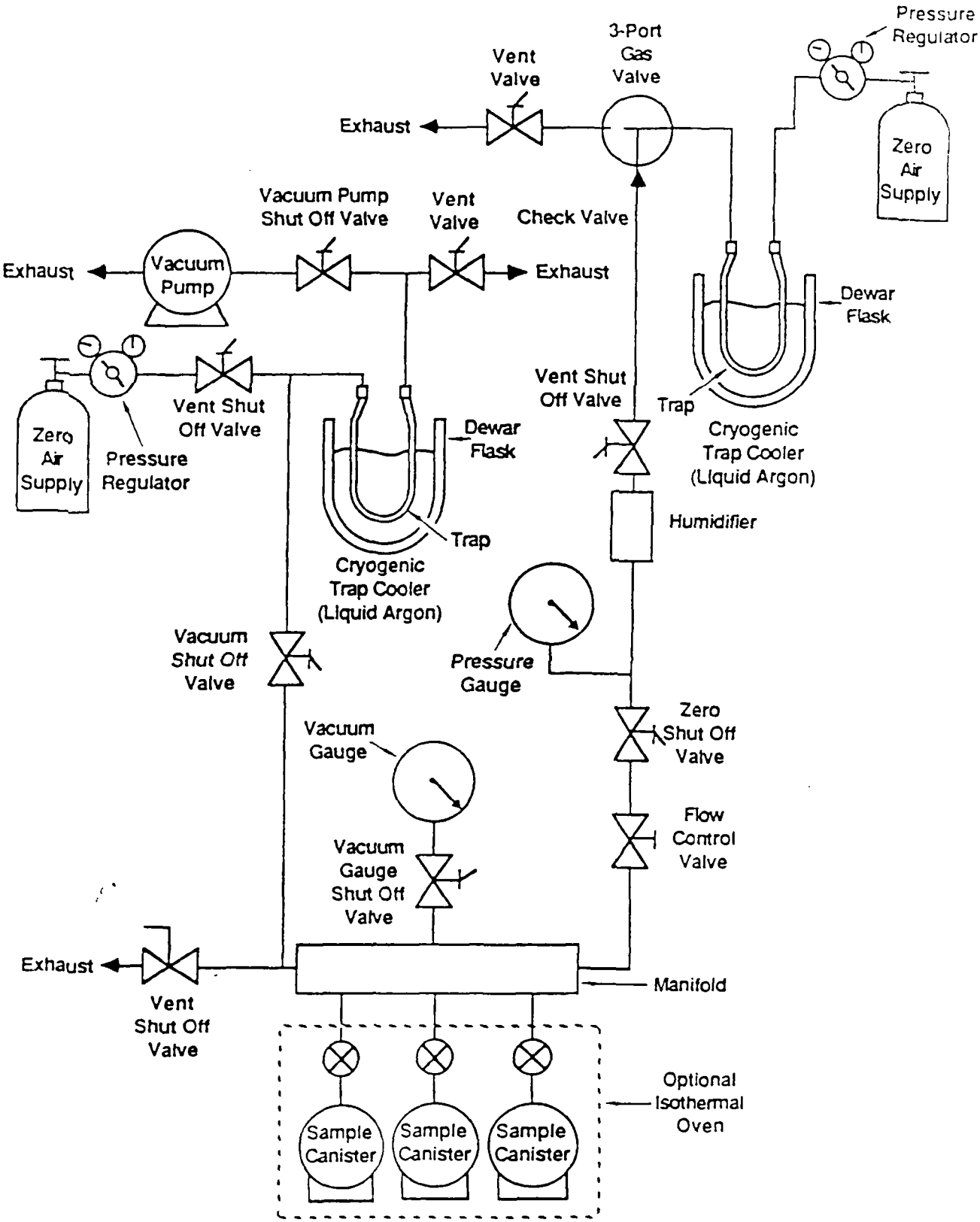


FIGURE 7. CANISTER CLEANING SYSTEM

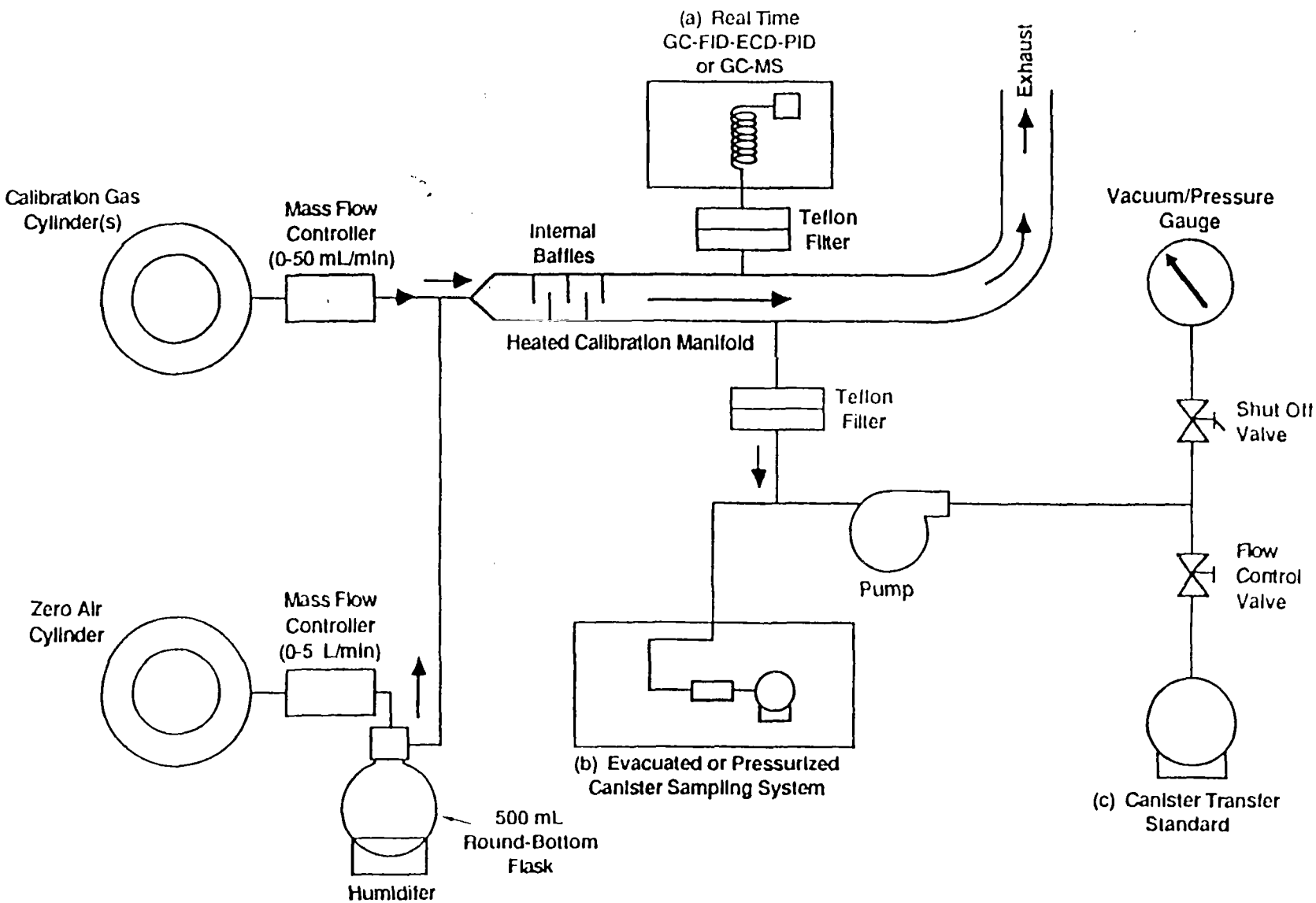
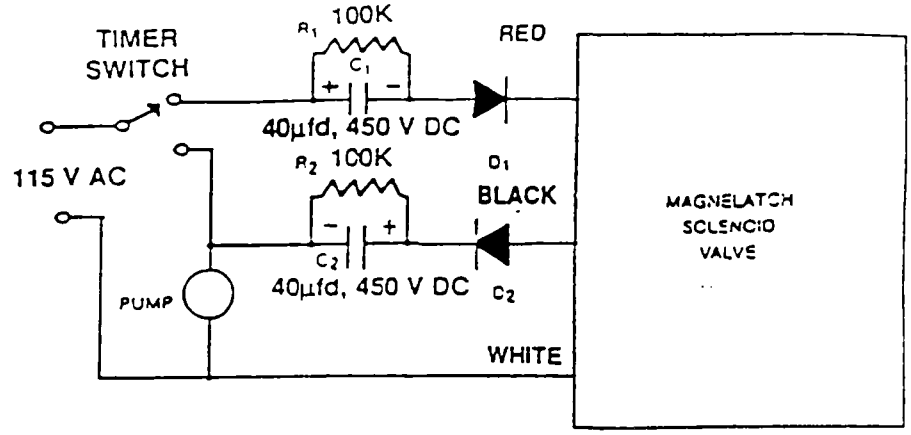


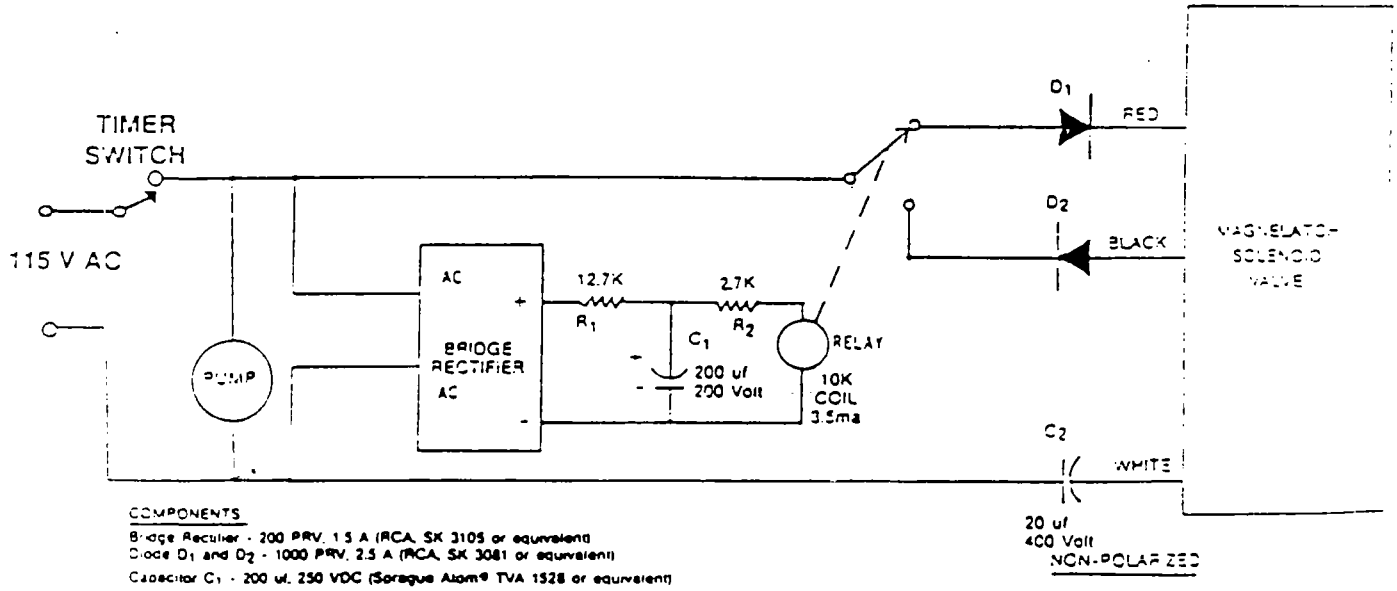
FIGURE 8. SCHEMATIC OF CALIBRATION SYSTEM AND MANIFOLD FOR (a) ANALYTICAL SYSTEM CALIBRATION, (b) TESTING CANISTER SAMPLING SYSTEM AND (c) PREPARING CANISTER TRANSFER STANDARDS.

T014-84



COMPONENTS
Capacitor C_1 and C_2 - 40 uf, 450 VDC (Sprague Atom® TVA 1712 or equivalent)
Resistor R_1 and R_2 - 0.5 watt, 5% tolerance
Diode D_1 and D_2 - 1000 PRV 2.5 A (RCA SK 3081 or equivalent)

(a). Simple Circuit For Operating Magnelatch Valve



COMPONENTS
Bridge Rectifier - 200 PRV, 1.5 A (RCA SK 3105 or equivalent)
Diode D_1 and D_2 - 1000 PRV, 2.5 A (RCA SK 3081 or equivalent)
Capacitor C_1 - 200 uf, 250 VDC (Sprague Atom® TVA 1528 or equivalent)
Capacitor C_2 - 20 uf, 400 VDC Non-Polarized (Sprague Atom® TVAN 1652 or equivalent)
Relay - 10,000 ohm coil, 3.5 ma (AMF Potter and Brumfield, KCP 5 or equivalent)
Resistor R_1 and R_2 - 0.5 watt, 5% tolerance

(b). Improved Circuit Designed To Handle Power Interruptions

FIGURE 9. ELECTRICAL PULSE CIRCUITS FOR DRIVING SKINNER MAGNELATCH SOLENOID VALVE WITH A MECHANICAL TIMER

CANISTER SAMPLING FIELD DATA SHEET

A. GENERAL INFORMATION

SITE LOCATION: _____
SITE ADDRESS: _____
_____
_____
SAMPLING DATE: _____

SHIPPING DATE: _____
CANISTER SERIAL NO.: _____
SAMPLER ID: _____
OPERATOR: _____
CANISTER LEAK CHECK DATE: _____

B. SAMPLING INFORMATION

TEMPERATURE

	INTERIOR	AMBIENT	MAXIMUM	MINIMUM
START				
STOP				

PRESSURE

CANISTER PRESSURE	

SAMPLING TIMES

	LOCAL TIME	ELAPSED TIME METER READING
START		
STOP		

FLOW RATES

MANIFOLD FLOW RATE	CANISTER FLOW RATE	FLOW CONTROLLER READOUT

SAMPLING SYSTEM CERTIFICATION DATE: _____
QUARTERLY RECERTIFICATION DATE: _____

C. LABORATORY INFORMATION

DATE RECEIVED: _____
RECEIVED BY: _____
INITIAL PRESSURE: _____
FINAL PRESSURE: _____
DILUTION FACTOR: _____
ANALYSIS
GC-FID-ECD DATE: _____
GC-MSD-SCAN DATE: _____
GC-MSD-SIM DATE: _____
RESULTS*: _____
_____
GC-FID-ECD: _____
GC-MSD-SCAN: _____
GC-MSD-SIM: _____

_____
SIGNATURE-TITLE

* ATTACH DATA SHEETS

FIGURE 10. CANISTER SAMPLING FIELD DATA SHEET

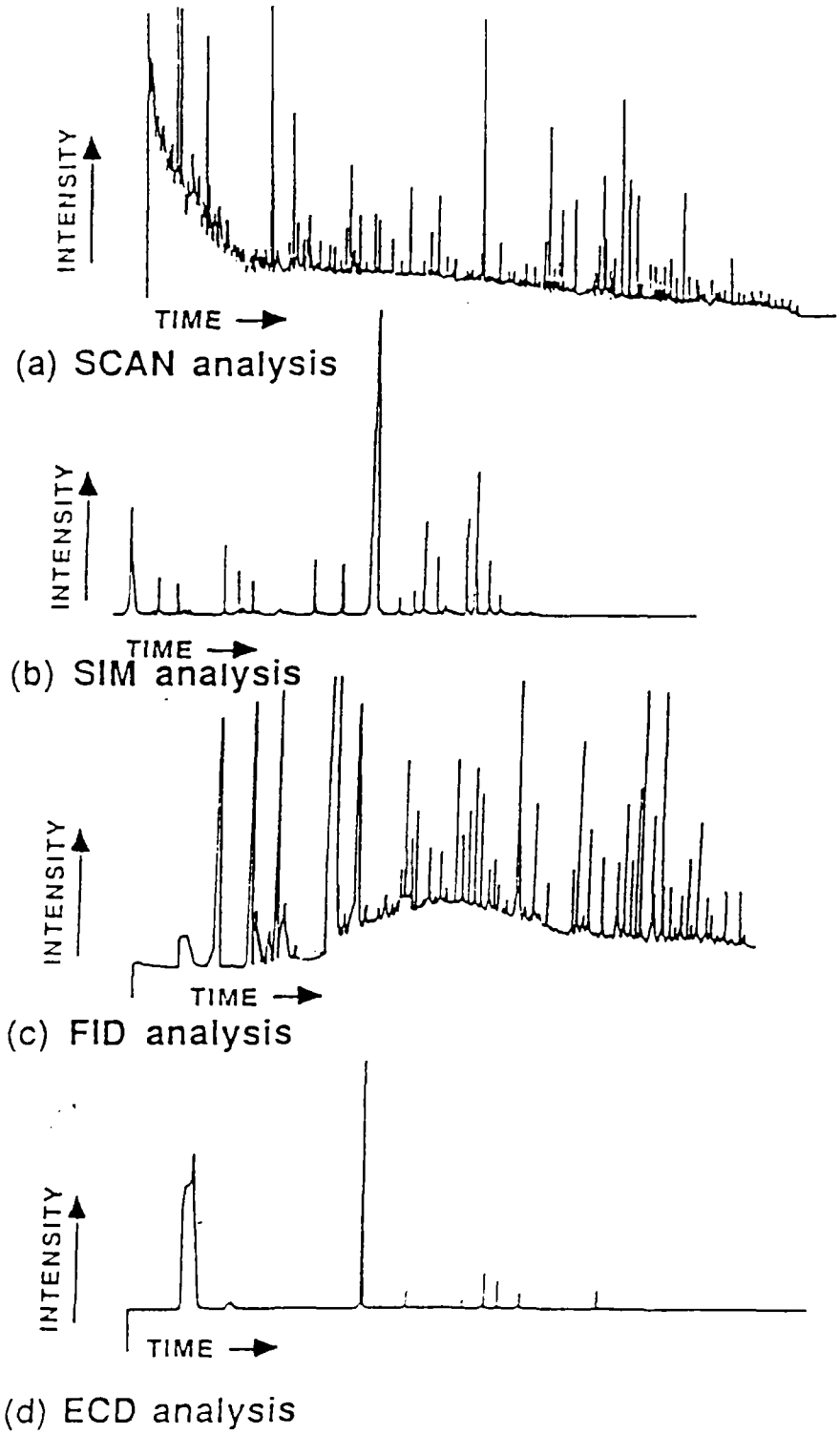


FIGURE 11. TYPICAL CHROMATOGRAMS OF A VOC SAMPLE ANALYZED BY GC-MS-SCAN-SIM MODE AND GC-MULTIDETECTOR MODE

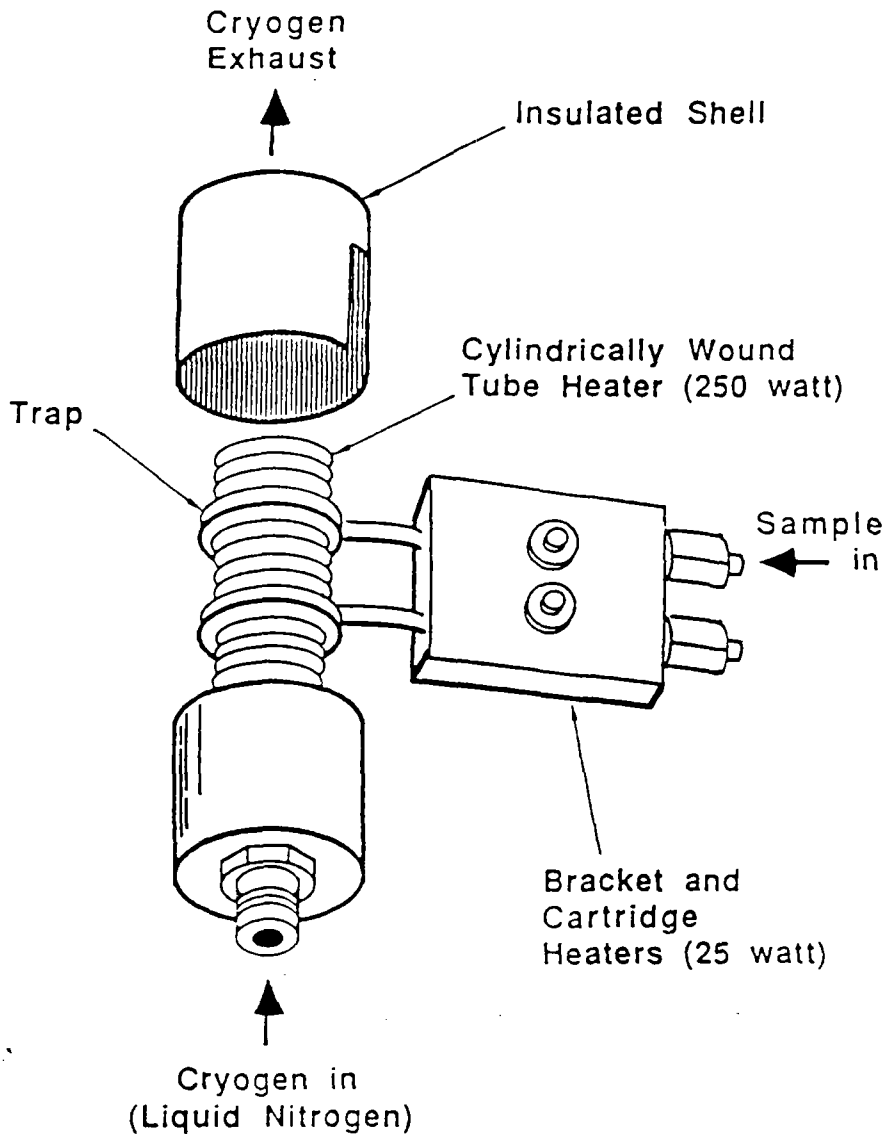


FIGURE 12. CRYOGENIC TRAPPING UNIT

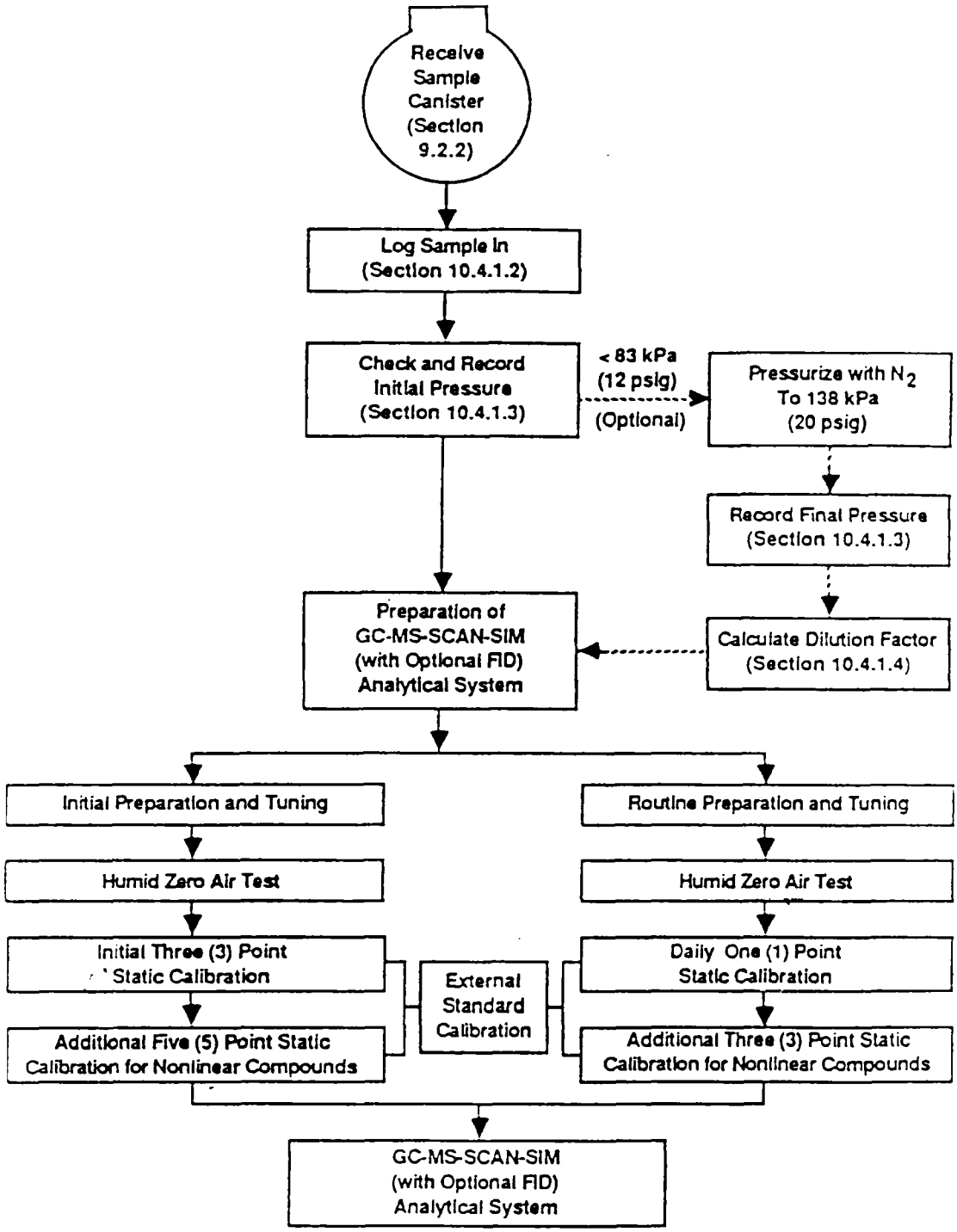


FIGURE 13. FLOWCHART OF GC-MS-SCAN-SIM ANALYTICAL SYSTEM PREPARATION (WITH OPTIONAL FID SYSTEM)

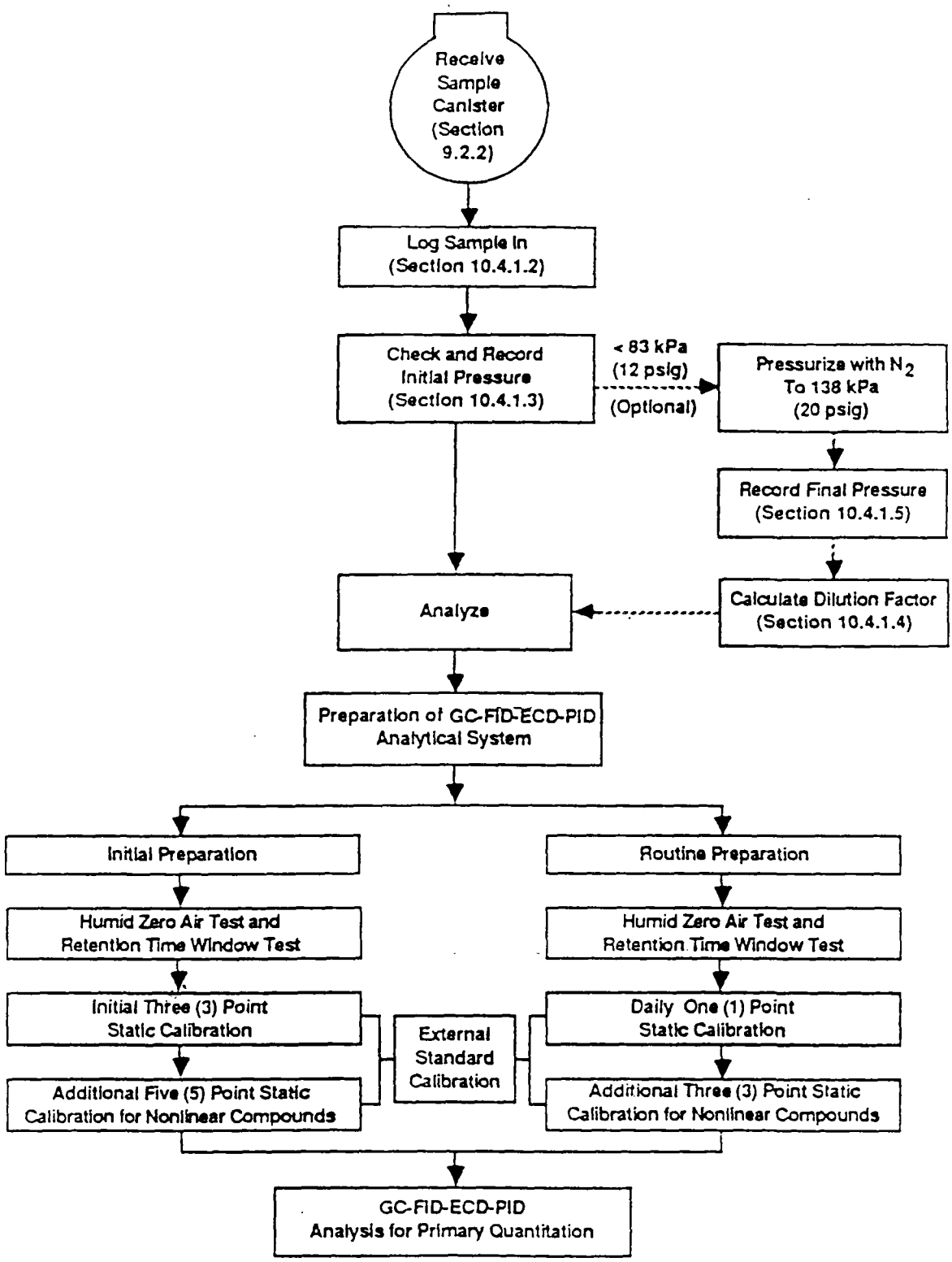
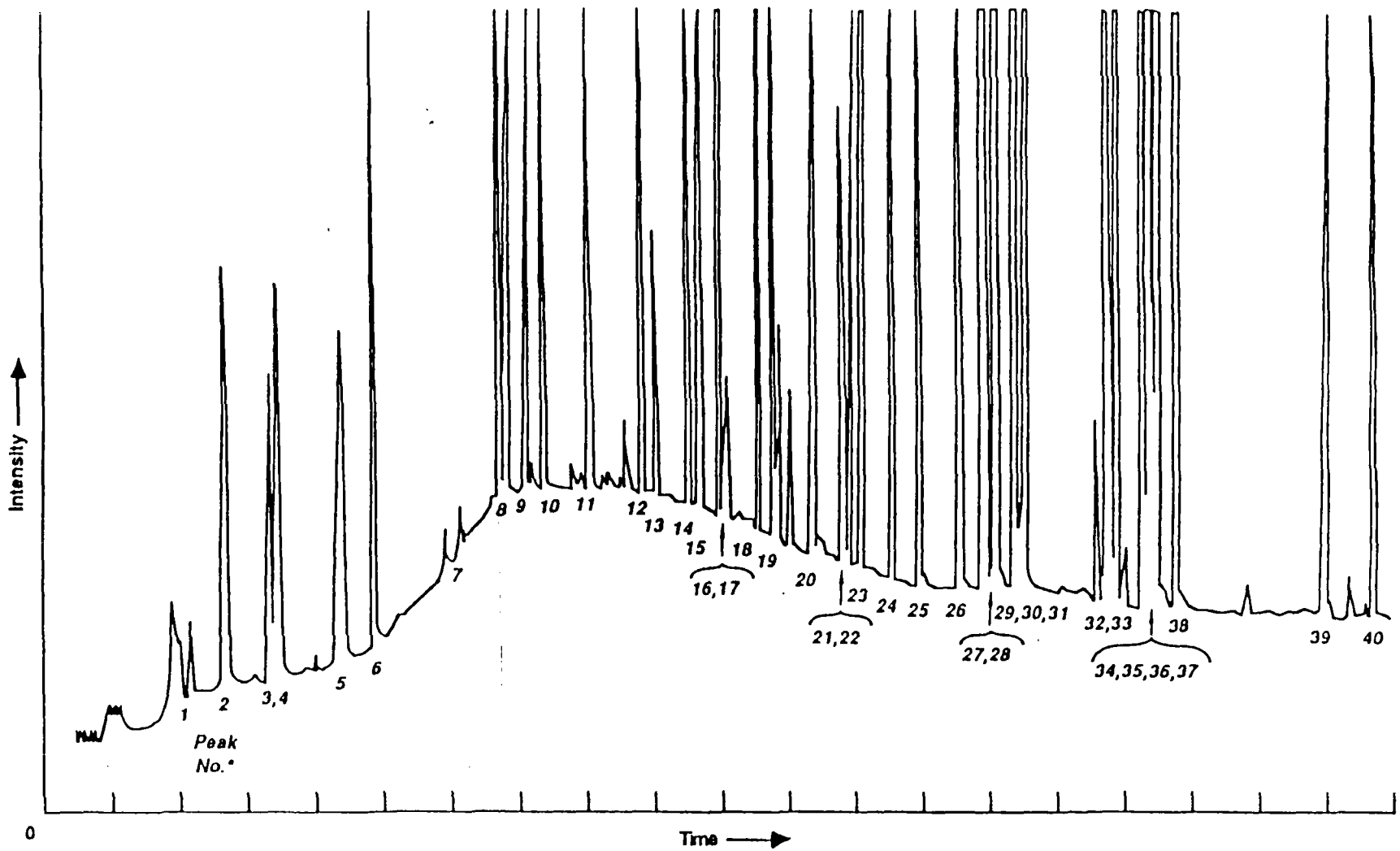


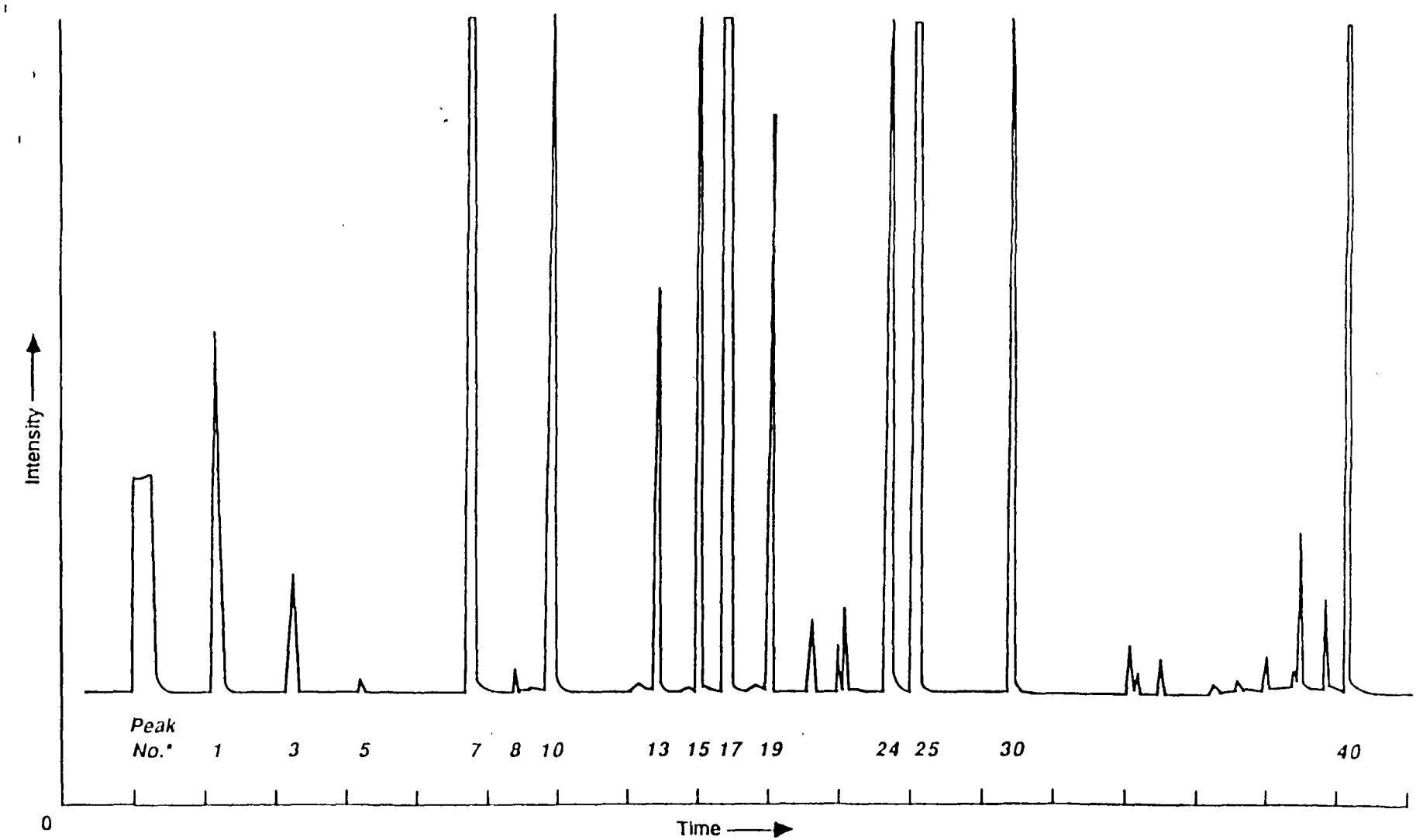
FIGURE 14. FLOWCHART OF GC-FID-ECD-PID ANALYTICAL SYSTEM PREPARATION



* See Table 7.0 For Peak Identification

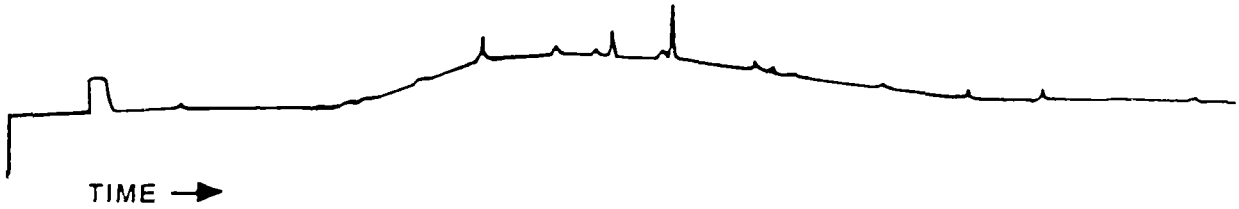
FIGURE 15. TYPICAL FID RESPONSE TO SELECTIVE VOCs

T014-91

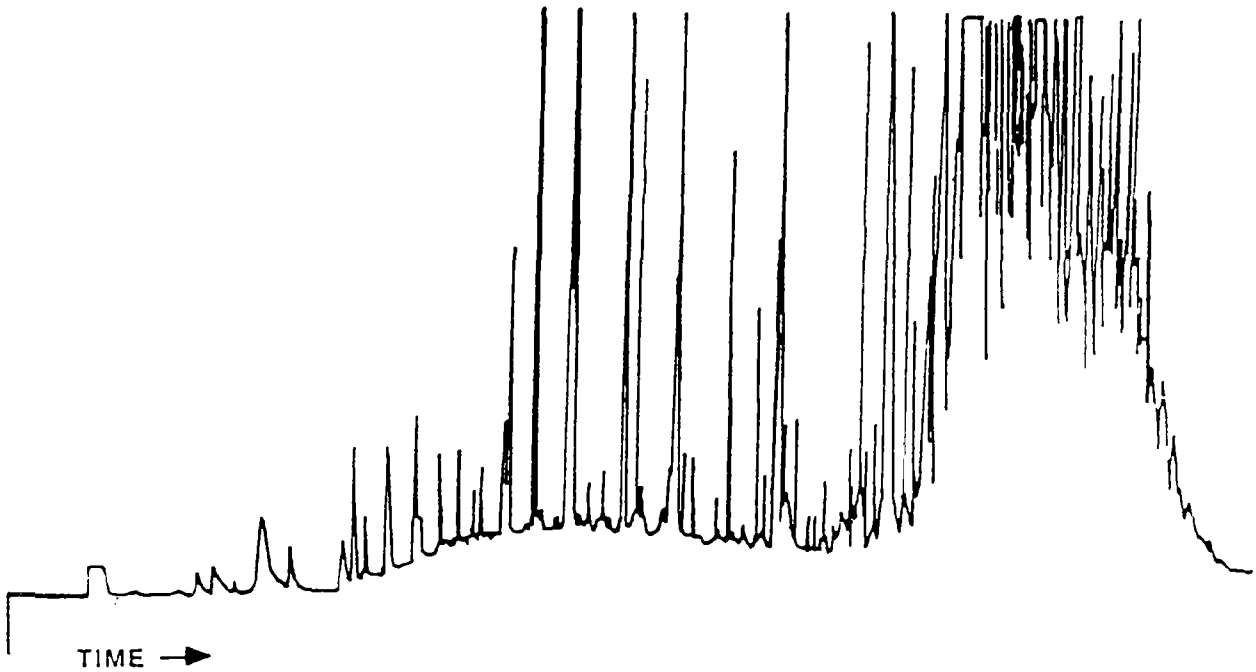


* See Table 7.0 For Peak Identification

FIGURE 16. TYPICAL ECD RESPONSE TO SELECTIVE VOCs



(a). Certified Sampler



(b). Contaminated Sampler

FIGURE 17. EXAMPLE OF HUMID ZERO AIR TEST RESULTS FOR A CLEAN SAMPLER (a) AND A CONTAMINATED SAMPLER (b)

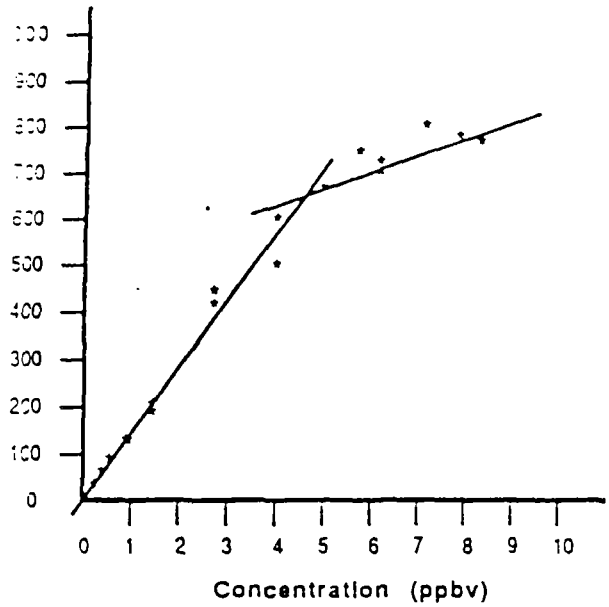


FIGURE 18(a). NONLINEAR RESPONSE OF TETRACHLOROETHYLENE ON THE ECD

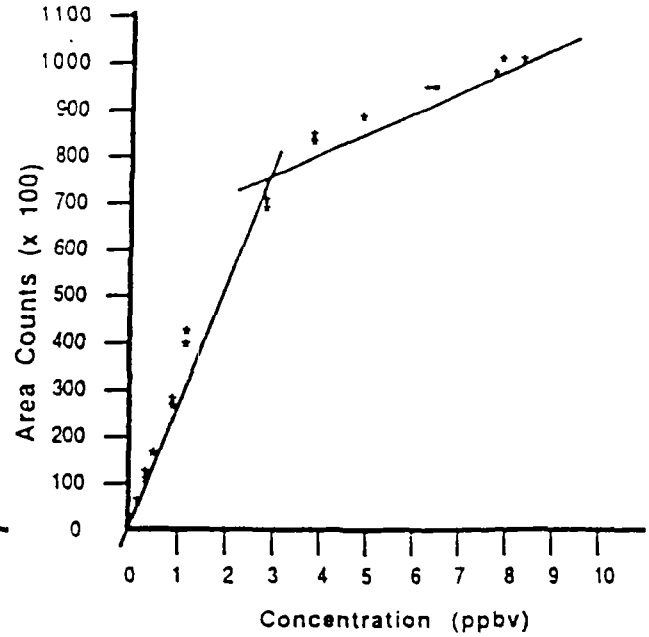


FIGURE 18(b). NONLINEAR RESPONSE OF CARBON TETRACHLORIDE ON THE ECD

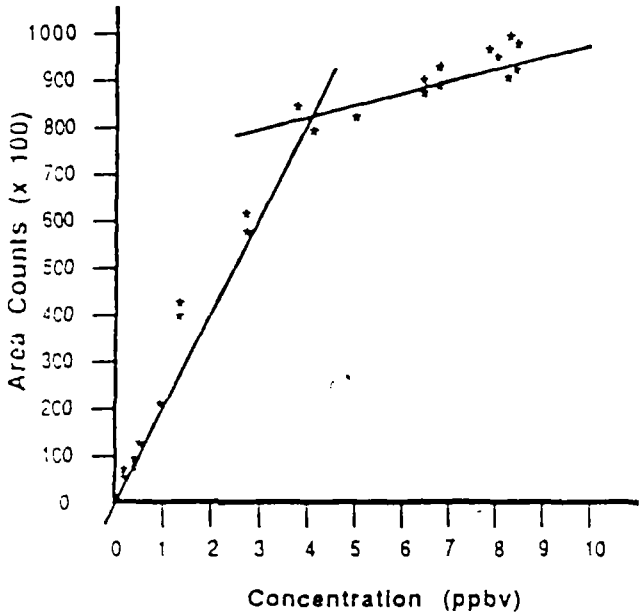


FIGURE 18(c). NONLINEAR RESPONSE OF HEXACHLOROBUTADIENE ON THE ECD

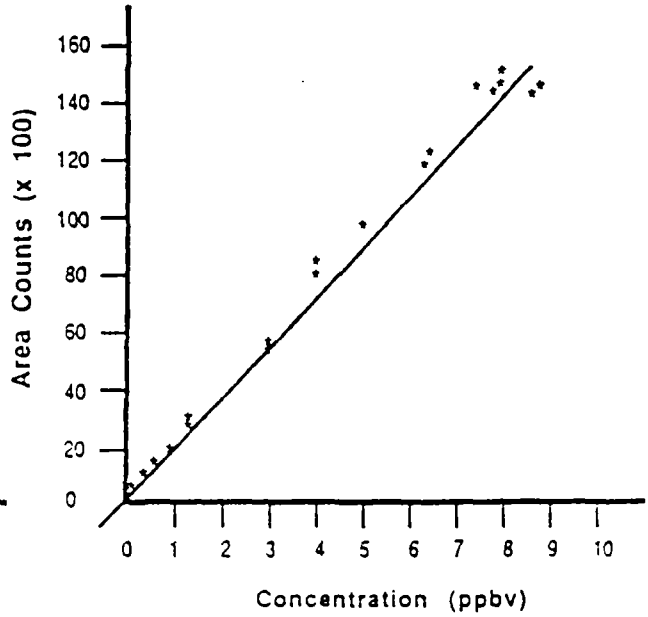


FIGURE 18(d). LINEAR RESPONSE OF CHLOROFORM ON THE ECD

FIGURE 18. RESPONSE OF ECD TO VARIOUS VOCs

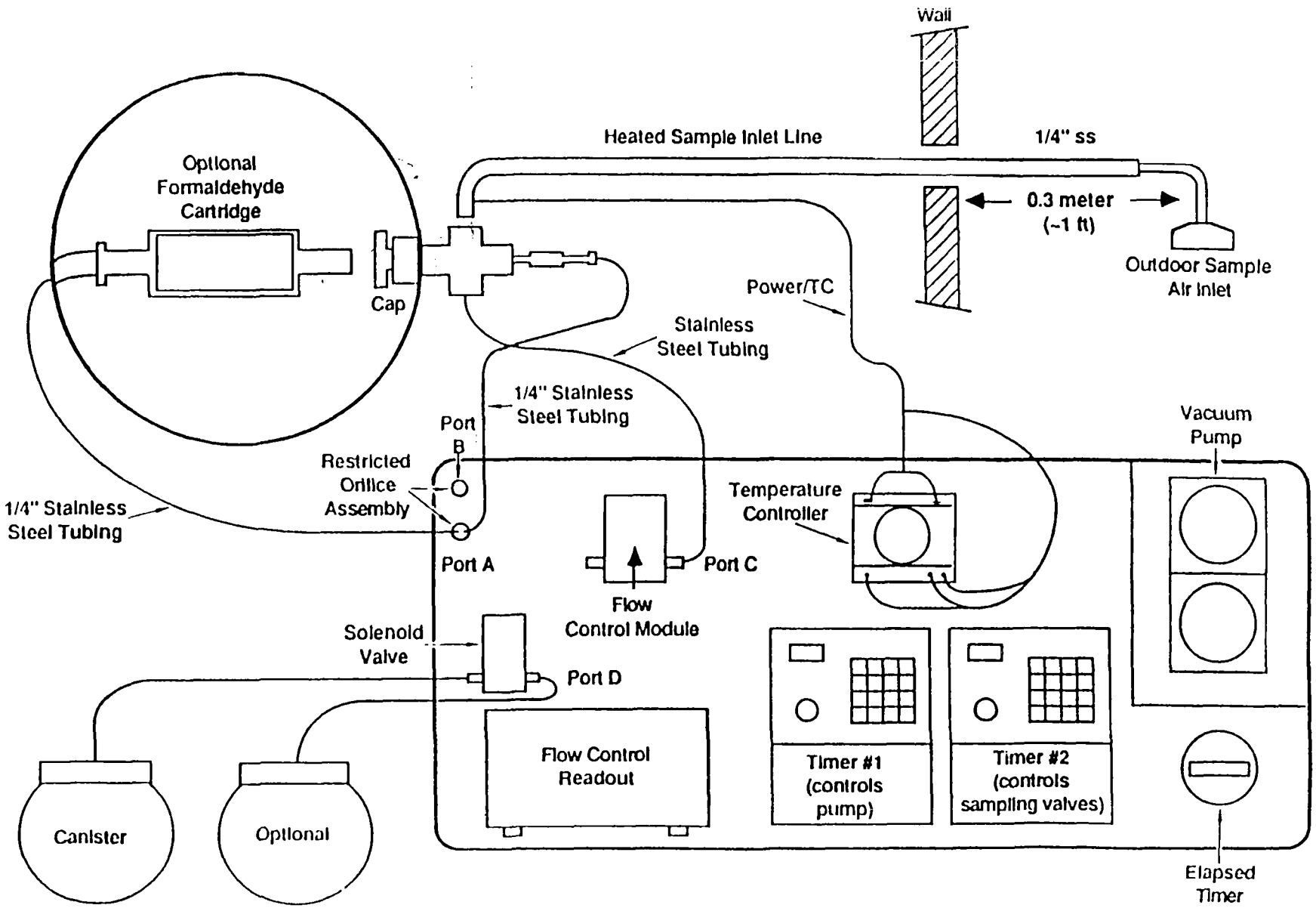


FIGURE 19. U.S. ENVIRONMENTAL PROTECTION AGENCY UTAP, SCHEMATIC OF SAMPLE INLET CONNECTIONS SAMPLER

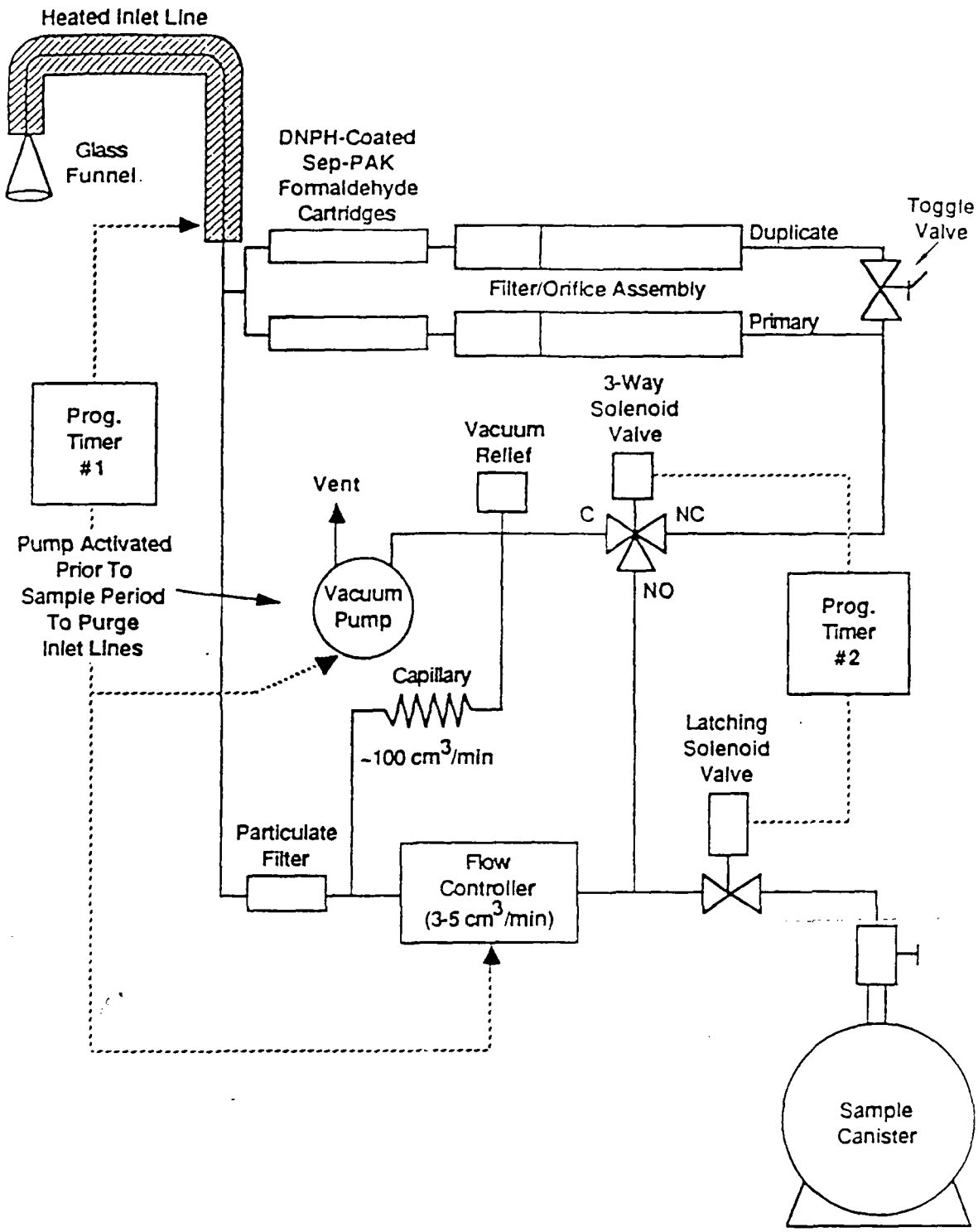


FIGURE 20. U.S. ENVIRONMENTAL PROTECTION AGENCY URBAN TOXIC AIR POLLUTANT PROGRAM (UTAP) WHOLE AIR SAMPLER-INTERNAL SCHEMATIC

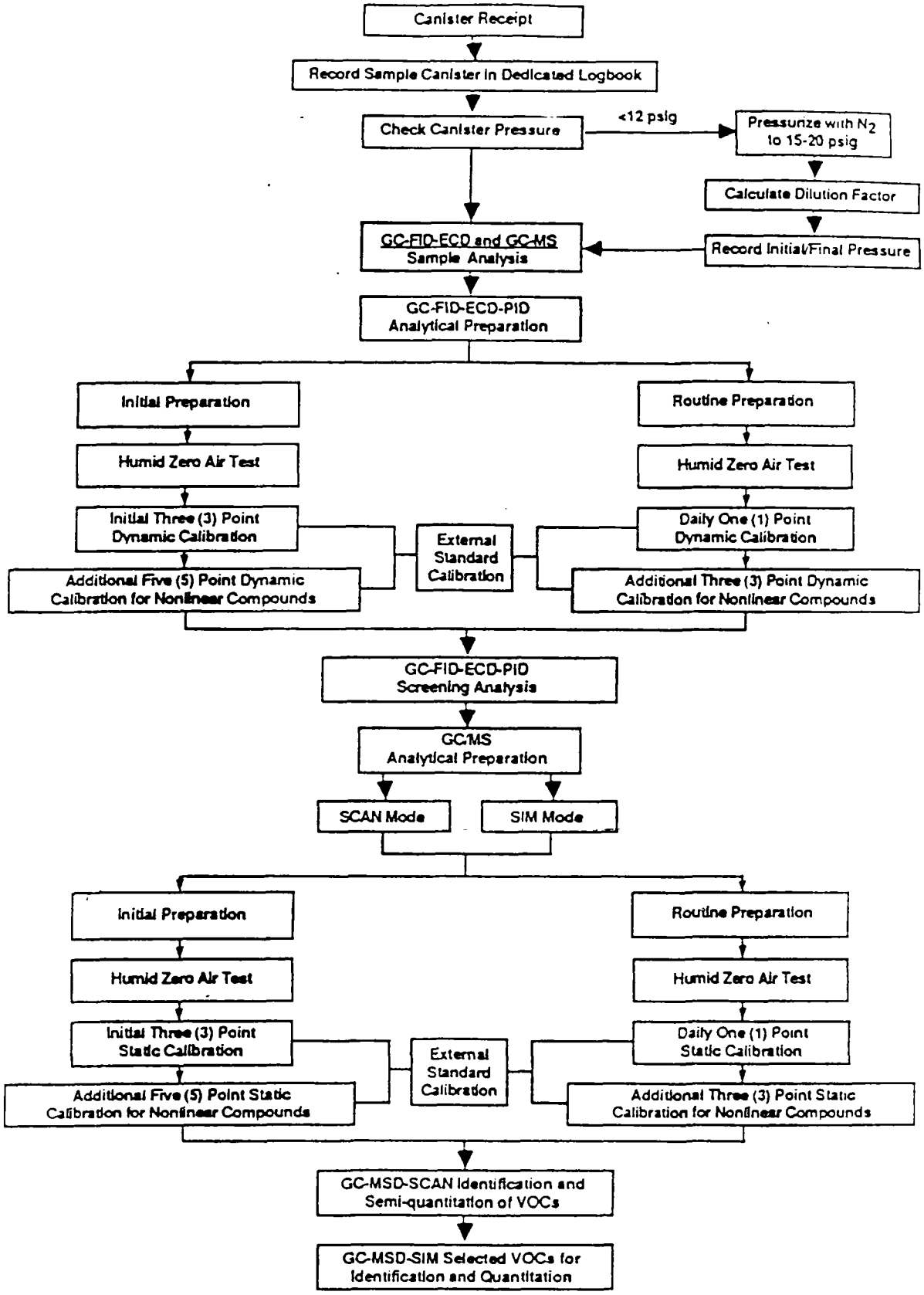


FIGURE 21. FLOWCHART OF ANALYTICAL SYSTEMS PREPARATION.

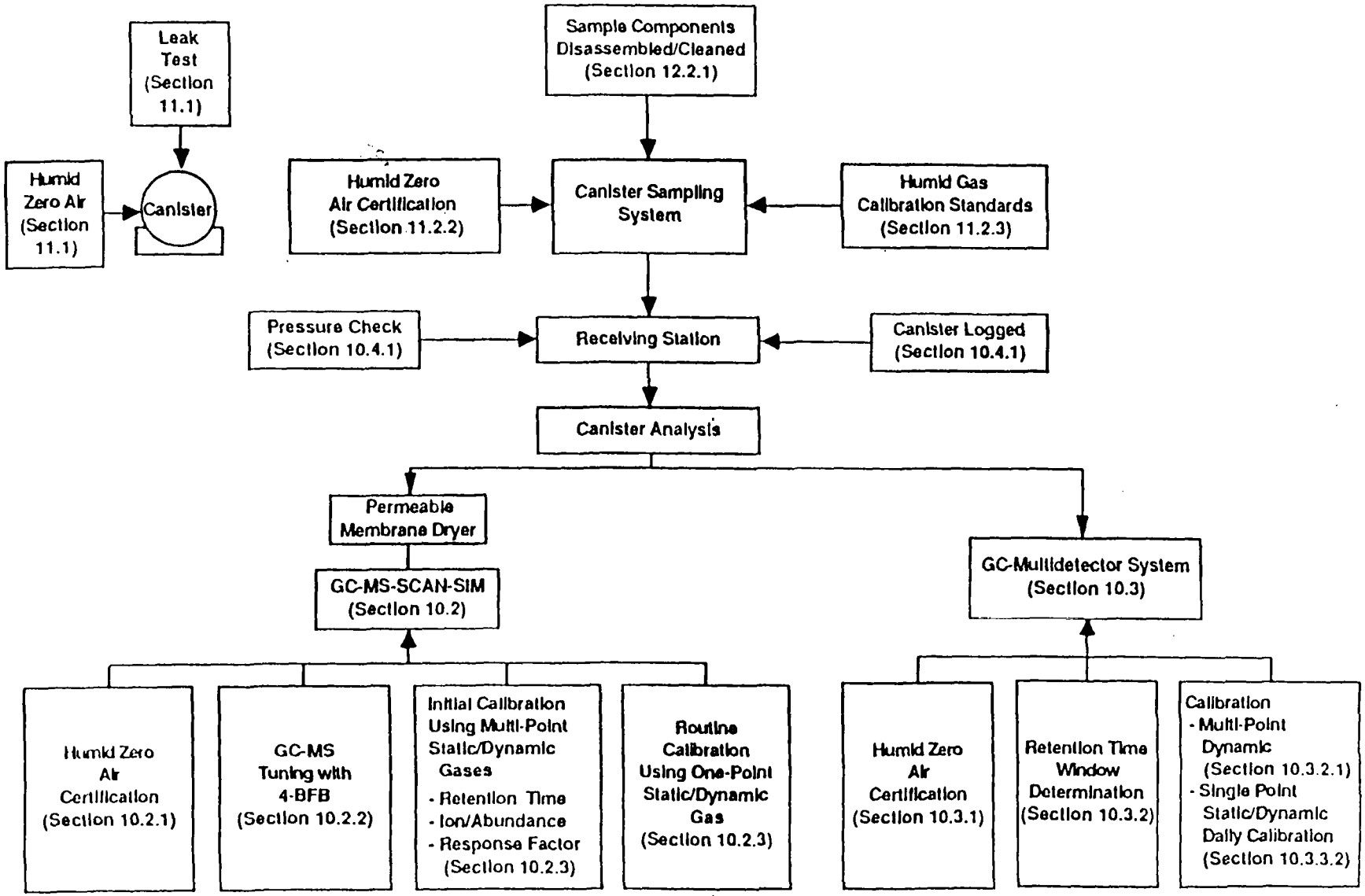


FIGURE 22. SYSTEM QUALITY ASSURANCE / QUALITY CONTROL (QA/QC) ACTIVITIES ASSOCIATED WITH VARIOUS ANALYTICAL SYSTEMS

APPENDIX A
AVAILABILITY OF AUDIT CYLINDERS FROM USEPA TO USEPA PROGRAMS/REGIONAL OFFICES, STATE AND LOCAL AGENCIES AND THEIR CONTRACTORS

1.0 Availability of Audit Cylinders

1.1 The USEPA has available, at no charge, cylinder gas standard of hazardous organic compounds at the ppb level that may be used to audit the performance of ambient air and stationary source measurement systems.

1.2 Each audit cylinder contains 5 to 18 hazardous organic compounds in a balance of N_2 gas. Audit cylinders are available in several concentration ranges. The concentration of each organic compound in the audit cylinder is within the range illustrated in Table A-1.

2.0 Audit Cylinder Certification

2.1 All audit cylinders are periodically analyzed to assure that cylinder concentrations have remained stable.

2.2 All stability analyses include quality control analyses of ppb hazardous organic gas standards prepared by the National Bureau of Standards for USEPA.

3.0 Audit Cylinder Acquisition

3.1 USEPA program/regional offices, state/local agencies, and their contractors may obtain audit cylinders (and an audit gas delivery system, if applicable) for performance audits during:

- o RCRA Hazardous Waste Trial Burns For PHOC's; and
- o Ambient Air Measurement of Toxic Organics.

3.2 The audit cylinders may be acquired by contacting:

Robert L. Lampe
U.S. Environmental Protection Agency
Environmental Monitoring Systems Laboratory
Quality Assurance Division
MD-77B
Research Triangle Park, NC 27711
919-541-4531

TABLE A-1. AVAILABLE USEPA PERFORMANCE AUDIT CYLINDERS

Group I Compounds	Group II Compounds	Group III Compounds
Carbon tetrachloride	Trichloroethylene	Pyridine (Pyridine in Group III cylinders but certified analysis not available)
Chloroform	1,2-dichloroethane	Vinylidene chloride
Perchloroethylene	1,2-dibromoethane	1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113)
Vinyl chloride	Acetonitrile	1,2-dichloro-1,1,2,2-tetrafluoroethane (Freon-114)
Benzene	Trichlorofluoromethane (Freon-11)	Acetone
	Dichlorodifluoromethane (Freon-12)	1-4 Dioxane
	Bromomethane	Toluene
	Methyl ethyl ketone	Chlorobenzene
	1,1,1-trichloroethane	

Group I Ranges	Group II Ranges	Group III Ranges
7 to 90 ppb	7 to 90 ppb	7 to 90 ppb
90 to 430 ppb	90 to 430 ppb	90 to 430 ppb
430 to 10,000 ppb		

Group IV	Group V	
Acrylonitrile	Carbon tetrachloride	Methylene chloride
1,3-butadiene	Chloroform	Trichlorofluoromethane (Freon-11)
Ethylene oxide	Perchloroethylene	Bromomethane
Methylene chloride	Vinyl chloride	Toluene
Propylene oxide	Benzene	Chlorobenzene
o-xylene	Trichloroethylene	1,3-Butadiene
	1,2-dichloroethane	o-xylene
	1,2-dibromoethane	Ethyl benzene
	1,1,1-trichloroehtane	1,2-dichloropropane

Group IV Ranges	Group V Ranges
7 to 90 ppb	1 to 40 ppb
430 to 10,000 ppb	

APPENDIX B

OPERATING PROCEDURES FOR A PORTABLE GAS CHROMATOGRAPH (GC) EQUIPPED WITH A PHOTOIONIZATION DETECTOR (PID)

1. Scope

This procedure is intended to screen ambient air environments for volatile organic compounds. Screening is accomplished by collection of VOC samples within an area and analysis onsite using a portable gas chromatograph/integrator (Photovac Models 10S10, 10S50, or equivalent). This procedure is not intended to yield quantitative or definite qualitative information regarding the substances detected. Rather, it provides a chromatographic "profile" of the occurence and intensity of unknown volatile compounds which assists in placement of fixed-site samplers.

2. Applicable Documents

2.1 ASTM Standards

E260 - Recommended Practice for General Gas Chromatography Procedures

E355 - Practice for Gas Chromatography Terms and Relationships

2.2 Other Documents

Portable Instruments User's Manual for Monitoring VOC Sources, EPA-34011-86-015, U.S. Environmental Protection Agency, Washington, DC, June, 1986.

3. Summary of Method

3.1 An air sample is extracted directly from ambient air and analyzed on-site by a portable GC.

3.2 Analysis is accomplished by drawing an accurate volume of ambient air through a sampling port and into a concentrator, then the sample air is transported by carrier gas onto a packed column and into a PID, resulting in response peak(s). Retention times are compared with those in a standard chromatogram to predict the probable identity of the sample components.

4. Significance

4.1 VOCs are emitted into the atmosphere from a variety of sources including petroleum refineries, synthetic organic chemical plants,

natural gas processing plants, and automobile exhaust. Many of these VOC emissions are acutely toxic; therefore, their determination in ambient air is necessary to assess human health impacts.

4.2 Conventional methods for VOC determination use solid sorbent and canister sampling techniques.

4.3 Collection of ambient air samples in canisters provides (1) convenient integration of ambient samples over a specific time period, (e.g., 24 hours); (2) remote sampling and central analysis; (3) ease of storing and shipping samples, if necessary; (4) unattended sample collection; (5) analysis of samples from multiple sites with one analytical system; and (6) collection of sufficient sample volume to allow assessment of measurement precision and/or analysis of samples by several analytical systems.

4.4 The use of portable GC equipped with multi-detectors have assisted air toxics programs by using the portable GC as a "screening tool" to determine "hot spots," potential interferences, and semi-quantitation of VOCs/SVOCs, prior to locating more traditional fixed-site samplers.

5. Definitions

Definitions used in this document and in any user-prepared Standard Operating Procedures (SOPs) should be consistent with ASTM Methods D1356 and E355. Abbreviations and symbols pertinent to this method are defined at point of use.

6. Interferences

6.1 The most significant interferences result from extreme differences in limits of detection (LOD) among the target VOCs (Table B-1). Limitations in resolution associated with ambient temperature chromatography and the relatively large number of chemicals result in coelution of many of the target components. Coelution of compounds with significantly different PID sensitivities will mask compounds with more modest sensitivities. This will be most dramatic in interferences from benzene and toluene, two common ambient contaminants.

6.2 A typical chromatogram and peak assignments of a standard mixture of target VOCs (under the prescribed analytical conditions of this method) are illustrated in Figure B-1. Samples which contain a highly complex mixture of components and/or interfering levels of benzene and toluene are analyzed on a second, longer chromatographic column. The same liquid phase in the primary column is contained in the alternate column but at a higher percent loading.

6.3 Recent designs in commercially available GCs (Table B-2) have preconcentrator capabilities for sampling lower concentrations of VOCs, pre-column detection with back-flush capability for shorter analytical time, constant column temperature for method precision and accuracy and multidetector (PID, ECD, and FID) capability for versatility. Many of these newer features address the weaknesses and interferences mentioned above.

7. Apparatus

7.1 Gas chromatograph. A GC (Photovac Inc., 739 B Parks Ave, Huntington, NY, 11743, Model 10S10 or 10S50), or equivalent used for surveying ambient air environments (which could employ a multidetector) for sensing numerous VOCs compounds eluting from a packed column at ambient temperatures. This particular portable GC procedure is written employing the photoionization detector as its major sensing device, as part of the Photovac Model 10S10 portable GC survey tool. Chromatograms are developed on a column of 3% SP-2100 on 100/120 Supelcoport (0.66 m x 3.2 mm I.D.) with a flow of 30 cm^3/min air.

7.2 GC accessories. In addition to the basic gas chromatograph, several other pieces of equipment are required to execute the survey sampling. Those include gas-tight syringes for standard injection, alternate carrier gas supplies, high pressure connections for filling the internal carrier gas reservoir, and if the Model 10S10 is used, a recording integrator (Hewlett Packard, Avondale, PA, Model 3390A, or equivalent).

8. Reagents and Materials

8.1 Carrier gas. "Zero" air [<0.1 ppm total hydrocarbon (THC)] is used as the carrier gas. This gas is conveniently contained in 0.84 m3 (30 ft3) aluminum cylinders. Carrier gas of poorer quality

may result in spurious peaks in sample chromatograms. A Brooks, Type 1355-00F1AAA rotameter (or equivalent) with an R-215-AAA tube and glass float is used to set column flow.

8.2 System performance mixture. A mixture of three target compounds (e.g., benzene, trichloroethylene, and styrene) in nitrogen is used for monitoring instrument performance. The approximate concentration for each of the compounds in this mixture is approximately to be 10 parts per billion (ppb). This mixture is manufactured in small, disposable gas cylinders [at 275 kPa (40 psi)] from Scott Specialty Gases, or equivalent.

8.3 Reagent grade nitrogen gas. A small disposable cylinder of high purity nitrogen gas is used for blank injections.

8.4 Sampling syringes. Gas-tight syringes, without attached shut-off valves (Hamilton Model 1002LT), or equivalent, are used to introduce accurate sample volumes into the high pressure injectors on the portable gas chromatograph. Gas syringes with shut-off valves are not recommended because of memory problems associated with the valves. For samples suspected of containing high concentrations of volatile compounds, disposable glass syringes (e.g, Glaspak) with stainless steel/Teflon hub needles are used.

8.5 High pressure filler. An adapter (Photovac SA101) for filling the internal carrier gas reservoir on the portable GC is used to deliver "zero" air.

9. Procedure

9.1 Instrument Setup

9.1.1 The portable gas chromatograph must be prepared prior to use in the ambient survey sampling. The pre-sampling activities consist of filling the internal carrier gas cylinder, charging the internal power supply, adjusting individual column carrier gas flows, and stabilizing the photoionization detector.

9.1.2 The internal reservoir is filled with "zero" air. The internal 12V, 6AH lead/acid battery can be recharged to provide up to eight hours of operation. A battery which is discharged will automatically cause the power to the instrument to be shut down and will require an overnight charge. During AC operation, the batteries will automatically be trickle-charged or in a standby mode.

9.1.3 The portable GC should be operated(using the internal battery power supply) at least forty minutes prior to collection of the first sample to insure that the photoionzation detector has stabilized. Upon arriving at the area to be sampled, the unit should be connected to AC power, if available.

9.2 Sample Collection

9.2.1 After the portable gas chromatograph is located and connected to 110V AC, the carrier gas flows must be adjusted. Flows to the 1.22 meter, 5% SE-30 and 0.66 meter, 3% SP2100 columns are adjusted with needle valves. Flows of 60 cm^3/min (5% SE-30) and 30 cm^3/min (3% SP2100) are adjusted by means of a calibrated rotameter. Switching between the two columns is accomplished by turning the valve located beneath the electronic module. During long periods of inactivity, the flows to both columns should be reduced to conserve pressure in the internal carrier gas supply. The baseline on the recorder/integrator is set to 20% full scale.

9.2.2 Prior to analysis of actual samples, an injection of the performance evaluation mixture must be made to verify chromatographic and detector performance. This is accomplished by withdrawing 1.0 mL samples of this mixture from the calibration cylinder and injecting it onto the 3% SP2100 column. The next sample analyzed should be a blank, consisting of reagent grade nitrogen.

9.2.3 Ambient air samples are injected onto the 3% SP2100 column. The chromatogram is developed for 15 minutes. Samples which produce particularly complex chromatograms, especially for early eluting components, are reinjected on the 5% SE-30 column. [Note: In no instance should a syringe which has been used for the injection of the calibrant/system performance mixture be used for the acquisition and collection of samples, or vice versa.]

9.2.4 Samples have generally been collected from the ambient air at sites which are near suspected sources of VOCs and SVOCs and compared with those which are not. Typically, selection of ambient sample locations is based on the presence of chemical odors. Samples collected in areas without detectable odors have not shown significant PID responses. Therefore, sampling efforts should be initially concentrated on "suspect" environments (i.e., those which have appreciable odors). The objective of the sampling is to locate sources of the target compounds. Ultimately, samples should be collected throughout the entire location, but with particular attention given to areas of high or frequent occupation.

9.3 Sample Analysis

9.3.1 Qualitative analysis. Positive identification of sample components is not the objective of this "screening" procedure. Visual comparison of retention times to those in a standard chromatogram (Figure B-1) are used only to predict the probable sample component types.

9.3.2 Estimation of levels. As with qualitative analysis, estimates of component concentrations are extremely tentative and are based on instrument responses to the calibrant species (e.g., benzene, trichloroethylene, styrene), the proposed component identification, and the difference in response between sample component and calibrant. For

purposes of locating chemical emission sources, roughly estimated concentrations and suspected compound types are considered sufficient.

10. Performance Criteria and Quality Assurance

Required quality assurance measures and guidance concerning performance criteria that should be achieved within each laboratory are summarized and provided in the following section.

10.1 Standard Operating Procedures (SOPs)

10.1.1 SOPs should be generated by the users to describe and document the following activities in their laboratory: (1) assembly, calibration, leak check, and operation of the specific portable GC sampling system and equipment used; (2) preparation, storage, shipment, and handling of the portable GC sampler; (3) purchase, certification, and transport of standard references materials; and (4) all aspects of data recording and processing, including lists of computer hardware and software used.

10.1.2 Specific stepwise instructions should be provided in the SOPs and should be readily available to and understood by the personnel conducting the survey work.

10.2 Quality Assurance Program

10.2.1 Reagent and materials control. The carrier gas employed with the portable GC is "zero air" containing less than 0.1 ppm VOCs. System performance mixtures are certified standard mixtures purchased from Scott Specialty Gases, or equivalent.

10.2.2 Sampling protocol and chain of custody. Sampling protocol sheets must be completed for each sample. Specifics of the sample with regard to sampling location, sample volume, analysis conditions, and supporting calibration and visual inspection information are detailed by these documents. An example form is exhibited in Table B-3.

10.2.3 Blanks, Duplicates, and System Performance Samples

10.2.3.1 Blanks and duplicates. Ten percent of all injections made to the portable GC are blanks, where the blank is reagent grade nitrogen gas. This is the second injection in each sampling location. An additional 10% of all injections made are duplicate injections. This will enhance the probability that the chromatogram of a sample reflects only the composition of that sample and not any previous injection. Blank injections showing a significant amount of contaminants will be cause for remedial action.

10.2.3.2 System performance mixture. An injection of the system performance mixture will be made at the beginning of a visit to a particular sampling location (i.e., the first injection). The range of acceptable chromatographic system performance criteria and detector response is shown in Table B-4. These criteria are selected with regard to the intended application of this protocol and the limited availability of standard mixtures in this area. Corrective action should be taken with the column or PID before sample injections are made if the performance is deemed out-of-range. Under this regimen of blanks and system performance samples, approximately eight samples can be collected and analyzed in a three hour visit to each sampling location.

10.3 Method Precision and Accuracy

The purpose of the analytical approach outlined in this method is to provide presumptive information regarding the presence of selected VOCs and SVOCs emissions. In this context, precision and accuracy are to be determined. However, quality assurance criteria are described in Section 10.2 which insure the samples collected represent the ambient environment.

10.4 Range and Limits of Detection

The range and limits of detection of this method are highly compound dependent due to large differences in response of the portable GC's photoionization detector to the various target compounds. Aromatic compounds and olefinic halogenated compounds will be detected at lower levels than the halomethanes or aliphatic hydrocarbons. The concentration range of application of this method is approximately two orders of magnitude.

TABLE B-1

ESTIMATED LIMITS OF DETECTION (LOD) FOR SELECTED VOCs
BASED ON 1 uL SAMPLE VOLUME

Compound	LOD (ng)	LOD (ppb)
Chloroform[a]	2	450
1,1.1-Trichloroethane[a]	2	450
Carbon tetrachloride[a]	2	450
Benzene	.006	2
1,2-Dichloroethane[b]	.05	14
Trichloroethylene[b]	.05	14
Tetrachloroethylene[b]	.05	14
1,2-Dibromoethane	.02	2
p-Xylene[c]	.02	4
m-Xylene[c]	.02	4
o-Xylene[d]	.01	3
Styrene[d]	.01	3

[a]Chloroform, 1,1,1-Trichloroethane, and Carbon tetrachloride coelute on 0.66 m 3% SP2100.
[b]1,2-Dichloroethane, Tricholroethylene, and Tetrachloroethylene coelute on 0.66 m 3% SP2100.
[c]p-Xylene and m-Xylene coelute on 0.66 m 3% SP2100.
[d]Styrene and o-Xylene coelute on 0.66 m 3% SP2100.

TABLE B-2

COMMERCIALLY AVAILABLE PORTABLE VOC DETECTION INSTRUMENTS

Monitor	Detection principle	Range, ppm	Sensitivity	Response time, s	Accessories	Calibration Techniques	Weaknesses	Service Rate	Lack of Response	Cost, $	Start Rate
550,551 555,580 (AID, Inc.	PID, FID	0-200, 0-2000, 0-10,000	0.1 ppm at 0-200 ppm	<5		o Bag Sampling	o Umbilical cord too short o Digital readout hard to read o Flame out frequently	8 hrs		4,300	1.5
OVA 108, 128 Century Systems, inc. (Foxboro)	FID	0-10, 0-100, 0-1000, 0-10,000, 0-100,000	0.2 ppm (Model 128) 0.5 ppm (Model 108)	2 2	o Thermal Desorbers available o Optional GC available	o Hand Space o Direct Injection o Bag Samp.	o Battery failure o Sample line kinks o Compounds containing O_2/N give low response o Neg. reso. to CO/CO_2	8 hrs		5,300	
PI-101 (HNu Systems, Inc)	PID	1 1-20 1-200 1-2000	0.1 ppm Low molecular weights aromatics	<5	o Three lamps available o 9.5 (aromatics) o 10.2 (2-4 compounds) o 11.7 (halocarbons)	o External Gas Cyl. o Bag Samp.	o Three lamps - may miss something	10 hrs	o Cl hydrocarbons o CH_4	4,955	3.5
TLV Sniffer (Bacharach)	Catalytic combustion	0-500 0-5000 0-50,000	2.0 ppm	5		o Bag Samp. o Head Space				500	
Ecolyzer 400 (Energetics Science)	Catalytic combustion	0-100% LFL	1% LFL	15		o Bag Samp.	o Changes in gas temp/humidity affects response				
Miran 1A (Foxboro)	IR	ppm to %	1 ppm	1,4,10 and 40						9,550	
Miran 1B (Foxboro)	IR	ppm to %								12,500	
Scentor (Sentex)	GC/EC, Argon Ionization PID		0.01 ppb Cl organics	2	Preconcentrator Thermal Desorption GC Columns Auto Cal. from Integral Gas Cylinder	o Internal gas cyl. o Preconcentrator o GC Column				12,950	
Photovac Standard Automatic Computer Auto Comp. Communication	PID (UV Light)	0	0.1 ppb benzene with signal-to-noise ratio 4:1, Good for aromatics	2	o Dual Column o Manual/Auto Injection o Column Cond. o Pre-flush o Auto Dial Modem o Programmable		o Column operates at ambient temp. o STD in lab, then to field at diff. temp o Can't inject liquid samp. o Light fractions interfere		o H_2O o O_2	6,395 8,395 10,500 10,395 12,395	
Photovac TIP	PID	0-2000 ppm	0.05 ppm Benzene	3							

TABLE B-3

PORTABLE GAS CHROMATOGRAPH
FIELD DATA SHEET

DATE: _____ LOCATION: _____ TIME: _____

CHROMATOGRAPHIC CONDITIONS: _____

COLUMN 1: COLUMN TYPE:

I.D. (mm):_____ LENGTH (mm):_____ FLOW (mL/min):_____

COLUMN 2: COLUMN TYPE:

I.D. (mm):_____ LENGTH (mm):_____ FLOW (mL/min):_____

INJ. NO.	INJ. VOL.	COLUMN NO.	SETTING	LOCATION

SITE PLAN (indicate sampling locations):

DATE SIGNATURE

TABLE B-4

SYSTEM PERFORMANCE CRITERIA FOR PORTABLE GC[a]

Criterion	Test Compound	Acceptable Range	Suggested Corrective Action
PID Response	Trichloroethylene	$\geq 10^8$ uV-sec/ng	Re-tune or replace lamp
Elution Time	Styrene	2.65 $\pm$ 0.15 min	Inspect for leaks, adjust carrier flow
Resolution[b]	Benzene/Trichloro-ethylene	$\geq$ 1.4	Replace column

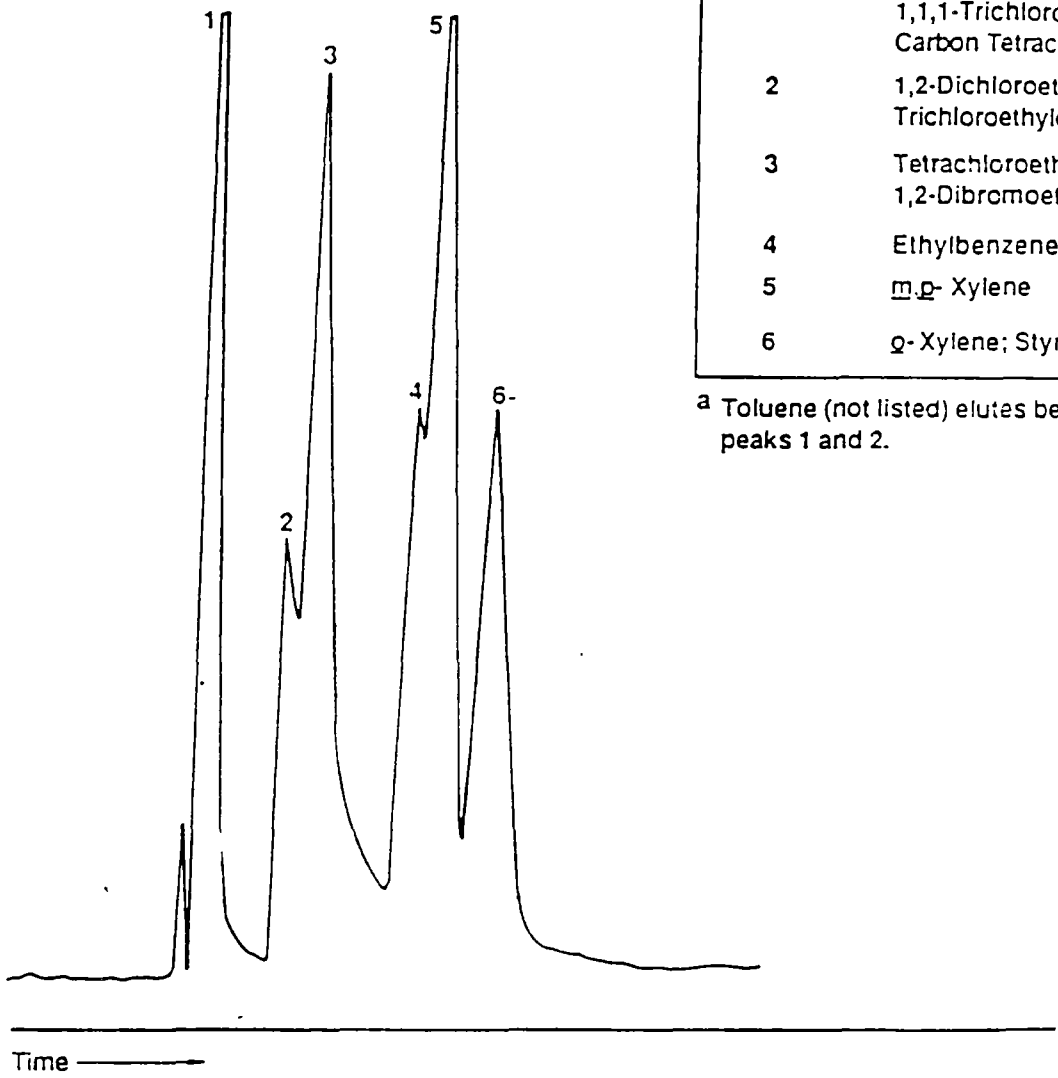
[a]Based on analysis of a vapor mixture of benzene, styrene, and trichloroethylene.
[b]Define by: $R + = 2d/(W_1+W_2)$; where d = distance between the peaks and W = peak width at base.

TABLE B-5

ESTIMATED LIMITS OF DETECTION (LOD) FOR SELECTED VOCs

Compound	LOD (ng)	LOD (ppb)
Chloroform[a]	2	450
1,1,1-Trichloroethane[a]	2	450
Carbon tetrachloride[a]	2	450
Benzene	.006	2
1,2-Dichloroethane[b]	.05	14
Trichloroethylene[b]	.05	14
Tetrachloroethylene[b]	.05	14
1,2-Dibromoethane	.02	2
p-Xylene[c]	.02	4
m-Xylene[c]	.02	4
o-Xylene[d]	.01	3
Styrene[d]	.01	3

[a]Chloroform, 1,1,1-Trichloroethane, and Carbon tetrachloride coelute on 0.66 m 3% SP2100.
[b]1,2-Dichloroethane, Trichloroethylene, and Tetrachloroethylene coelute on 0.66 m 3% SP2100.
[c]p-Xylene and m-Xylene coelute on 0.66 m 3% SP2100.
[d]Styrene and o-Xylene coelute on 0.66 m 3% SP2100.



Peak Assignments For Standard Mixture

Peak No.	Compound(s) [a]
1	Benzene; Chloroform; 1,1,1-Trichloroethane; Carbon Tetrachloride
2	1,2-Dichloroethane; Trichloroethylene
3	Tetrachloroethylene; 1,2-Dibromoethane
4	Ethylbenzene
5	m,p- Xylene
6	o- Xylene; Styrene

[a] Toluene (not listed) elutes between peaks 1 and 2.

FIGURE B-1. TYPICAL CHROMATOGRAM OF VOCs DETERMINED BY A PORTABLE GC

APPENDIX C

INSTALLATION AND OPERATION PROCEDURES FOR U.S. ENVIRONMENTAL PROTECTION AGENCY'S URBAN TOXIC AIR POLLUTANT PROGRAM SAMPLER

1. Sampler Installation

1.1 The sampler must be operated indoors with the temperature between 20-32°C (68 to 90°F). The sampler case should be located conveniently on a table, shelf, or other flat surface. Access to a source of 115 vac line power (500 watts min) is also required. The pump is removed from the sampler case and located remotely from the sampler (connected with a 1/4 inch O.D. extension tubing and a suitable electrical extension cord).

1.2 Electrical Connections (Figure C-1)

1.2.1 The sampler cover is removed. The sampler is not plugged into the 115 vac power until all other electrical connections are completed.

1.2.2 The pump is plugged into its power connector (if not already connected) and the battery connectors are snapped onto the battery packs on the covers of both timers.

1.2.3 The sampler power plug is inserted into a 115 volts ac line grounded receptacle. The sampler must be grounded for operator safety. The electrical wires are routed and tied so they remain out of the way.

1.3 Pneumatic Connections

1.3.1 The length of 1/16 inch O.D. stainless steel tubing is connected from port A of the sampler (on the right side of the flow controller module) to the air inlet line.

1.3.2 The pump is connected to the sampler with 1/4 inch O.D. plastic tubing. This tubing may be up to 7 meters (20 feet) long. A short length of tubing is installed to reduce pump noise. All tubing is conveniently routed and, if necessary, tied in place.

2. Sampler Preparation

2.1 Canister

2.1.1 The sample canister is installed no more than 2 days before the scheduled sampling day.

2.1.2 With timer #1 ON, the flow controller is allowed to warm up for at least 15 minutes, longer if possible.

2.1.3 An evacuated canister is connected to one of the short lengths of 1/8 inch O.D. stainless steel tubing from port B (solenoid valve) of the sampler. The canister valve is left closed. The Swagelok fitting on the canister must not be cross-threaded. The connection is tightened snugly with a wrench.

2.1.4 The end of the other length of stainless steel tubing from port B (solenoid valve) is connected with a Swagelok plug.

2.1.5 If duplicate canisters are to be sampled, the plug is removed from the second 1/8 inch O.D. stainless steel tubing from port B (solenoid valve) and the second canister is connected. The canister valve is left closed.

2.1.6 The ON button of timer #2 is pressed. The flow through the flow controller should be stopped by this action.

2.1.7 The flow controller switch is turned to "READ" and the zero flow reading is obtained. If this reading is not stable, wait until the reading is stabilized.

2.1.8 The flow controller switch is turned to "SET" and the flow setting is adjusted to the algebraic SUM of the most recent entry on Table C-1 and the zero reading obtained in step 2.1.7 (If the zero reading is negative, SUBTRACT the zero reading from the Table C-1 value). Be sure to use the correct Table C-1 flow value for one or two canisters, as appropriate. [Note: If the analysis laboratory determines that the canister sample pressure is too low or too high, a new flow setting or settings will be issued for the sampler. The new flow setting should be recorded in Table B-1 and used until superseded by new settings.]

2.1.9 Timer #2 is turned OFF to again start the flow through the flow controller. With the pump (timer #1) ON and the sampling valve (timer #2) OFF, the flow controller is turned to "READ" and the flow is verified to be the same as the flow setting made in step 2.1.8. If not, the flow setting is rechecked in step 2.1.8 and the flow setting is readjusted if necessary.

2.1.10 The OFF button of timer #1 is pressed to stop the pump.

2.1.11 The canister valve(s) are fully opened.

2.2 Timers

2.2.1 Timer #2 is set to turn ON at the scheduled ON time for the sample period, and OFF at the scheduled OFF time. (See the subsequent section on setting the timers.)
Normal ON time: 12:00 AM on the scheduled sampling day.
Normal OFF time: 11:59 PM on the scheduled sampling day.
(The OFF time is 11:59 PM instead of 12:00 AM so that the day number for the OFF time is the same as the day number for the ON time.) Be sure to set the correct day number.

2.2.2 Timer #1 is set to turn ON six (6) hours before the beginning of the scheduled sample period and OFF at the scheduled OFF time for the sample period (same OFF time as for timer #2). (See the subsequent section on setting the timers.)
Normal ON time: 06:00 PM on the day prior to the scheduled sampling day.
Normal OFF time: 11:59 PM on the scheduled sampling day.
[Note: The timers are wired so that the pump will be on whenever either timer is on. Thus the pump will run if timer #2 is ON even if timer #1 is OFF.]

2.2.3 The elapsed minutes meter is set to 0.

2.3 Sampler Check

2.3.1 The following must be verified before leaving the sampling site:

(1) Canister(s) is (are) connected properly and the unused connection is capped if only one canister is used.
(2) Canister valve(s) is (are) opened.
(3) Both timers are programmed correctly for the scheduled sample period.
(4) Both timers are set to "AUTO".
(5) Both timers are initially OFF.
(6) Both timers are set to the correct current time of day and day number.
(7) Elasped time meter is set to 0.

2.4 Sampler Recovery (Post Sampling)

2.4.1 The valve on the canister is closed.

2.4.2 The canister is disconnected from the sampler, the sample data sheet is completed, and the canister is prepared for shipment to the analytical laboratory.

2.4.3 If two canisters were sampled, step 2.4.2 is repeated for the other canister.

3. Timer Setting

Since the timers are 7-day timers, the days of the week are numbered from 1 to 7. The assignment of day numbers to days of the week is indicated on the timer keypad: 1 = Sunday, 2 = Monday, 3 = Tuesday, 4 = Wednesday, 5 = Thursday, 6 = Friday, and 7 = Saturday. This programming is quite simple, but some timers may malfunction or operate erratically if not programmed exactly right. To assure correct operation, the timers should be reset and completely reprogrammed "from scratch" for each sample. The correct current time of day is re-entered to re-program the timer. Any program in the timer's memory is erased by resetting the timer (pressing the reset button). The timer is set by the following:

(1) pressing the reset button,
(2) entering the correct day number and time of day,
(3) entering the ON and OFF times for the sample period, and
(4) verifying that the ON and OFF time settings are correct.

3.1 Timer Reset

The timer reset button is pressed, which is recessed in a small hole located just above the LED (light emitting diode) indicator light. A small object that will fit through the hole, such as a pencil, match, or pen is used to press the timer. After reset, the timer display should show |1| |10:00|. [Note: The timers may operate erratically when the batteries are discharged, which happens when the sampler is unplugged or without power for several hours. When the sampler is again powered up, several hours may be required to recharge the batteries. To avoid discharging the batteries, the battery pack should be disconnected from the timer when the sampler is unplugged.]

3.2 Date and Time Entry

The selector switch is turned to SET and the number button corresponding to the day number is pressed. (For example, a "2" is pressed for Monday.) The current time of day is entered. (For example, if the time is 9:00 AM, 900 is pressed.) AM or PM is pressed as applicable. (Display should show |2| |'9:00| for 9:00 AM Monday.) [Note: ' indicates AM and indicates PM.] The CLOCK button is pressed. (Display should show |-| |--:--|) If an error is made, |E| |EE:EE| is shown on the display. The CLEAR button is pressed and the above steps are repeated. The selector switch is turned to AUTO or MAN to verify correct time setting.

3.3 ON and OFF Entry

The selector switch is turned to SET. The ON and OFF program is entered in the following order: day, number, time, AM or PM, ON or OFF. (Example: To turn ON at 12:00 AM on day 5 (Thursday); 5, 1200, AM, ON is entered). (Example: To turn OFF at 11:59 PM on day 5 (Thursday), 5, 11:59, PM, OFF is entered.) If the display indicates an error (|E| |EE:EE|), the timer is reset. The selector switch is turned to AUTO.

3.4 ON and OFF Verification

3.4.1 The selector switch is turned to REVIEW. The number of the scheduled sample day is pressed. ON is pressed. The display should show the time of the beginning of the sample period (for example, |5| |'12:00|). [' indicates AM.] ON is pressed again. The display should show |5| |--:--|, indicating no other ON times are programmed.

3.4.2 OFF is pressed. The display should show the time of the end of the sample period, (for example, |5| |, 11:59|). PM is indicated by the "," mark before the time. OFF is pressed again. The display should show |5| |--:--|, indicating

no other OFF times are programmed. The selector is switched to AUTO. If anything is incorrect, the timer is reset and reprogrammed.

TABLE C-1

NET FLOW CONTROLLER SETTING

DATE	1 CANISTER	2 CANISTERS

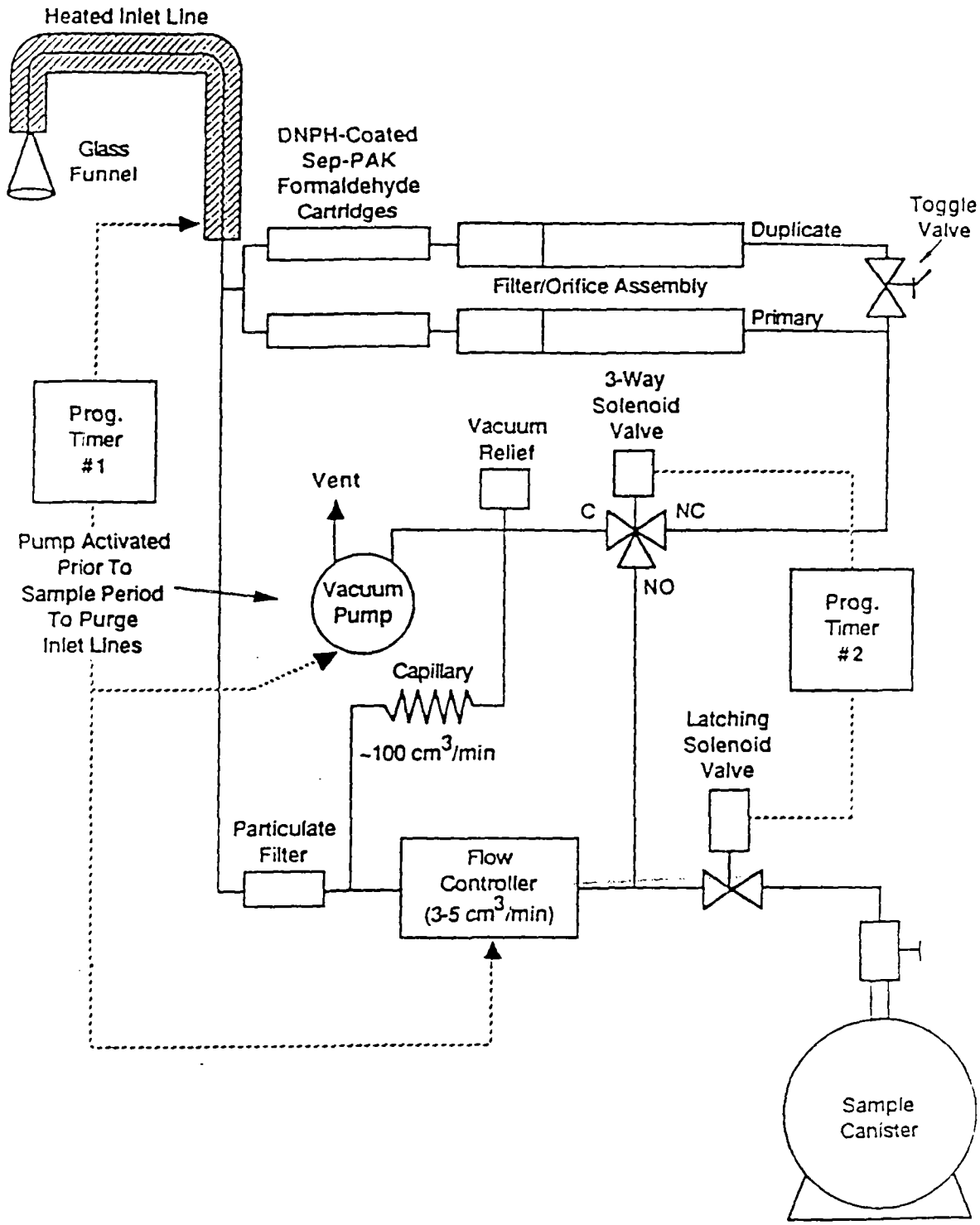


FIGURE C-1. U.S. ENVIRONMENTAL PROTECTION AGENCY UTAP SAMPLER SCHEMATIC OF SAMPLE INLET CONNECTIONS

Appendix J--Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere

1.0 Applicability.

1.1 This method provides for the measurement of the mass concentration of particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers (PM10) in ambient air over a 24-hour period for purposes of determining attainment and maintenance of the primary and secondary national ambient air quality standards for particulate matter specified in Sec. 50.6 of this chapter. The measurement process is nondestructive, and the PM10 sample can be subjected to subsequent physical or chemical analyses. Quality assurance procedures and guidance are provided in Part 58, Appendices A and B, of this chapter and in References 1 and 2.

2.0 Principle.

2.1 An air sampler draws ambient air at a constant flow rate into a specially shaped inlet where the suspended particulate matter is inertially separated into one or more size fractions within the PM10 size range. Each size fraction in the PM10 size range is then collected on a separate filter over the specified sampling period. The particle size discrimination characteristics (sampling effectiveness and 50 percent cutpoint) of the sampler inlet are prescribed as performance specifications in Part 53 of this chapter.

2.2 Each filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain due to collected PM10. The total volume of air sampled, corrected to EPA reference conditions (25 deg. C, 101.3 kPa), is determined from the measured flow rate and the sampling time. The mass concentration of PM10 in the ambient air is computed as the total mass of collected particles in the PM10 size range divided by the volume of air sampled, and is expressed in micrograms per standard cubic meter (micro-g/ std m**3). For PM10 samples collected at temperatures and pressures significantly different from EPA reference conditions, these corrected concentrations sometimes differ substantially from actual concentrations (in micrograms per actual cubic meter), particularly at high elevations. Although not required, the actual PM10 concentration can be calculated from the corrected concentration, using the average ambient temperature and barometric pressure during the sampling period.

2.3 A method based on this principle will be considered a reference method only if (a) the associated sampler meets the requirements specified in this appendix and the requirements in Part 53 of this chapter, and (b) the method has been designated as a reference method in accordance with Part 53 of this chapter.

3.0 Range.

3.1 The lower limit of the mass concentration range is determined by the repeatability of filter tare weights, assuming the nominal air sample volume for the sampler. For samplers having an automatic filter-changing mechanism, there may be no upper limit. For samplers that do not have an automatic filter-changing mechanism, the upper limit is determined by the filter mass loading beyond which the sampler no longer maintains the operating flow rate within specified limits due to increased pressure drop across the loaded filter. This upper limit cannot be specified precisely because it is a complex function of the ambient particle size distribution and type, humidity, filter type, and perhaps other factors. Nevertheless, all samplers should be

capable of measuring 24-hour PM10 mass concentrations of at least 300 micro-g/std m**3 while maintaining the operating flow rate within the specified limits.

4.0 Precision.

4.1 The precision of PM10 samplers must be 5 micro-g/m**3 for PM10 concentrations below 80 micro-g/m**3 and 7 percent for PM10 concentrations above 80 micro-g/m**3, as required by Part 53 of this chapter, which prescribes a test procedure that determines the variation in the PM10 concentration measurements of identical samplers under typical sampling conditions. Continual assessment of precision via collocated samplers is required by Part 58 of this chapter for PM10 samplers used in certain monitoring networks.

5.0 Accuracy.

5.1 Because the size of the particles making up ambient particulate matter varies over a wide range and the concentration of particles varies with particle size, it is difficult to define the absolute accuracy of PM10 samplers. Part 53 of this chapter provides a specification for the sampling effectiveness of PM10 samplers. This specification requires that the expected mass concentration calculated for a candidate PM10 sampler, when sampling a specified particle size distribution, be within +/-10 percent of that calculated for an ideal sampler whose sampling effectiveness is explicitly specified. Also, the particle size for 50 percent sampling effectivensss is required to be 10+/-0.5 micrometers. Other specifications related to accuracy apply to flow measurement and calibration, filter media, analytical (weighing) procedures, and artifact. The flow rate accuracy of PM10 samplers used in certain monitoring networks is required by Part 58 of this chapter to be assessed periodically via flow rate audits.

6.0 Potential Sources of Error.

6.1 Volatile Particles. Volatile particles collected on filters are often lost during shipment and/or storage of the filters prior to the post-sampling weighing /3/. Although shipment or storage of loaded filters is sometimes unavoidable, filters should be reweighed as soon as practical to minimize these losses.

6.2 Artifacts. Positive errors in PM10 concentration measurements may result from retention of gaseous species on filters 4, 5. Such errors include the retention of sulfur dioxide and nitric acid. Retention of sulfur dioxide on filters, followed by oxidation to sulfate, is referred to as artifact sulfate formation, a phenomenon which increases with increasing filter alkalinity /6/. Little or no artifact sulfate formation should occur using filters that meet the alkalinity specification in section 7.2.4. Artifact nitrate formation, resulting primarily from retention of nitric acid, occurs to varying degrees on many filter types, including glass fiber, cellulose ester, and many quartz fiber filters 5, 7, 8, 9, 10. Loss of true atmospheric particulate nitrate during or following sampling may also occur due to dissociation or chemical reaction. This phenomenon has been observed on Teflon(R) filters /8/ and inferred for quartz fiber filters 11, 12. The magnitude of nitrate artifact errors in PM10 mass concentration measurements will vary with location and ambient temperature; however, for most sampling locations, these errors are expected to be small.

6.3 Humidity. The effects of ambient humidity on the sample

are unavoidable. The filter equilibration procedure in section 9.0 is designed to minimize the effects of moisture on the filter medium.

6.4 Filter Handling. Careful handling of filters between presampling and postsampling weighings is necessary to avoid errors due to damaged filters or loss of collected particles from the filters. Use of a filter cartridge or cassette may reduce the magnitude of these errors. Filters must also meet the integrity specification in section 7.2.3.

6.5 Flow Rate Variation. Variations in the sampler's operating flow rate may alter the particle size discrimination characteristics of the sampler inlet. The magnitude of this error will depend on the sensitivity of the inlet to variations in flow rate and on the particle distribution in the atmosphere during the sampling period. The use of a flow control device (section 7.1.3) is required to minimize this error.

6.6 Air Volume Determination. Errors in the air volume determination may result from errors in the flow rate and/or sampling time measurements. The flow control device serves to minimize errors in the flow rate determination, and an elapsed time meter (section 7.1.5) is required to minimize the error in the sampling time measurement.

7.0 Apparatus.

7.1 PM10 Sampler.

7.1.1 The sampler shall be designed to:

a. Draw the air sample into the sampler inlet and through the particle collection filter at a uniform face velocity.

b. Hold and seal the filter in a horizontal position so that sample air is drawn downward through the filter.

c. Allow the filter to be installed and removed conveniently.

d. Protect the filter and sampler from precipitation and prevent insects and other debris from being sampled.

e. Minimize air leaks that would cause error in the measurement of the air volume passing through the filter.

f. Discharge exhaust air at a sufficient distance from the sampler inlet to minimize the sampling of exhaust air.

g. Minimize the collection of dust from the supporting surface.

7.1.2 The sampler shall have a sample air inlet system that, when operated within a specified flow rate range, provides particle size discrimination characteristics meeting all of the applicable performance specifications prescribed in Part 53 of this chapter. The sampler inlet shall show no significant wind direction dependence. The latter requirement can generally be satisfied by an inlet shape that is circularly symmetrical about a vertical axis.

7.1.3 The sampler shall have a flow control device capable of maintaining the sampler's operating flow rate within the flow rate limits specified for the sampler inlet over normal variations in line voltage and filter pressure drop.

7.1.4 The sampler shall provide a means to measure the total flow rate during the sampling period. A continuous flow recorder is recommended but not required. The flow measurement device shall be accurate to +/-2 percent.

7.1.5 A timing/control device capable of starting and stopping the sampler shall be used to obtain a sample collection period of 24 +/-1 hr (1,440 +/-60 min). An elapsed time meter, accurate to within +/-15 minutes, shall be used to measure sampling time.

This meter is optional for samplers with continuous flow recorders if the sampling time measurement obtained by means of the recorder meets the +/-15 minute accuracy specification.

7.1.6 The sampler shall have an associated operation or instruction manual as required by Part 53 of this chapter which includes detailed instructions on the calibration, operation, and maintenance of the sampler.

7.2 Filters.

7.2.1 Filter Medium. No commercially available filter medium is ideal in all respects for all samplers. The user's goals in sampling determine the relative importance of various filter characteristics (e.g., cost, ease of handling, physical and chemical characteristics, etc.) and, consequently, determine the choice among acceptable filters. Furthermore, certain types of filters may not be suitable for use with some samplers, particularly under heavy loading conditions (high mass concentrations), because of high or rapid increase in the filter flow resistance that would exceed the capability of the sampler's flow control device. However, samplers equipped with automatic filter-changing mechanisms may allow use of these types of filters. The specifications given below are minimum requirements to ensure acceptability of the filter medium for measurement of PM10 mass concentrations. Other filter evaluation criteria should be considered to meet individual sampling and analysis objectives.

7.2.2 Collection Efficiency. >/=99 percent, as measured by the DOP test (ASTM-2986) with 0.3 micro-m particles at the sampler's operating face velocity.

7.2.3 Integrity. +/-5 micro-g/m**3 (assuming sampler's nominal 24-hour air sample volume). Integrity is measured as the PM10 concentration equivalent corresponding to the average difference between the initial and the final weights of a random sample of test filters that are weighed and handled under actual or simulated sampling conditions, but have no air sample passed through them (i.e., filter blanks). As a minimum, the test procedure must include initial equilibration and weighing, installation on an inoperative sampler, removal from the sampler, and final equilibration and weighing.

7.2.4 Alkalinity. <25 microequivalents/gram of filter, as measured by the procedure given in Reference 13 following at least two months storage in a clean environment (free from contamination by acidic gases) at room temperature and humidity.

7.3 Flow Rate Transfer Standard. The flow rate transfer standard must be suitable for the sampler's operating flow rate and must be calibrated against a primary flow or volume standard that is traceable to the National Bureau of Standards (NBS). The flow rate transfer standard must be capable of measuring the sampler's operating flow rate with an accuracy of +/-2 percent.

7.4 Filter Conditioning Environment.

7.4.1 Temperature range: 15 deg. to 30 deg. C.

7.4.2 Temperature control: +/-3 deg. C.

7.4.3 Humidity range: 20% to 45% RH.

7.4.4 Humidity control: +/-5% RH.

7.5 Analytical Balance. The analytical balance must be suitable for weighing the type and size of filters required by the sampler. The range and sensitivity required will depend on the filter tare weights and mass loadings. Typically, an analytical balance with a sensitivity of 0.1 mg is required for high volume

samplers (flow rates >0.5 m**3/min). Lower volume samplers (flow rates <0.5 m**3/min) will require a more sensitive balance.

8.0 Calibration.

8.1 General Requirements.

8.1.1 Calibration of the sampler's flow measurement device is required to establish traceability of subsequent flow measurements to a primary standard. A flow rate transfer standard calibrated against a primary flow or volume standard shall be used to calibrate or verify the accuracy of the sampler's flow measurement device.

8.1.2 Particle size discrimination by inertial separation requires that specific air velocities be maintained in the sampler's air inlet system. Therefore, the flow rate through the sampler's inlet must be maintained throughout the sampling period within the design flow rate range specified by the manufacturer. Design flow rates are specified as actual volumetric flow rates, measured at existing conditions of temperature and pressure (Qa). In contrast, mass concentrations of PM10 are computed using flow rates corrected to EPA reference conditions of temperature and pressure (Qstd).

8.2 Flow Rate Calibration Procedure.

8.2.1 PM10 samplers employ various types of flow control and flow measurement devices. The specific procedure used for flow rate calibration or verification will vary depending on the type of flow controller and flow indicator employed. Calibration in terms of actual volumetric flow rates (Qa) is generally recommended, but other measures of flow rate (e.g., Qstd) may be used provided the requirements of section 8.1 are met. The general procedure given here is based on actual volumetric flow units (Qa) and serves to illustrate the steps involved in the calibration of a PM10 sampler. Consult the sampler manufacturer's instruction manual and Reference 2 for specific guidance on calibration. Reference 14 provides additional information on the use of the commonly used measures of flow rate and their interrelationships.

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

8.2.3 Following the sampler manufacturer's instruction manual, remove the sampler inlet and connect the flow rate transfer standard to the sampler such that the transfer standard accurately measures the sampler's flow rate. Make sure there are no leaks between the transfer standard and the sampler.

8.2.4 Choose a minimum of three flow rates (actual m**3/min), spaced over the acceptable flow rate range specified for the inlet (see 7.1.2) that can be obtained by suitable adjustment of the sampler flow rate. In accordance with the sampler manufacturer's instruction manual, obtain or verify the calibration relationship between the flow rate (actual m**3/min) as indicated by the transfer standard and the sampler's flow indicator response. Record the ambient temperature and barometric pressure. Temperature and pressure corrections to subsequent flow indicator readings may be required for certain types of flow measurement devices. When such corrections are necessary, correction on an individual or daily

basis is preferable. However, seasonal average temperature and average barometric pressure for the sampling site may be incorporated into the sampler calibration to avoid daily corrections. Consult the sampler manufacturer's instruction manual and Reference 2 for additional guidance.

8.2.5 Following calibration, verify that the sampler is operating at its design flow rate (actual m**3/min) with a clean filter in place.

8.2.6 Replace the sampler inlet.

9.0 Procedure.

9.1 The sampler shall be operated in accordance with the specific guidance provided in the sampler manufacturer's instruction manual and in Reference 2. The general procedure given here assumes that the sampler's flow rate calibration is based on flow rates at ambient conditions (Qa) and serves to illustrate the steps involved in the operation of a PM10 sampler.

9.2 Inspect each filter for pinholes, particles, and other imperfections. Establish a filter information record and assign an identification number to each filter.

9.3 Equilibrate each filter in the conditioning environment (see 7.4) for at least 24 hours.

9.4 Following equilibration, weigh each filter and record the presampling weight with the filter identification number.

9.5 Install a preweighed filter in the sampler following the instructions provided in the sampler manufacturer's instruction manual.

9.6 Turn on the sampler and allow it to establish run-temperature conditions. Record the flow indicator reading and, if needed, the ambient temperature and barometric pressure. Determine the sampler flow rate (actual m**3/min) in accordance with the instructions provided in the sampler manufacturer's instruction manual. NOTE.--No onsite temperature or pressure measurements are necessary if the sampler's flow indicator does not require temperature or pressure corrections or if seasonal average temperature and average barometric pressure for the sampling site are incorporated into the sampler calibration (see step 8.2.4). If individual or daily temperature and pressure corrections are required, ambient temperature and barometric pressure can be obtained by on-site measurements or from a nearby weather station. Barometric pressure readings obtained from airports must be station pressure, not corrected to sea level, and may need to be corrected for differences in elevation between the sampling site and the airport.

9.7 If the flow rate is outside the acceptable range specified by the manufacturer, check for leaks, and if necessary, adjust the flow rate to the specified setpoint. Stop the sampler.

9.8 Set the timer to start and stop the sampler at appropriate times. Set the elapsed time meter to zero or record the initial meter reading.

9.9 Record the sample information (site location or identification number, sample date, filter identification number, and sampler model and serial number).

9.10 Sample for 24+/-1 hours.

9.11 Determine and record the average flow rate (Qa) in actual m/3//min for the sampling period in accordance with the instructions provided in the sampler manufacturer's instruction manual. Record the elapsed time meter final reading and, if

needed, the average ambient temperature and barometric pressure for the sampling period (see note following step 9.6).

9.12 Carefully remove the filter from the sampler, following the sampler manufacturer's instruction manual. Touch only the outer edges of the filter.

9.13 Place the filter in a protective holder or container (e.g., petri dish, glassine envelope, or manila folder).

9.14 Record any factors such as meteorological conditions, construction activity, fires or dust storms, etc., that might be pertinent to the measurement on the filter information record.

9.15 Transport the exposed sample filter to the filter conditioning environment as soon as possible for equilibration and subsequent weighing.

9.16 Equilibrate the exposed filter in the conditioning environment for at least 24 hours under the same temperature and humidity conditions used for presampling filter equilibration (see 9.3).

9.17 Immediately after equilibration, reweigh the filter and record the postsampling weight with the filter identification number.

10.0 Sampler Maintenance.

10.1 The PM10 sampler shall be maintained in strict accordance with the maintenance procedures specified in the sampler manufacturer's instruction manual.

11.0 Calculations.

11.1 Calculate the average flow rate over the sampling period corrected to EPA reference conditions as Qstd. When the sampler's flow indicator is calibrated in actual volumetric units (Qa), Qstd is calculated as:

Qstd=Qax(Pav/Tav)(Tstd/Pstd)

where

Qstd=average flow rate at EPA reference conditions, std m**3 min;
Qa=average flow rate at ambient conditions, m**3 min;
Pav=average barometric pressure during the sampling period or average barometric pressure for the sampling site, kPa (or mm Hg);
Tav=average ambient temperature during the sampling period or seasonal average ambient temperature for the sampling site, K;
Tstd=standard temperature, defined as 298 K;
Pstd=standard pressure, defined as 101.3 kPa (or 760 mm Hg).

11.2 Calculate the total volume of air sampled as:

Vstd=Qstdxt

where

Vstd=total air sampled in standard volume units, std m**3;
t=sampling time, min.

11.3 Calculate the PM10 concentration as:

PM10=(Wf-Wi)x10**6 Vstd

where

PM10=mass concentration of PM10, micro-g/std m**3;
Wf, Wi=final and initial weights of filter collecting PM10 particles, g; 10**6=conversion of g to micro-g.

Note: If more than one size fraction in the PM10 size range is collected by the sampler, the sum of the net weight gain by each collection filter [S(Wf- Wi)] is used to calculate the PM10 mass concentration.

12.0 References.

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[52 FR 24664, July 1, 1987; 52 FR 29467, Aug. 7, 1987]

APPENDIX C

COPIES OF LABORATORY DATA

Fibertec
Environmental
Services

June 18, 1998

Fibertec Project # 57012
Project Identification: Toledo Project

Mr. Dan Rochette
Derenzo & Associates
39201 Schoolcraft, Suite B-10
Livonia, MI 48150

Dear Mr. Rochette,

Thank you for selecting Fibertec Environmental Services as your analytical laboratory. The samples you submitted have been analyzed as requested and the results compiled in the enclosed report. Please note samples will be disposed of 30 days after reporting date.

If you should have any questions regarding these results or if we may be of further assistance to you, please contact me at (517) 699-0345.

Sincerely,

Daryl P. Strandbergh
Laboratory Director

DPS/th

Enclosures

2280 Aurelius Road Holt, Michigan 48842 Telephone: (517) 699-0345 Facsimile: (517) 699-0388
29300 Wall Street Wixom, Michigan 48393 Telephone: (248) 348-5598 Facsimile: (248) 348-4896

SITE #1-FILTER #59

Code	Parameter Name	Parameter #	5/28/98
PM	INHALABLE PARTICULATES (PM-10)	81102	99

SITE #2-FILTER #60

Code	Parameter Name	Parameter #	5/28/98
PM	INHALABLE PARTICULATES (PM-10)	81102	100

SITE #3-FILTER #61

Code	Parameter Name	Parameter #	5/28/98
PM	INHALABLE PARTICULATES (PM-10)	81102	96

SITE #4-FILTER #62

Code	Parameter Name	Parameter #	5/28/98
PM	INHALABLE PARTICULATES (PM-10)	81102	110

SITE #1-FILTER #71

Code	Parameter Name	Parameter #	5/29/98
PM	INHALABLE PARTICULATES (PM-10)	81102	77

SITE #2-FILTER #70

Code	Parameter Name	Parameter #	5/29/98
PM	INHALABLE PARTICULATES (PM-10)	81102	74

SITE #3-FILTER #72

Code	Parameter Name	Parameter #	5/29/98
PM	INHALABLE PARTICULATES (PM-10)	81102	75

SITE #4-FILTER #69

Code	Parameter Name	Parameter #	5/29/98
PM	INHALABLE PARTICULATES (PM-10)	81102	83

NOTE: Particulate results reported as mg

57012 (SPECIAL PROJECT)

TOLEDO PROJECT

DERENZO & ASSOCIATES 39201 SCHOOLCRAFT SUITE B-10 LIVONIA, MI. 48150 PHONE:(313) 464-3880 FAX:(313) 464-4368

CHAIN OF CUSTODY

Send report to:	Project:	Lab/Lab Number:	Due Date: A.S.A.P.	PM-10			
Sample ID	Date/ Time	Filter Number	# Containers Matrix				
1 SITE #1 PM10	5-28-98	59	1	✓			
2 SITE #2 PM10	5-28-98	60	1	✓			
3 SITE #3 PM10	5-28-98	61	1	✓			
4 SITE #4 PM10	5-28-98	62	1	✓			
5							
6 SITE #1 PM10	5-29-98	71	1	✓			
7 SITE #2 PM10	5-29-98	70	1	✓			
8 SITE #3 PM10	5-29-98	72	1	✓			
9 SITE #4 PM10	5-29-98	69	1	✓			
10							
11							
12							

Collected By: KRIS SIMONIAN	Date: 5-30-98		Time:
Relinquished By: K. Simonian	Date:		Time:
Received By: [signature]	Date: 4/3/98		Time:
Relinquished By:	Date:		Time:
Received for Lab by:	Date:		Time:
Relinquished by Lab by:	Date:		Time:
Received By:	Date:		Time:

Remarks:

*Matrix S=Solid L=Liquid W=Water GW=ground Water SL=Sludge

Fibertec
Environmental
Services

July 15, 1998

Mr. Chris Simonian
Derenzo and Associates, Inc.
39201 Schoolcraft
Suite B-10
Livonia, MI 48150

Dear Mr. Simonian:

Enclosed are copies of our laboratory notebook with gravimetric results for your Toledo project PM-10 results, Fibertec project No. 57012.

It is a pleasure to be of service to you. If you have any questions or need further information, please call me at (517) 699-0345.

Sincerely,

Fibertec Environmental Services

Daryl P. Strandbergh
Laboratory Director

DPS/nw

2280 Aurelius Road Holt, Michigan 48842 Telephone: (517) 699-0345 Facsimile: (517) 699-0388
29300 Wall Street Wixom, Michigan 48393 Telephone: (248) 348-5598 Facsimile: (248) 348-4896

Post weights

Date	6-5-98	6-8-98
Temp (°F)	67	69
Hum (%)	37	37
5.0g calibration	5.0003	5.0002
Analyst	AR	AR

Filter #	wt (gm)	wt (gm)	preweight (gm)	Difference (mg)
000059	4.4816	4.4820 ✓	4.3832	98.8
000060	4.4808	4.4812 ✓	4.3778	103.4
000061	4.4663	4.4666 ✓	4.3707	95.9
000062	4.5166	4.5168 ✓	4.4098	107.0
000069	4.4779	4.4775 ✓	4.3941	83.4
000070	4.4403	4.4404 ✓	~~4.3667~~ 4.3667 JPS	73.7
000071	4.4475	4.4478 ✓	4.3708	77.0
000072	4.4638	4.4634 ✓	4.3881	75.3

FILTER (8x10 Quartz) Preweights

Date:	5-14-98	5-14-98	5-14-98	5-14-98	5-14-98
Temp°:	68	71	71	72	72
Humidity:	49	48	~~47~~ 47 DPS	46	47
5.0g cal.	5.0002	5.0003	5.0002	5.0002	5.0002
Analyst	DPS	DPS	DPS	DPS	DPS
Filter #					
000051	4.4118	4.4166 ~~4.3803~~	4.4170 ✓		
52	4.3724	4.3780	4.3789	4.3793 ✓	
53	4.3633	4.3691	4.3697	4.3707	4.3709 ✓
54	4.3585	4.3632	4.3643	4.3654	4.3655 ✓
55	4.3749	4.3803	4.3816	4.3825	4.3827 ✓
56	4.3740	4.3794	4.3803	4.3809	4.3813 ✓
57	4.3735	4.3785	4.3799	4.3807	4.3811 ✓
58	4.3809	4.3859	4.3872	4.3882	4.3887 ✓
59	4.3761	4.3813 ~~4.3809~~ DPS	4.3827	4.3832 ✓	
60	4.3715	4.3758	4.3773	4.3778 ✓	
61	4.3638	4.3680	4.3697	4.3707	4.3708 ✓
62	4.4024	4.4065	4.4084	4.4095	4.4098 ✓
63	4.3843	4.3883	4.3903	4.3914	4.3914 ✓
64	4.3831	4.3871	4.3892	4.3901	4.3902 ✓
65	4.4032	4.4067	4.4087	4.4096	4.4099 ✓
66	4.3875	4.3908	4.3930	4.3936	4.3939 ✓
67	4.3837	4.3869	4.3892	4.3900	4.3902 ✓
68	4.3759	4.3788	4.3812	4.3819	4.3819 ✓
69	4.3878	4.3909	4.3933	4.3941	4.3941 ✓
70	4.3604	4.3640	4.3657	4.3664	4.3667 ✓
71	4.3645	4.3676	4.3698	4.3706	4.3708 ✓
72	4.3821	4.3851	4.3876	4.3881 ✓	
73	4.3732	4.3756	4.3784	4.3791	4.3794 ✓
74	4.3739	4.3765	4.3794	4.3798 ✓	
75	4.3779	4.3803	4.3837	4.3846	4.3848 ✓

Fibertec
Environmental
Services

July 20, 1998

Mr. Brian Kuehn
Derenzo and Associates, Inc.
39201 Schoolcraft
Suite B-10
Livonia, MI 48150

Dear Mr. Kuehn:

Following up on our discussion about the Toledo project, Fibertec project No. 57012, a blank filter was not analyzed along with the samples. However, the samples along with a blank were available for re-weighing. The results of the re-weighing are enclosed. Please note the blank corrected results correlate well with the reported results.

I apologize for any inconvenience this may have caused and assure you that this procedural error has been corrected.

It is a pleasure to be of service to you. If you have any questions or need further information, please call me at (517) 699-0345.

Sincerely,

Fibertec Environmental Services

Daryl P. Strandbergh
Laboratory Director

DPS/nw

2280 Aurelius Road Holt, Michigan 48842 Telephone: (517) 699-0345 Facsimile: (517) 699-0388
29300 Wall Street Wixom, Michigan 48393 Telephone: (248) 348-5598 Facsimile: (248) 348-4896

DATE	7/20/98
TEMP (°F)	73
HUM (%)	50
5.0g calib	5.0000
Analyst	DBS

Filter #	(gm)	Tare wt (gm)	particulate wt (mg)	Blank corrected (mg)	Reco (mg)
000002 (BLANK)	4.4131	4.4044	8.7	—	
59	4.4889	4.3832	105.7	97	99
60	4.4883	4.3778	110.5	101.8	100
61	4.4727	4.3708	101.9	93.2	96
62	4.5239	4.4098	114.1	105.4	110
69	4.4848	4.3941	90.7	82.0	83
70	4.4476	~~4.3667~~ 4.3667	80.9	72.2	74
71	4.4558	4.3708	85.0	76.3	77
72	4.4719	4.3881	83.8	75.1	75

@AIR TOXICS LTD.

AN ENVIRONMENTAL ANALYTICAL LABORATORY

WORK ORDER #: 9806030

Work Order Summary

CLIENT: Kris Simonian
Derenzo & Associates
39201 Schoolcraft Rd., Ste B-10
Livonia, MI 48150

BILL TO: Same

PHONE: 313-464-3880
FAX: 313-464-4368
DATE RECEIVED: 6/2/98
DATE COMPLETED: 6/12/98

P.O. # 21978
PROJECT # 9804002 Hull & Assoc

FRACTION #	NAME	TEST	RECEIPT VAC./PRES.
01A	Site #1 WED 769	TO-14/TIC's	10.0 "Hg
02A	Site #2 WED 12937	TO-14/TIC's	11.5 "Hg
03A	Site #3 WED 20951	TO-14/TIC's	8.5 "Hg
04A	Site #4 WED 12717	TO-14/TIC's	10.5 "Hg
05A	Site #1 THUR 12943	TO-14/TIC's	9.0 "Hg
06A	Site #2 THUR 10782	TO-14/TIC's	8.5 "Hg
07A	Site #3 THUR 10763	TO-14/TIC's	9.5 "Hg
08A	Site #4 THUR 23888	TO-14/TIC's	10.0 "Hg
09A	pit THUR 487	TO-14/TIC's	10.0 "Hg
10A	Site #1 FRI 22513	TO-14/TIC's	9.0 "Hg
10AA	Site #1 FRI 22513 Duplicate	TO-14/TIC's	9.0 "Hg
11A	Site #2 FRI 2993	TO-14/TIC's	6.0 "Hg
12A	Site #3 FRI 96115	TO-14/TIC's	9.5 "Hg
13A	Site #4 FRI 12704	TO-14/TIC's	7.5 "Hg
14A	Method Spike	TO-14/TIC's	NA
15A	Lab Blank	TO-14/TIC's	NA
15B	Lab Blank	TO-14/TIC's	NA
15C	Lab Blank	TO-14/TIC's	NA

CERTIFIED BY: [signature] for
Laboratory Director

DATE: 6/12/98

Certification numbers: CA ELAP - 1149, NY ELAP - 11291, UT ELAP - E-217

180 BLUE RAVINE ROAD, SUITE B FOLSOM, CA 95630
(916) 985-1000 • (800) 985-5955 • FAX (916) 985-1020

AIR TOXICS LTD.

SAMPLE NAME : Site #1 WED 769

ID#: 9806030-01A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060415	Date of Collection:	5/27/98
Dil. Factor:	2.01	Date of Analysis:	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	1.0	Not Detected
Freon 114	1.0	Not Detected
Chloromethane	1.0	1.2
Vinyl Chloride	1.0	Not Detected
Bromomethane	1.0	Not Detected
Chloroethane	1.0	Not Detected
Freon 11	1.0	Not Detected
1,1-Dichloroethene	1.0	Not Detected
Freon 113	1.0	Not Detected
Methylene Chloride	1.0	Not Detected
1,1-Dichloroethane	1.0	Not Detected
cis-1,2-Dichloroethene	1.0	Not Detected
Chloroform	1.0	Not Detected
1,1,1-Trichloroethane	1.0	Not Detected
Carbon Tetrachloride	1.0	Not Detected
Benzene	1.0	Not Detected
1,2-Dichloroethane	1.0	Not Detected
Trichloroethene	1.0	Not Detected
1,2-Dichloropropane	1.0	Not Detected
cis-1,3-Dichloropropene	1.0	Not Detected
Toluene	1.0	1.7
trans-1,3-Dichloropropene	1.0	Not Detected
1,1,2-Trichloroethane	1.0	Not Detected
Tetrachloroethene	1.0	Not Detected
Ethylene Dibromide	1.0	Not Detected
Chlorobenzene	1.0	Not Detected
Ethyl Benzene	1.0	Not Detected
m,p-Xylene	1.0	Not Detected
o-Xylene	1.0	Not Detected
Styrene	1.0	Not Detected
1,1,2,2-Tetrachloroethane	1.0	Not Detected
1,3,5-Trimethylbenzene	1.0	Not Detected
1,2,4-Trimethylbenzene	1.0	Not Detected
1,3-Dichlorobenzene	1.0	Not Detected
1,4-Dichlorobenzene	1.0	Not Detected
Chlorotoluene	1.0	Not Detected
1,2-Dichlorobenzene	1.0	Not Detected
1,2,4-Trichlorobenzene	1.0	Not Detected
Hexachlorobutadiene	1.0	Not Detected
Propylene	4.0	Not Detected
1,3-Butadiene	4.0	Not Detected
Acetone	4.0	7.5
Carbon Disulfide	4.0	Not Detected
2-Propanol	4.0	Not Detected
trans-1,2-Dichloroethene	4.0	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #1 WED 769

ID#: 9806030-01A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060415	**Date of Collection:**	5/27/98
Dil. Factor:	2.01	**Date of Analysis:**	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	4.0	Not Detected
2-Butanone (Methyl Ethyl Ketone)	4.0	Not Detected
Hexane	4.0	Not Detected
Tetrahydrofuran	4.0	Not Detected
Cyclohexane	4.0	Not Detected
1,4-Dioxane	4.0	7.0
Bromodichloromethane	4.0	Not Detected
4-Methyl-2-pentanone	4.0	Not Detected
2-Hexanone	4.0	Not Detected
Dibromochloromethane	4.0	Not Detected
Bromoform	4.0	Not Detected
4-Ethyltoluene	4.0	Not Detected
Ethanol	4.0	5.1
Methyl tert-Butyl Ether	4.0	Not Detected
Heptane	4.0	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Heptane, 4-ethyl-2,2,6,6-tetramethyl-	62108-31-0	72 %	6.1
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	114	70-130
Toluene-d8	109	70-130
4-Bromofluorobenzene	107	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #2 WED 12937

ID#: 9806030-02A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060416	**Date of Collection:**	5/27/98
Dil. Factor:	2.17	**Date of Analysis:**	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	1.1	Not Detected
Freon 114	1.1	Not Detected
Chloromethane	1.1	1.2
Vinyl Chloride	1.1	Not Detected
Bromomethane	1.1	Not Detected
Chloroethane	1.1	Not Detected
Freon 11	1.1	Not Detected
1,1-Dichloroethene	1.1	Not Detected
Freon 113	1.1	Not Detected
Methylene Chloride	1.1	Not Detected
1,1-Dichloroethane	1.1	Not Detected
cis-1,2-Dichloroethene	1.1	Not Detected
Chloroform	1.1	Not Detected
1,1,1-Trichloroethane	1.1	Not Detected
Carbon Tetrachloride	1.1	Not Detected
Benzene	1.1	1.6
1,2-Dichloroethane	1.1	Not Detected
Trichloroethene	1.1	Not Detected
1,2-Dichloropropane	1.1	Not Detected
cis-1,3-Dichloropropene	1.1	Not Detected
Toluene	1.1	3.4
trans-1,3-Dichloropropene	1.1	Not Detected
1,1,2-Trichloroethane	1.1	Not Detected
Tetrachloroethene	1.1	Not Detected
Ethylene Dibromide	1.1	Not Detected
Chlorobenzene	1.1	Not Detected
Ethyl Benzene	1.1	Not Detected
m,p-Xylene	1.1	Not Detected
o-Xylene	1.1	Not Detected
Styrene	1.1	Not Detected
1,1,2,2-Tetrachloroethane	1.1	Not Detected
1,3,5-Trimethylbenzene	1.1	Not Detected
1,2,4-Trimethylbenzene	1.1	Not Detected
1,3-Dichlorobenzene	1.1	Not Detected
1,4-Dichlorobenzene	1.1	Not Detected
Chlorotoluene	1.1	Not Detected
1,2-Dichlorobenzene	1.1	Not Detected
1,2,4-Trichlorobenzene	1.1	Not Detected
Hexachlorobutadiene	1.1	Not Detected
Propylene	4.3	Not Detected
1,3-Butadiene	4.3	Not Detected
Acetone	4.3	8.6
Carbon Disulfide	4.3	Not Detected
2-Propanol	4.3	Not Detected
trans-1,2-Dichloroethene	4.3	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #2 WED 12937

ID#: 9806030-02A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060416	Date of Collection:	5/27/98
Dil. Factor:	2.17	Date of Analysis:	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	4.3	Not Detected
2-Butanone (Methyl Ethyl Ketone)	4.3	Not Detected
Hexane	4.3	Not Detected
Tetrahydrofuran	4.3	Not Detected
Cyclohexane	4.3	Not Detected
1,4-Dioxane	4.3	8.2
Bromodichloromethane	4.3	Not Detected
4-Methyl-2-pentanone	4.3	Not Detected
2-Hexanone	4.3	Not Detected
Dibromochloromethane	4.3	Not Detected
Bromoform	4.3	Not Detected
4-Ethyltoluene	4.3	Not Detected
Ethanol	4.3	6.2
Methyl tert-Butyl Ether	4.3	Not Detected
Heptane	4.3	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Aniline	62-53-3	87 %	6.0
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	108	70-130
Toluene-d8	107	70-130
4-Bromofluorobenzene	103	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #3 WED 20951

ID#: 9806030-03A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060417**	**Date of Collection: 5/27/98**
Dil. Factor:	**1.87**	**Date of Analysis: 6/4/98**

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.94	Not Detected
Freon 114	0.94	Not Detected
Chloromethane	0.94	1.1
Vinyl Chloride	0.94	Not Detected
Bromomethane	0.94	Not Detected
Chloroethane	0.94	Not Detected
Freon 11	0.94	Not Detected
1,1-Dichloroethene	0.94	Not Detected
Freon 113	0.94	Not Detected
Methylene Chloride	0.94	Not Detected
1,1-Dichloroethane	0.94	Not Detected
cis-1,2-Dichloroethene	0.94	Not Detected
Chloroform	0.94	Not Detected
1,1,1-Trichloroethane	0.94	Not Detected
Carbon Tetrachloride	0.94	Not Detected
Benzene	0.94	1.1
1,2-Dichloroethane	0.94	Not Detected
Trichloroethene	0.94	Not Detected
1,2-Dichloropropane	0.94	Not Detected
cis-1,3-Dichloropropene	0.94	Not Detected
Toluene	0.94	1.7
trans-1,3-Dichloropropene	0.94	Not Detected
1,1,2-Trichloroethane	0.94	Not Detected
Tetrachloroethene	0.94	Not Detected
Ethylene Dibromide	0.94	Not Detected
Chlorobenzene	0.94	Not Detected
Ethyl Benzene	0.94	Not Detected
m,p-Xylene	0.94	Not Detected
o-Xylene	0.94	Not Detected
Styrene	0.94	Not Detected
1,1,2,2-Tetrachloroethane	0.94	Not Detected
1,3,5-Trimethylbenzene	0.94	Not Detected
1,2,4-Trimethylbenzene	0.94	Not Detected
1,3-Dichlorobenzene	0.94	Not Detected
1,4-Dichlorobenzene	0.94	Not Detected
Chlorotoluene	0.94	Not Detected
1,2-Dichlorobenzene	0.94	Not Detected
1,2,4-Trichlorobenzene	0.94	Not Detected
Hexachlorobutadiene	0.94	Not Detected
Propylene	3.7	Not Detected
1,3-Butadiene	3.7	Not Detected
Acetone	3.7	6.0
Carbon Disulfide	3.7	Not Detected
2-Propanol	3.7	Not Detected
trans-1,2-Dichloroethene	3.7	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #3 WED 20951

ID#: 9806030-03A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060417	Date of Collection:	5/27/98
Dil. Factor:	1.87	Date of Analysis:	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	3.7	Not Detected
2-Butanone (Methyl Ethyl Ketone)	3.7	Not Detected
Hexane	3.7	Not Detected
Tetrahydrofuran	3.7	Not Detected
Cyclohexane	3.7	Not Detected
1,4-Dioxane	3.7	6.9
Bromodichloromethane	3.7	Not Detected
4-Methyl-2-pentanone	3.7	Not Detected
2-Hexanone	3.7	Not Detected
Dibromochloromethane	3.7	Not Detected
Bromoform	3.7	Not Detected
4-Ethyltoluene	3.7	Not Detected
Ethanol	3.7	7.6
Methyl tert-Butyl Ether	3.7	Not Detected
Heptane	3.7	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Undecane, 3,6-dimethyl-	17301-28-9	72 %	6.8
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	108	70-130
Toluene-d8	107	70-130
4-Bromofluorobenzene	105	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #4 WED 12717

ID#: 9806030-04A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060418	**Date of Collection:**	5/27/98
Dil. Factor:	2.06	**Date of Analysis:**	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	1.0	Not Detected
Freon 114	1.0	Not Detected
Chloromethane	1.0	1.1
Vinyl Chloride	1.0	Not Detected
Bromomethane	1.0	Not Detected
Chloroethane	1.0	Not Detected
Freon 11	1.0	Not Detected
1,1-Dichloroethene	1.0	Not Detected
Freon 113	1.0	Not Detected
Methylene Chloride	1.0	Not Detected
1,1-Dichloroethane	1.0	Not Detected
cis-1,2-Dichloroethene	1.0	Not Detected
Chloroform	1.0	Not Detected
1,1,1-Trichloroethane	1.0	Not Detected
Carbon Tetrachloride	1.0	Not Detected
Benzene	1.0	Not Detected
1,2-Dichloroethane	1.0	Not Detected
Trichloroethene	1.0	Not Detected
1,2-Dichloropropane	1.0	Not Detected
cis-1,3-Dichloropropene	1.0	Not Detected
Toluene	1.0	2.2
trans-1,3-Dichloropropene	1.0	Not Detected
1,1,2-Trichloroethane	1.0	Not Detected
Tetrachloroethene	1.0	Not Detected
Ethylene Dibromide	1.0	Not Detected
Chlorobenzene	1.0	Not Detected
Ethyl Benzene	1.0	Not Detected
m,p-Xylene	1.0	Not Detected
o-Xylene	1.0	Not Detected
Styrene	1.0	Not Detected
1,1,2,2-Tetrachloroethane	1.0	Not Detected
1,3,5-Trimethylbenzene	1.0	Not Detected
1,2,4-Trimethylbenzene	1.0	Not Detected
1,3-Dichlorobenzene	1.0	Not Detected
1,4-Dichlorobenzene	1.0	Not Detected
Chlorotoluene	1.0	Not Detected
1,2-Dichlorobenzene	1.0	Not Detected
1,2,4-Trichlorobenzene	1.0	Not Detected
Hexachlorobutadiene	1.0	Not Detected
Propylene	4.1	Not Detected
1,3-Butadiene	4.1	Not Detected
Acetone	4.1	7.0
Carbon Disulfide	4.1	Not Detected
2-Propanol	4.1	Not Detected
trans-1,2-Dichloroethene	4.1	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #4 WED 12717

ID#: 9806030-04A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060418	Date of Collection:	5/27/98
Dil. Factor:	2.06	Date of Analysis:	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	4.1	Not Detected
2-Butanone (Methyl Ethyl Ketone)	4.1	Not Detected
Hexane	4.1	Not Detected
Tetrahydrofuran	4.1	Not Detected
Cyclohexane	4.1	Not Detected
1,4-Dioxane	4.1	4.2
Bromodichloromethane	4.1	Not Detected
4-Methyl-2-pentanone	4.1	Not Detected
2-Hexanone	4.1	Not Detected
Dibromochloromethane	4.1	Not Detected
Bromoform	4.1	Not Detected
4-Ethyltoluene	4.1	Not Detected
Ethanol	4.1	8.2
Methyl tert-Butyl Ether	4.1	Not Detected
Heptane	4.1	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	109	70-130
Toluene-d8	107	70-130
4-Bromofluorobenzene	104	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #1 THUR 12943

ID#: 9806030-05A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060419d**	**Date of Collection:**	**5/28/98**
Dil. Factor:	**1.91**	**Date of Analysis:**	**6/ 4/98**

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.96	Not Detected
Freon 114	0.96	Not Detected
Chloromethane	0.96	1.5
Vinyl Chloride	0.96	Not Detected
Bromomethane	0.96	Not Detected
Chloroethane	0.96	Not Detected
Freon 11	0.96	Not Detected
1,1-Dichloroethene	0.96	Not Detected
Freon 113	0.96	Not Detected
Methylene Chloride	0.96	Not Detected
1,1-Dichloroethane	0.96	Not Detected
cis-1,2-Dichloroethene	0.96	Not Detected
Chloroform	0.96	Not Detected
1,1,1-Trichloroethane	0.96	Not Detected
Carbon Tetrachloride	0.96	Not Detected
Benzene	0.96	Not Detected
1,2-Dichloroethane	0.96	Not Detected
Trichloroethene	0.96	Not Detected
1,2-Dichloropropane	0.96	Not Detected
cis-1,3-Dichloropropene	0.96	Not Detected
Toluene	0.96	2.2
trans-1,3-Dichloropropene	0.96	Not Detected
1,1,2-Trichloroethane	0.96	Not Detected
Tetrachloroethene	0.96	Not Detected
Ethylene Dibromide	0.96	Not Detected
Chlorobenzene	0.96	Not Detected
Ethy Benzene	0.96	Not Detected
m,p-Xylene	0.96	Not Detected
o-Xylene	0.96	Not Detected
Styrene	0.96	Not Detected
1,1,2,2-Tetrachloroethane	0.96	Not Detected
1,3,5-Trimethylbenzene	0.96	Not Detected
1,2,4-Trimethylbenzene	0.96	Not Detected
1,3-Dichlorobenzene	0.96	Not Detected
1,4-Dichlorobenzene	0.96	Not Detected
Chlorotoluene	0.96	Not Detected
1,2-Dichlorobenzene	0.96	Not Detected
1,2,4-Trichlorobenzene	0.96	Not Detected
Hexachlorobutadiene	0.96	Not Detected
Propylene	3.8	Not Detected
1,3-Butadiene	3.8	Not Detected
Acetone	3.8	6.5
Carbon Disulfide	3.8	Not Detected
2-Propanol	3.8	Not Detected
trans-1,2-Dichloroethene	3.8	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #1 THUR 12943

ID#: 9806030-05A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060419d	Date of Collection:	5/28/98
Dil. Factor:	1.91	Date of Analysis:	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	3.8	Not Detected
2-Butanone (Methyl Ethyl Ketone)	3.8	Not Detected
Hexane	3.8	Not Detected
Tetrahydrofuran	3.8	Not Detected
Cyclohexane	3.8	Not Detected
1,4-Dioxane	3.8	4.4
Bromodichloromethane	3.8	Not Detected
4-Methyl-2-pentanone	3.8	Not Detected
2-Hexanone	3.8	Not Detected
Dibromochloromethane	3.8	Not Detected
Bromoform	3.8	Not Detected
4-Ethyltoluene	3.8	Not Detected
Ethanol	3.8	Not Detected
Methyl tert-Butyl Ether	3.8	Not Detected
Heptane	3.8	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	111	70-130
Toluene-d8	108	70-130
4-Bromofluorobenzene	106	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #2 THUR 10782

ID#: 9806030-06A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060420	Date of Collection:	5/28/98
Dil. Factor:	1.87	Date of Analysis:	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.94	Not Detected
Freon 114	0.94	Not Detected
Chloromethane	0.94	2.9
Vinyl Chloride	0.94	Not Detected
Bromomethane	0.94	Not Detected
Chloroethane	0.94	Not Detected
Freon 11	0.94	Not Detected
1,1-Dichloroethene	0.94	Not Detected
Freon 113	0.94	Not Detected
Methylene Chloride	0.94	Not Detected
1,1-Dichloroethane	0.94	Not Detected
cis-1,2-Dichloroethene	0.94	Not Detected
Chloroform	0.94	Not Detected
1,1,1-Trichloroethane	0.94	Not Detected
Carbon Tetrachloride	0.94	Not Detected
Benzene	0.94	Not Detected
1,2-Dichloroethane	0.94	Not Detected
Trichloroethene	0.94	Not Detected
1,2-Dichloropropane	0.94	Not Detected
cis-1,3-Dichloropropene	0.94	Not Detected
Toluene	0.94	3.5
trans-1,3-Dichloropropene	0.94	Not Detected
1,1,2-Trichloroethane	0.94	Not Detected
Tetrachloroethene	0.94	Not Detected
Ethylene Dibromide	0.94	Not Detected
Chlorobenzene	0.94	Not Detected
Ethyl Benzene	0.94	Not Detected
m,p-Xylene	0.94	0.99
o-Xylene	0.94	Not Detected
Styrene	0.94	Not Detected
1,1,2,2-Tetrachloroethane	0.94	Not Detected
1,3,5-Trimethylbenzene	0.94	Not Detected
1,2,4-Trimethylbenzene	0.94	Not Detected
1,3-Dichlorobenzene	0.94	Not Detected
1,4-Dichlorobenzene	0.94	Not Detected
Chlorotoluene	0.94	Not Detected
1,2-Dichlorobenzene	0.94	Not Detected
1,2,4-Trichlorobenzene	0.94	Not Detected
Hexachlorobutadiene	0.94	Not Detected
Propylene	3.7	Not Detected
1,3-Butadiene	3.7	Not Detected
Acetone	3.7	10
Carbon Disulfide	3.7	Not Detected
2-Propanol	3.7	Not Detected
trans-1,2-Dichloroethene	3.7	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #2 THUR 10782

ID#: 9806030-06A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060420	Date of Collection:	5/28/98
Dil. Factor:	1.87	Date of Analysis:	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	3.7	Not Detected
2-Butanone (Methyl Ethyl Ketone)	3.7	Not Detected
Hexane	3.7	Not Detected
Tetrahydrofuran	3.7	Not Detected
Cyclohexane	3.7	Not Detected
1,4-Dioxane	3.7	Not Detected
Bromodichloromethane	3.7	Not Detected
4-Methyl-2-pentanone	3.7	Not Detected
2-Hexanone	3.7	Not Detected
Dibromochloromethane	3.7	Not Detected
Bromoform	3.7	Not Detected
4-Ethyltoluene	3.7	Not Detected
Ethanol	3.7	Not Detected
Methyl tert-Butyl Ether	3.7	Not Detected
Heptane	3.7	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	109	70-130
Toluene-d8	107	70-130
4-Bromofluorobenzene	104	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #3 THUR 10763

ID#: 9806030-07A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060421**	**Date of Collection:**	**5/28/98**
Dil. Factor:	**1.96**	**Date of Analysis:**	**6/ 4/98**

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.98	Not Detected
Freon 114	0.98	Not Detected
Chloromethane	0.98	1.3
Vinyl Chloride	0.98	Not Detected
Bromomethane	0.98	Not Detected
Chloroethane	0.98	Not Detected
Freon 11	0.98	Not Detected
1,1-Dichloroethene	0.98	Not Detected
Freon 113	0.98	Not Detected
Methylene Chloride	0.98	Not Detected
1,1-Dichloroethane	0.98	Not Detected
cis-1,2-Dichloroethene	0.98	Not Detected
Chloroform	0.98	Not Detected
1,1,1-Trichloroethane	0.98	Not Detected
Carbon Tetrachloride	0.98	Not Detected
Benzene	0.98	Not Detected
1,2-Dichloroethane	0.98	Not Detected
Trichloroethene	0.98	Not Detected
1,2-Dichloropropane	0.98	Not Detected
cis-1,3-Dichloropropene	0.98	Not Detected
Toluene	0.98	1.7
trans-1,3-Dichloropropene	0.98	Not Detected
1,1,2-Trichloroethane	0.98	Not Detected
Tetrachloroethene	0.98	Not Detected
Ethylene Dibromide	0.98	Not Detected
Chlorobenzene	0.98	Not Detected
Ethyl Benzene	0.98	Not Detected
m,p-Xylene	0.98	Not Detected
o-Xylene	0.98	Not Detected
Styrene	0.98	Not Detected
1,1,2,2-Tetrachloroethane	0.98	Not Detected
1,3,5-Trimethylbenzene	0.98	Not Detected
1,2,4-Trimethylbenzene	0.98	Not Detected
1,3-Dichlorobenzene	0.98	Not Detected
1,4-Dichlorobenzene	0.98	Not Detected
Chlorotoluene	0.98	Not Detected
1,2-Dichlorobenzene	0.98	Not Detected
1,2,4-Trichlorobenzene	0.98	Not Detected
Hexachlorobutadiene	0.98	Not Detected
Propylene	3.9	Not Detected
1,3-Butadiene	3.9	Not Detected
Acetone	3.9	5.7
Carbon Disulfide	3.9	Not Detected
2-Propanol	3.9	Not Detected
trans-1,2-Dichloroethene	3.9	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #3 THUR 10763

ID#: 9806030-07A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060421	**Date of Collection:**	5/28/98
Dil. Factor:	1.96	**Date of Analysis:**	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	3.9	Not Detected
2-Butanone (Methyl Ethyl Ketone)	3.9	Not Detected
Hexane	3.9	Not Detected
Tetrahydrofuran	3.9	Not Detected
Cyclohexane	3.9	Not Detected
1,4-Dioxane	3.9	17
Bromodichloromethane	3.9	Not Detected
4-Methyl-2-pentanone	3.9	Not Detected
2-Hexanone	3.9	Not Detected
Dibromochloromethane	3.9	Not Detected
Bromoform	3.9	Not Detected
4-Ethyltoluene	3.9	Not Detected
Ethanol	3.9	Not Detected
Methyl tert-Butyl Ether	3.9	Not Detected
Heptane	3.9	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	109	70-130
Toluene-d8	106	70-130
4-Bromofluorobenzene	105	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #4 THUR 23888

ID#: 9806030-08A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060422	Date of Collection:	5/28/98
Dil. Factor:	2.01	Date of Analysis:	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	1.0	Not Detected
Freon 114	1.0	Not Detected
Chloromethane	1.0	1.1
Vinyl Chloride	1.0	Not Detected
Bromomethane	1.0	Not Detected
Chloroethane	1.0	Not Detected
Freon 11	1.0	Not Detected
1,1-Dichloroethene	1.0	Not Detected
Freon 113	1.0	Not Detected
Methylene Chloride	1.0	Not Detected
1,1-Dichloroethane	1.0	Not Detected
cis-1,2-Dichloroethene	1.0	Not Detected
Chloroform	1.0	Not Detected
1,1,1-Trichloroethane	1.0	Not Detected
Carbon Tetrachloride	1.0	Not Detected
Benzene	1.0	Not Detected
1,2-Dichloroethane	1.0	Not Detected
Trichloroethene	1.0	Not Detected
1,2-Dichloropropane	1.0	Not Detected
cis-1,3-Dichloropropene	1.0	Not Detected
Toluene	1.0	1.8
trans-1,3-Dichloropropene	1.0	Not Detected
1,1,2-Trichloroethane	1.0	Not Detected
Tetrachloroethene	1.0	Not Detected
Ethylene Dibromide	1.0	Not Detected
Chlorobenzene	1.0	Not Detected
Ethyl Benzene	1.0	Not Detected
m,p-Xylene	1.0	Not Detected
o-Xylene	1.0	Not Detected
Styrene	1.0	Not Detected
1,1,2,2-Tetrachloroethane	1.0	Not Detected
1,3,5-Trimethylbenzene	1.0	Not Detected
1,2,4-Trimethylbenzene	1.0	Not Detected
1,3-Dichlorobenzene	1.0	Not Detected
1,4-Dichlorobenzene	1.0	Not Detected
Chlorotoluene	1.0	Not Detected
1,2-Dichlorobenzene	1.0	Not Detected
1,2,4-Trichlorobenzene	1.0	Not Detected
Hexachlorobutadiene	1.0	Not Detected
Propylene	4.0	Not Detected
1,3-Butadiene	4.0	Not Detected
Acetone	4.0	5.4
Carbon Disulfide	4.0	Not Detected
2-Propanol	4.0	Not Detected
trans-1,2-Dichloroethene	4.0	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #4 THUR 23888

ID#: 9806030-08A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060422**	**Date of Collection: 5/28/98**
Dil. Factor:	**2.01**	**Date of Analysis: 6/ 4/98**

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	4.0	Not Detected
2-Butanone (Methyl Ethyl Ketone)	4.0	Not Detected
Hexane	4.0	Not Detected
Tetrahydrofuran	4.0	Not Detected
Cyclohexane	4.0	Not Detected
1,4-Dioxane	4.0	20
Bromodichloromethane	4.0	Not Detected
4-Methyl-2-pentanone	4.0	Not Detected
2-Hexanone	4.0	Not Detected
Dibromochloromethane	4.0	Not Detected
Bromoform	4.0	Not Detected
4-Ethyltoluene	4.0	Not Detected
Ethanol	4.0	Not Detected
Methyl tert-Butyl Ether	4.0	Not Detected
Heptane	4.0	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	107	70-130
Toluene-d8	106	70-130
4-Bromofluorobenzene	103	70-130

AIR TOXICS LTD.

SAMPLE NAME : pit THUR 487

ID#: 9806030-09A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060423	**Date of Collection:**	5/28/98
Dil. Factor:	2.01	**Date of Analysis:**	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	1.0	Not Detected
Freon 114	1.0	Not Detected
Chloromethane	1.0	1.7
Vinyl Chloride	1.0	Not Detected
Bromomethane	1.0	Not Detected
Chloroethane	1.0	Not Detected
Freon 11	1.0	Not Detected
1,1-Dichloroethene	1.0	Not Detected
Freon 113	1.0	Not Detected
Methylene Chloride	1.0	Not Detected
1,1-Dichloroethane	1.0	Not Detected
cis-1,2-Dichloroethene	1.0	Not Detected
Chloroform	1.0	Not Detected
1,1,1-Trichloroethane	1.0	Not Detected
Carbon Tetrachloride	1.0	Not Detected
Benzene	1.0	19
1,2-Dichloroethane	1.0	Not Detected
Trichloroethene	1.0	Not Detected
1,2-Dichloropropane	1.0	Not Detected
cis-1,3-Dichloropropene	1.0	Not Detected
Toluene	1.0	27
trans-1,3-Dichloropropene	1.0	Not Detected
1,1,2-Trichloroethane	1.0	Not Detected
Tetrachloroethene	1.0	Not Detected
Ethylene Dibromide	1.0	Not Detected
Chlorobenzene	1.0	Not Detected
Ethyl Benzene	1.0	39
m,p-Xylene	1.0	51
o-Xylene	1.0	21
Styrene	1.0	2.9
1,1,2,2-Tetrachloroethane	1.0	Not Detected
1,3,5-Trimethylbenzene	1.0	10
1,2,4-Trimethylbenzene	1.0	19
1,3-Dichlorobenzene	1.0	Not Detected
1,4-Dichlorobenzene	1.0	Not Detected
Chlorotoluene	1.0	Not Detected
1,2-Dichlorobenzene	1.0	Not Detected
1,2,4-Trichlorobenzene	1.0	Not Detected
Hexachlorobutadiene	1.0	Not Detected
Propylene	4.0	Not Detected
1,3-Butadiene	4.0	Not Detected
Acetone	4.0	8.7
Carbon Disulfide	4.0	Not Detected
2-Propanol	4.0	Not Detected
trans-1,2-Dichloroethene	4.0	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : pit THUR 487

ID#: 9806030-09A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060423	**Date of Collection:**	5/28/98
Dil. Factor:	2.01	**Date of Analysis:**	6/ 4/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	4.0	Not Detected
2-Butanone (Methyl Ethyl Ketone)	4.0	Not Detected
Hexane	4.0	Not Detected
Tetrahydrofuran	4.0	Not Detected
Cyclohexane	4.0	Not Detected
1,4-Dioxane	4.0	7.4
Bromodichloromethane	4.0	Not Detected
4-Methyl-2-pentanone	4.0	Not Detected
2-Hexanone	4.0	Not Detected
Dibromochloromethane	4.0	Not Detected
Bromoform	4.0	Not Detected
4-Ethyltoluene	4.0	17
Ethanol	4.0	Not Detected
Methyl tert-Butyl Ether	4.0	Not Detected
Heptane	4.0	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Benzofuran	271-89-6	91 %	7.2
Benzene, 1,2,3-trimethyl-	526-73-8	94 %	7.8
1H-Indene, 2,3-dihydro-	496-11-7	87 %	76
Benzene, 1-propynyl-	673-32-5	91 %	35
Benzene, (1-methyl-1-propenyl)-, (E)-	768-00-3	90 %	7.2
Benzofuran, 2-methyl-	4265-25-2	90 %	7.1
2,3-Dihydro-1-methylindene	27133-93-3	93 %	5.6
1H-Indene, 2,3-dihydro-2-methyl-	824-63-5	90 %	6.7
Benzene, (1-methyl-2-cyclopropen-1-yl)-	65051-83-4	86 %	5.1
Benzothiophene	11095-43-5	91 %	19
Naphthalene	91-20-3	93 %	290

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	112	70-130
Toluene-d8	108	70-130
4-Bromofluorobenzene	107	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #1 FRI 22513

ID#: 9806030-10A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060517	Date of Collection: 5/29/98
Dil. Factor:	1.91	Date of Analysis: 6/ 5/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.96	Not Detected
Freon 114	0.96	Not Detected
Chloromethane	0.96	1.1
Vinyl Chloride	0.96	Not Detected
Bromomethane	0.96	Not Detected
Chloroethane	0.96	Not Detected
Freon 11	0.96	Not Detected
1,1-Dichloroethene	0.96	Not Detected
Freon 113	0.96	Not Detected
Methylene Chloride	0.96	Not Detected
1,1-Dichloroethane	0.96	Not Detected
cis-1,2-Dichloroethene	0.96	Not Detected
Chloroform	0.96	Not Detected
1,1,1-Trichloroethane	0.96	Not Detected
Carbon Tetrachloride	0.96	Not Detected
Benzene	0.96	Not Detected
1,2-Dichloroethane	0.96	Not Detected
Trichloroethene	0.96	Not Detected
1,2-Dichloropropane	0.96	Not Detected
cis-1,3-Dichloropropene	0.96	Not Detected
Toluene	0.96	1.9
trans-1,3-Dichloropropene	0.96	Not Detected
1,1,2-Trichloroethane	0.96	Not Detected
Tetrachloroethene	0.96	Not Detected
Ethylene Dibromide	0.96	Not Detected
Chlorobenzene	0.96	Not Detected
Ethyl Benzene	0.96	Not Detected
m,p-Xylene	0.96	Not Detected
o-Xylene	0.96	Not Detected
Styrene	0.96	Not Detected
1,1,2,2-Tetrachloroethane	0.96	Not Detected
1,3,5-Trimethylbenzene	0.96	Not Detected
1,2,4-Trimethylbenzene	0.96	Not Detected
1,3-Dichlorobenzene	0.96	Not Detected
1,4-Dichlorobenzene	0.96	Not Detected
Chlorotoluene	0.96	Not Detected
1,2-Dichlorobenzene	0.96	Not Detected
1,2,4-Trichlorobenzene	0.96	Not Detected
Hexachlorobutadiene	0.96	Not Detected
Propylene	3.8	Not Detected
1,3-Butadiene	3.8	Not Detected
Acetone	3.8	6.5
Carbon Disulfide	3.8	Not Detected
2-Propanol	3.8	Not Detected
trans-1,2-Dichloroethene	3.8	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #1 FRI 22513

ID#: 9806030-10A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060517	Date of Collection:	5/29/98
Dil. Factor:	1.91	Date of Analysis:	6/ 5/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	3.8	Not Detected
2-Butanone (Methyl Ethyl Ketone)	3.8	Not Detected
Hexane	3.8	Not Detected
Tetrahydrofuran	3.8	Not Detected
Cyclohexane	3.8	Not Detected
1,4-Dioxane	3.8	7.8
Bromodichloromethane	3.8	Not Detected
4-Methyl-2-pentanone	3.8	Not Detected
2-Hexanone	3.8	Not Detected
Dibromochloromethane	3.8	Not Detected
Bromoform	3.8	Not Detected
4-Ethyltoluene	3.8	Not Detected
Ethanol	3.8	6.0
Methyl tert-Butyl Ether	3.8	Not Detected
Heptane	3.8	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Undecane, 2,6-dimethyl-	17301-23-4	Manual ID	3.9
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	104	70-130
Toluene-d8	109	70-130
4-Bromofluorobenzene	110	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #1 FRI 22513 Duplicate

ID#: 9806030-10AA

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060610	**Date of Collection:**	5/29/98
Dil. Factor:	2.25	**Date of Analysis:**	6/ 6/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	1.1	Not Detected
Freon 114	1.1	Not Detected
Chloromethane	1.1	1.4
Vinyl Chloride	1.1	Not Detected
Bromomethane	1.1	Not Detected
Chloroethane	1.1	Not Detected
Freon 11	1.1	Not Detected
1,1-Dichloroethene	1.1	Not Detected
Freon 113	1.1	Not Detected
Methylene Chloride	1.1	Not Detected
1,1-Dichloroethane	1.1	Not Detected
cis-1,2-Dichloroethene	1.1	Not Detected
Chloroform	1.1	Not Detected
1,1,1-Trichloroethane	1.1	Not Detected
Carbon Tetrachloride	1.1	Not Detected
Benzene	1.1	Not Detected
1,2-Dichloroethane	1.1	Not Detected
Trichloroethene	1.1	Not Detected
1,2-Dichloropropane	1.1	Not Detected
cis-1,3-Dichloropropene	1.1	Not Detected
Toluene	1.1	2.0
trans-1,3-Dichloropropene	1.1	Not Detected
1,1,2-Trichloroethane	1.1	Not Detected
Tetrachloroethene	1.1	Not Detected
Ethylene Dibromide	1.1	Not Detected
Chlorobenzene	1.1	Not Detected
Ethyl Benzene	1.1	Not Detected
m,p-Xylene	1.1	Not Detected
o-Xylene	1.1	Not Detected
Styrene	1.1	Not Detected
1,1,2,2-Tetrachloroethane	1.1	Not Detected
1,3,5-Trimethylbenzene	1.1	Not Detected
1,2,4-Trimethylbenzene	1.1	Not Detected
1,3-Dichlorobenzene	1.1	Not Detected
1,4-Dichlorobenzene	1.1	Not Detected
Chlorotoluene	1.1	Not Detected
1,2-Dichlorobenzene	1.1	Not Detected
1,2,4-Trichlorobenzene	1.1	Not Detected
Hexachlorobutadiene	1.1	Not Detected
Propylene	4.5	Not Detected
1,3-Butadiene	4.5	Not Detected
Acetone	4.5	6.4
Carbon Disulfide	4.5	Not Detected
2-Propanol	4.5	Not Detected
trans-1,2-Dichloroethene	4.5	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #1 FRI 22513 Duplicate

ID#: 9806030-10AA

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060610	Date of Collection:	5/29/98
Dil. Factor:	2.25	Date of Analysis:	6/ 6/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	4.5	Not Detected
2-Butanone (Methyl Ethyl Ketone)	4.5	Not Detected
Hexane	4.5	Not Detected
Tetrahydrofuran	4.5	Not Detected
Cyclohexane	4.5	Not Detected
1,4-Dioxane	4.5	8.0
Bromodichloromethane	4.5	Not Detected
4-Methyl-2-pentanone	4.5	Not Detected
2-Hexanone	4.5	Not Detected
Dibromochloromethane	4.5	Not Detected
Bromoform	4.5	Not Detected
4-Ethyltoluene	4.5	Not Detected
Ethanol	4.5	6.7
Methyl tert-Butyl Ether	4.5	Not Detected
Heptane	4.5	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Undecane, 3,6-dimethyl-	17301-28-9	Manual ID	6.1
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	107	70-130
Toluene-d8	107	70-130
4-Bromofluorobenzene	101	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #2 FRI 2993

ID#: 9806030-11A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060518**	**Date of Collection:**	**5/29/98**
Dil. Factor:	**1.68**	**Date of Analysis:**	**6/ 5/98**

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.84	0.89
Freon 114	0.84	Not Detected
Chloromethane	0.84	0.95
Vinyl Chloride	0.84	Not Detected
Bromomethane	0.84	Not Detected
Chloroethane	0.84	Not Detected
Freon 11	0.84	Not Detected
1,1-Dichloroethene	0.84	Not Detected
Freon 113	0.84	Not Detected
Methylene Chloride	0.84	Not Detected
1,1-Dichloroethane	0.84	Not Detected
cis-1,2-Dichloroethene	0.84	Not Detected
Chloroform	0.84	Not Detected
1,1,1-Trichloroethane	0.84	Not Detected
Carbon Tetrachloride	0.84	Not Detected
Benzene	0.84	Not Detected
1,2-Dichloroethane	0.84	Not Detected
Trichloroethene	0.84	Not Detected
1,2-Dichloropropane	0.84	Not Detected
cis-1,3-Dichloropropene	0.84	Not Detected
Toluene	0.84	2.3
trans-1,3-Dichloropropene	0.84	Not Detected
1,1,2-Trichloroethane	0.84	Not Detected
Tetrachloroethene	0.84	Not Detected
Ethylene Dibromide	0.84	Not Detected
Chlorobenzene	0.84	Not Detected
Ethyl Benzene	0.84	Not Detected
m,p-Xylene	0.84	0.91
o-Xylene	0.84	Not Detected
Styrene	0.84	Not Detected
1,1,2,2-Tetrachloroethane	0.84	Not Detected
1,3,5-Trimethylbenzene	0.84	Not Detected
1,2,4-Trimethylbenzene	0.84	Not Detected
1,3-Dichlorobenzene	0.84	Not Detected
1,4-Dichlorobenzene	0.84	Not Detected
Chlorotoluene	0.84	Not Detected
1,2-Dichlorobenzene	0.84	Not Detected
1,2,4-Trichlorobenzene	0.84	Not Detected
Hexachlorobutadiene	0.84	Not Detected
Propylene	3.4	3.4
1,3-Butadiene	3.4	Not Detected
Acetone	3.4	6.3
Carbon Disulfide	3.4	Not Detected
2-Propanol	3.4	Not Detected
trans-1,2-Dichloroethene	3.4	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #2 FRI 2993

ID#: 9806030-11A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060518	Date of Collection:	5/29/98
Dil. Factor:	1.68	Date of Analysis:	6/ 5/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	3.4	Not Detected
2-Butanone (Methyl Ethyl Ketone)	3.4	Not Detected
Hexane	3.4	Not Detected
Tetrahydrofuran	3.4	Not Detected
Cyclohexane	3.4	Not Detected
1,4-Dioxane	3.4	Not Detected
Bromodichloromethane	3.4	Not Detected
4-Methyl-2-pentanone	3.4	Not Detected
2-Hexanone	3.4	Not Detected
Dibromochloromethane	3.4	Not Detected
Bromoform	3.4	Not Detected
4-Ethyltoluene	3.4	Not Detected
Ethanol	3.4	5.3
Methyl tert-Butyl Ether	3.4	Not Detected
Heptane	3.4	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Heptane, 2,2,4-trimethyl-	14720-74-2	Manual ID	6.6
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	114	70-130
Toluene-d8	108	70-130
4-Bromofluorobenzene	104	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #3 FRI 96115

ID#: 9806030-12A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060519**	**Date of Collection:**	**5/29/98**
Dil. Factor:	**1.96**	**Date of Analysis:**	**6/ 5/98**

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.98	Not Detected
Freon 114	0.98	Not Detected
Chloromethane	0.98	1.2
Vinyl Chloride	0.98	Not Detected
Bromomethane	0.98	Not Detected
Chloroethane	0.98	Not Detected
Freon 11	0.98	Not Detected
1,1-Dichloroethene	0.98	Not Detected
Freon 113	0.98	Not Detected
Methylene Chloride	0.98	Not Detected
1,1-Dichloroethane	0.98	Not Detected
cis-1,2-Dichloroethene	0.98	Not Detected
Chloroform	0.98	Not Detected
1,1,1-Trichloroethane	0.98	Not Detected
Carbon Tetrachloride	0.98	Not Detected
Benzene	0.98	1.5
1,2-Dichloroethane	0.98	Not Detected
Trichloroethene	0.98	Not Detected
1,2-Dichloropropane	0.98	Not Detected
cis-1,3-Dichloropropene	0.98	Not Detected
Toluene	0.98	1.7
trans-1,3-Dichloropropene	0.98	Not Detected
1,1,2-Trichloroethane	0.98	Not Detected
Tetrachloroethene	0.98	Not Detected
Ethylene Dibromide	0.98	Not Detected
Chlorobenzene	0.98	1.1
Ethyl Benzene	0.98	Not Detected
m,p-Xylene	0.98	Not Detected
o-Xylene	0.98	Not Detected
Styrene	0.98	Not Detected
1,1,2,2-Tetrachloroethane	0.98	Not Detected
1,3,5-Trimethylbenzene	0.98	Not Detected
1,2,4-Trimethylbenzene	0.98	Not Detected
1,3-Dichlorobenzene	0.98	Not Detected
1,4-Dichlorobenzene	0.98	Not Detected
Chlorotoluene	0.98	Not Detected
1,2-Dichlorobenzene	0.98	Not Detected
1,2,4-Trichlorobenzene	0.98	Not Detected
Hexachlorobutadiene	0.98	Not Detected
Propylene	3.9	Not Detected
1,3-Butadiene	3.9	Not Detected
Acetone	3.9	8.3
Carbon Disulfide	3.9	Not Detected
2-Propanol	3.9	Not Detected
trans-1,2-Dichloroethene	3.9	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #3 FRI 96115

ID#: 9806030-12A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060519	Date of Collection:	5/29/98
Dil. Factor:	1.96	Date of Analysis:	6/5/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	3.9	Not Detected
2-Butanone (Methyl Ethyl Ketone)	3.9	Not Detected
Hexane	3.9	Not Detected
Tetrahydrofuran	3.9	Not Detected
Cyclohexane	3.9	Not Detected
1,4-Dioxane	3.9	Not Detected
Bromodichloromethane	3.9	Not Detected
4-Methyl-2-pentanone	3.9	Not Detected
2-Hexanone	3.9	Not Detected
Dibromochloromethane	3.9	Not Detected
Bromoform	3.9	Not Detected
4-Ethyltoluene	3.9	Not Detected
Ethanol	3.9	8.4
Methyl tert-Butyl Ether	3.9	Not Detected
Heptane	3.9	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	106	70-130
Toluene-d8	104	70-130
4-Bromofluorobenzene	102	70-130

AIR TOXICS LTD.

SAMPLE NAME : Site #4 FRI 12704

ID#: 9806030-13A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060609	Date of Collection:	5/29/98
Dil. Factor:	2.17	Date of Analysis:	6/ 6/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	1.1	Not Detected
Freon 114	1.1	Not Detected
Chloromethane	1.1	1.7
Vinyl Chloride	1.1	Not Detected
Bromomethane	1.1	Not Detected
Chloroethane	1.1	Not Detected
Freon 11	1.1	Not Detected
1,1-Dichloroethene	1.1	Not Detected
Freon 113	1.1	Not Detected
Methylene Chloride	1.1	Not Detected
1,1-Dichloroethane	1.1	Not Detected
cis-1,2-Dichloroethene	1.1	Not Detected
Chloroform	1.1	Not Detected
1,1,1-Trichloroethane	1.1	Not Detected
Carbon Tetrachloride	1.1	Not Detected
Benzene	1.1	3.0
1,2-Dichloroethane	1.1	Not Detected
Trichloroethene	1.1	Not Detected
1,2-Dichloropropane	1.1	Not Detected
cis-1,3-Dichloropropene	1.1	Not Detected
Toluene	1.1	1.6
trans-1,3-Dichloropropene	1.1	Not Detected
1,1,2-Trichloroethane	1.1	Not Detected
Tetrachloroethene	1.1	Not Detected
Ethylene Dibromide	1.1	Not Detected
Chlorobenzene	1.1	Not Detected
Ethyl Benzene	1.1	Not Detected
m,p-Xylene	1.1	Not Detected
o-Xylene	1.1	Not Detected
Styrene	1.1	Not Detected
1,1,2,2-Tetrachloroethane	1.1	Not Detected
1,3,5-Trimethylbenzene	1.1	Not Detected
1,2,4-Trimethylbenzene	1.1	Not Detected
1,3-Dichlorobenzene	1.1	Not Detected
1,4-Dichlorobenzene	1.1	Not Detected
Chlorotoluene	1.1	Not Detected
1,2-Dichlorobenzene	1.1	Not Detected
1,2,4-Trichlorobenzene	1.1	Not Detected
Hexachlorobutadiene	1.1	Not Detected
Propylene	4.3	Not Detected
1,3-Butadiene	4.3	Not Detected
Acetone	4.3	11
Carbon Disulfide	4.3	Not Detected
2-Propanol	4.3	Not Detected
trans-1,2-Dichloroethene	4.3	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Site #4 FRI 12704

ID#: 9806030-13A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060609	Date of Collection:	5/29/98
Dil. Factor:	2.17	Date of Analysis:	6/ 6/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	4.3	Not Detected
2-Butanone (Methyl Ethyl Ketone)	4.3	Not Detected
Hexane	4.3	Not Detected
Tetrahydrofuran	4.3	Not Detected
Cyclohexane	4.3	Not Detected
1,4-Dioxane	4.3	Not Detected
Bromodichloromethane	4.3	Not Detected
4-Methyl-2-pentanone	4.3	Not Detected
2-Hexanone	4.3	Not Detected
Dibromochloromethane	4.3	Not Detected
Bromoform	4.3	Not Detected
4-Ethyltoluene	4.3	Not Detected
Ethanol	4.3	Not Detected
Methyl tert-Butyl Ether	4.3	Not Detected
Heptane	4.3	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Benzene, (1-methylethenyl)-	98-83-9	Manual ID	6.2
Naphthalene	91-20-3	NA	Not Detected

Container Type: 6 Liter Summa Canister

Surrogates	% Recovery	Method Limits
Octafluorotoluene	111	70-130
Toluene-d8	108	70-130
4-Bromofluorobenzene	102	70-130

AIR TOXICS LTD.

SAMPLE NAME : Method Spike

ID#: 9806030-14A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060603**	**Date of Collection: NA**	
Dil. Factor:	**1.00**	**Date of Analysis: 6/ 6/98**	

Compound	Rpt. Limit (ppbv)	% Recovery
Freon 12	0.50	125
Freon 114	0.50	111
Chloromethane	0.50	186 Q
Vinyl Chloride	0.50	127
Bromomethane	0.50	112
Chloroethane	0.50	120
Freon 11	0.50	123
1,1-Dichloroethene	0.50	119
Freon 113	0.50	111
Methylene Chloride	0.50	127
1,1-Dichloroethane	0.50	118
cis-1,2-Dichloroethene	0.50	117
Chloroform	0.50	116
1,1,1-Trichloroethane	0.50	112
Carbon Tetrachloride	0.50	113
Benzene	0.50	122
1,2-Dichloroethane	0.50	117
Trichloroethene	0.50	110
1,2-Dichloropropane	0.50	116
cis-1,3-Dichloropropene	0.50	100
Toluene	0.50	107
trans-1,3-Dichloropropene	0.50	86
1,1,2-Trichloroethane	0.50	110
Tetrachloroethene	0.50	105
Ethylene Dibromide	0.50	106
Chlorobenzene	0.50	108
Ethyl Benzene	0.50	113
m,p-Xylene	0.50	112
o-Xylene	0.50	108
Styrene	0.50	96
1,1,2,2-Tetrachloroethane	0.50	113
1,3,5-Trimethylbenzene	0.50	104
1,2,4-Trimethylbenzene	0.50	99
1,3-Dichlorobenzene	0.50	97
1,4-Dichlorobenzene	0.50	98
Chlorotoluene	0.50	58 Q
1,2-Dichlorobenzene	0.50	98
1,2,4-Trichlorobenzene	0.50	80
Hexachlorobutadiene	0.50	94
Propylene	2.0	149 Q
1,3-Butadiene	2.0	122
Acetone	2.0	110
Carbon Disulfide	2.0	110
2-Propanol	2.0	101
trans-1,2-Dichloroethene	2.0	116

AIR TOXICS LTD.

SAMPLE NAME : Method Spike

ID#: 9806030-14A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060603**	**Date of Collection: NA**	
Dil. Factor:	**1.00**	**Date of Analysis: 6/ 6/98**	

Compound	Rpt. Limit (ppbv)	% Recovery
Vinyl Acetate	2.0	114
2-Butanone (Methyl Ethyl Ketone)	2.0	113
Hexane	2.0	124
Tetrahydrofuran	2.0	118
Cyclohexane	2.0	126
1,4-Dioxane	2.0	103
Bromodichloromethane	2.0	111
4-Methyl-2-pentanone	2.0	104
2-Hexanone	2.0	66
Dibromochloromethane	2.0	109
Bromoform	2.0	106
4-Ethyltoluene	2.0	109
Ethanol	2.0	114
Methyl tert-Butyl Ether	2.0	99
Heptane	2.0	122

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Spiked

Q = Exceeds Quality Control limits.

Container Type: NA

Surrogates	% Recovery	Method Limits
Octafluorotoluene	110	70-130
Toluene-d8	106	70-130
4-Bromofluorobenzene	104	70-130

AIR TOXICS LTD.

SAMPLE NAME : Lab Blank

ID#: 9806030-15A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060404**	**Date of Collection: NA**	
Dil. Factor:	**1.00**	**Date of Analysis: 6/ 4/98**	

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.50	Not Detected
Freon 114	0.50	Not Detected
Chloromethane	0.50	Not Detected
Vinyl Chloride	0.50	Not Detected
Bromomethane	0.50	Not Detected
Chloroethane	0.50	Not Detected
Freon 11	0.50	Not Detected
1,1-Dichloroethene	0.50	Not Detected
Freon 113	0.50	Not Detected
Methylene Chloride	0.50	0.59
1,1-Dichloroethane	0.50	Not Detected
cis-1,2-Dichloroethene	0.50	Not Detected
Chloroform	0.50	Not Detected
1,1,1-Trichloroethane	0.50	Not Detected
Carbon Tetrachloride	0.50	Not Detected
Benzene	0.50	Not Detected
1,2-Dichloroethane	0.50	Not Detected
Trichloroethene	0.50	Not Detected
1,2-Dichloropropane	0.50	Not Detected
cis-1,3-Dichloropropene	0.50	Not Detected
Toluene	0.50	Not Detected
trans-1,3-Dichloropropene	0.50	Not Detected
1,1,2-Trichloroethane	0.50	Not Detected
Tetrachloroethene	0.50	Not Detected
Ethylene Dibromide	0.50	Not Detected
Chlorobenzene	0.50	Not Detected
Ethyl Benzene	0.50	Not Detected
m,p-Xylene	0.50	Not Detected
o-Xylene	0.50	Not Detected
Styrene	0.50	Not Detected
1,1,2,2-Tetrachloroethane	0.50	Not Detected
1,3,5-Trimethylbenzene	0.50	Not Detected
1,2,4-Trimethylbenzene	0.50	Not Detected
1,3-Dichlorobenzene	0.50	Not Detected
1,4-Dichlorobenzene	0.50	Not Detected
Chlorotoluene	0.50	Not Detected
1,2-Dichlorobenzene	0.50	Not Detected
1,2,4-Trichlorobenzene	0.50	Not Detected
Hexachlorobutadiene	0.50	Not Detected
Propylene	2.0	Not Detected
1,3-Butadiene	2.0	Not Detected
Acetone	2.0	Not Detected
Carbon Disulfide	2.0	Not Detected
2-Propanol	2.0	Not Detected
trans-1,2-Dichloroethene	2.0	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Lab Blank

ID#: 9806030-15A

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060404**	**Date of Collection: NA**	
Dil. Factor:	**1.00**	**Date of Analysis: 6/ 4/98**	

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	2.0	Not Detected
2-Butanone (Methyl Ethyl Ketone)	2.0	Not Detected
Hexane	2.0	Not Detected
Tetrahydrofuran	2.0	Not Detected
Cyclohexane	2.0	Not Detected
1,4-Dioxane	2.0	Not Detected
Bromodichloromethane	2.0	Not Detected
4-Methyl-2-pentanone	2.0	Not Detected
2-Hexanone	2.0	Not Detected
Dibromochloromethane	2.0	Not Detected
Bromoform	2.0	Not Detected
4-Ethyltoluene	2.0	Not Detected
Ethanol	2.0	Not Detected
Methyl tert-Butyl Ether	2.0	Not Detected
Heptane	2.0	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: NA

Surrogates	% Recovery	Method Limits
Octafluorotoluene	110	70-130
Toluene-d8	109	70-130
4-Bromofluorobenzene	106	70-130

AIR TOXICS LTD.

SAMPLE NAME : Lab Blank

ID#: 9806030-15B

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060509	**Date of Collection:**	NA
Dil. Factor:	1.00	**Date of Analysis:**	6/ 5/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.50	Not Detected
Freon 114	0.50	Not Detected
Chloromethane	0.50	Not Detected
Vinyl Chloride	0.50	Not Detected
Bromomethane	0.50	Not Detected
Chloroethane	0.50	Not Detected
Freon 11	0.50	Not Detected
1,1-Dichloroethene	0.50	Not Detected
Freon 113	0.50	Not Detected
Methylene Chloride	0.50	1.0
1,1-Dichloroethane	0.50	Not Detected
cis-1,2-Dichloroethene	0.50	Not Detected
Chloroform	0.50	Not Detected
1,1,1-Trichloroethane	0.50	Not Detected
Carbon Tetrachloride	0.50	Not Detected
Benzene	0.50	Not Detected
1,2-Dichloroethane	0.50	Not Detected
Trichloroethene	0.50	Not Detected
1,2-Dichloropropane	0.50	Not Detected
cis-1,3-Dichloropropene	0.50	Not Detected
Toluene	0.50	Not Detected
trans-1,3-Dichloropropene	0.50	Not Detected
1,1,2-Trichloroethane	0.50	Not Detected
Tetrachloroethene	0.50	Not Detected
Ethylene Dibromide	0.50	Not Detected
Chlorobenzene	0.50	Not Detected
Ethyl Benzene	0.50	Not Detected
m,p-Xylene	0.50	Not Detected
o-Xylene	0.50	Not Detected
Styrene	0.50	Not Detected
1,1,2,2-Tetrachloroethane	0.50	Not Detected
1,3,5-Trimethylbenzene	0.50	Not Detected
1,2,4-Trimethylbenzene	0.50	Not Detected
1,3-Dichlorobenzene	0.50	Not Detected
1,4-Dichlorobenzene	0.50	Not Detected
Chlorotoluene	0.50	Not Detected
1,2-Dichlorobenzene	0.50	Not Detected
1,2,4-Trichlorobenzene	0.50	Not Detected
Hexachlorobutadiene	0.50	Not Detected
Propylene	2.0	Not Detected
1,3-Butadiene	2.0	Not Detected
Acetone	2.0	Not Detected
Carbon Disulfide	2.0	Not Detected
2-Propanol	2.0	Not Detected
trans-1,2-Dichloroethene	2.0	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Lab Blank

ID#: 9806030-15B

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060509**	**Date of Collection: NA**	
Dil. Factor:	**1.00**	**Date of Analysis: 6/ 5/98**	

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	2.0	Not Detected
2-Butanone (Methyl Ethyl Ketone)	2.0	Not Detected
Hexane	2.0	Not Detected
Tetrahydrofuran	2.0	Not Detected
Cyclohexane	2.0	Not Detected
1,4-Dioxane	2.0	Not Detected
Bromodichloromethane	2.0	Not Detected
4-Methyl-2-pentanone	2.0	Not Detected
2-Hexanone	2.0	Not Detected
Dibromochloromethane	2.0	Not Detected
Bromoform	2.0	Not Detected
4-Ethyltoluene	2.0	Not Detected
Ethanol	2.0	Not Detected
Methyl tert-Butyl Ether	2.0	Not Detected
Heptane	2.0	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: NA

Surrogates	% Recovery	Method Limits
Octafluorotoluene	103	70-130
Toluene-d8	107	70-130
4-Bromofluorobenzene	107	70-130

AIR TOXICS LTD.

SAMPLE NAME : Lab Blank

ID#: 9806030-15C

EPA METHOD TO-14 GC/MS Full Scan

File Name:	**1060606**	**Date of Collection:**	**NA**
Dil. Factor:	**1.00**	**Date of Analysis:**	**6/ 6/98**

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Freon 12	0.50	Not Detected
Freon 114	0.50	Not Detected
Chloromethane	0.50	Not Detected
Vinyl Chloride	0.50	Not Detected
Bromomethane	0.50	Not Detected
Chloroethane	0.50	Not Detected
Freon 11	0.50	Not Detected
1,1-Dichloroethene	0.50	Not Detected
Freon 113	0.50	Not Detected
Methylene Chloride	0.50	Not Detected
1,1-Dichloroethane	0.50	Not Detected
cis-1,2-Dichloroethene	0.50	Not Detected
Chloroform	0.50	Not Detected
1,1,1-Trichloroethane	0.50	Not Detected
Carbon Tetrachloride	0.50	Not Detected
Benzene	0.50	Not Detected
1,2-Dichloroethane	0.50	Not Detected
Trichloroethene	0.50	Not Detected
1,2-Dichloropropane	0.50	Not Detected
cis-1,3-Dichloropropene	0.50	Not Detected
Toluene	0.50	Not Detected
trans-1,3-Dichloropropene	0.50	Not Detected
1,1,2-Trichloroethane	0.50	Not Detected
Tetrachloroethene	0.50	Not Detected
Ethylene Dibromide	0.50	Not Detected
Chlorobenzene	0.50	Not Detected
Ethy Benzene	0.50	Not Detected
m,p-Xylene	0.50	Not Detected
o-Xylene	0.50	Not Detected
Styrene	0.50	Not Detected
1,1,2,2-Tetrachloroethane	0.50	Not Detected
1,3,5-Trimethylbenzene	0.50	Not Detected
1,2,4-Trimethylbenzene	0.50	Not Detected
1,3-Dichlorobenzene	0.50	Not Detected
1,4-Dichlorobenzene	0.50	Not Detected
Chlorotoluene	0.50	Not Detected
1,2-Dichlorobenzene	0.50	Not Detected
1,2,4-Trichlorobenzene	0.50	Not Detected
Hexachlorobutadiene	0.50	Not Detected
Propylene	2.0	Not Detected
1,3-Butadiene	2.0	Not Detected
Acetone	2.0	Not Detected
Carbon Disulfide	2.0	Not Detected
2-Propanol	2.0	Not Detected
trans-1,2-Dichloroethene	2.0	Not Detected

AIR TOXICS LTD.

SAMPLE NAME : Lab Blank

ID#: 9806030-15C

EPA METHOD TO-14 GC/MS Full Scan

File Name:	1060606	Date of Collection:	NA
Dil. Factor:	1.00	Date of Analysis:	6/ 6/98

Compound	Rpt. Limit (ppbv)	Amount (ppbv)
Vinyl Acetate	2.0	Not Detected
2-Butanone (Methyl Ethyl Ketone)	2.0	Not Detected
Hexane	2.0	Not Detected
Tetrahydrofuran	2.0	Not Detected
Cyclohexane	2.0	Not Detected
1,4-Dioxane	2.0	Not Detected
Bromodichloromethane	2.0	Not Detected
4-Methyl-2-pentanone	2.0	Not Detected
2-Hexanone	2.0	Not Detected
Dibromochloromethane	2.0	Not Detected
Bromoform	2.0	Not Detected
4-Ethyltoluene	2.0	Not Detected
Ethanol	2.0	Not Detected
Methyl tert-Butyl Ether	2.0	Not Detected
Heptane	2.0	Not Detected

TENTATIVELY IDENTIFIED COMPOUNDS

Compound	CAS Number	Match Quality	Amount (ppbv)
Naphthalene	91-20-3	NA	Not Detected

Container Type: NA

Surrogates	% Recovery	Method Limits
Octafluorotoluene	111	70-130
Toluene-d8	106	70-130
4-Bromofluorobenzene	103	70-130

AIR TOXICS LTD.
AN ENVIRONMENTAL ANALYTICAL LABORATORY

180 BLUE RAVINE ROAD, SUITE B
FOLSOM, CA 95630-4719
(916) 985-1000 FAX: (916) 985-1020

CHAIN-OF-CUSTODY RECORD

№ [illegible]7

Contact Person: KRIS SIMONIAN
Company: DARENZO + ASSOC
Address: 39201 SCHOOLCRAFT RD-10 City: LIVONIA State: MI Zip: 48150
Phone: 313-464-3880 FAX: 313-464-4368
Collected By: Signature: K. Simonian

Project info:
P.O. #: 21978
Project #: 9804002
Project Name: Hollx Assoc

Turn Around Time:
☑ Normal
☐ Rush _____ Specify

Lab I.D.	Field Sample I.D.	Date & Time	Analyses Requested	Canister Pressure / Vacuum Initial	Final	Receipt
01A	Site #1 WED 769	5-27-98	TO-14 (24-Hour Sample)	29	9.2	10 [illegible]
02A	Site #2 WED 12937	5-27-98	TO-14 (24-Hour Sample)	28.2	11.5	11.5 [illegible]
03A	Site #3 WED 20951	5-27-98	TO-14 (24-Hour Sample)	28.0	7.9	8.5 [illegible]
04A	Site #4 WED 12717	5-27-98	TO-14 (24-Hour Sample)	29.2	9.8	10.5 [illegible]
05A	Site #1 1HOUR 12943	5-28-98	TO-14 (1-Hour Sample)	30	10.2	9.0 [illegible]
06A	Site #2 1HOUR 10782	5-28-98	TO-14 (1 Hour Sample)	30	11	8.5 [illegible]
07A	Site #3 1HOUR 10763	5-28-98	TO-14 (1 Hour Sample)	30	11.3	9.5 [illegible]
08A	Site #4 1HOUR 23844	5-28-98	TO-14 (1 Hour Sample)	30	11.5	10.0 [illegible]
09A	pit 1HOUR 487	5-28-98	TO-14 (1 Hour Sample)	30	12.0	10.0 [illegible]

Relinquished By: (Signature) Date/Time 6-1-98 Print Name KRIS SIMONIAN
Relinquished By: (Signature) Date/Time
Received By: (Signature) Date/Time
Relinquished By: (Signature) Date/Time
Received By: (Signature) Date/Time Alex Atlanta Air 6/2/98 1010

Notes:

Lab Use Only	Shipper Name	Air Bill #	Opened By:	Date/Time	Temp. (°C)	Condition	Custody Seals Intact?	Work Order #
	Fed-x	8077898313	TS	6/2/98 1010	Ambient	Good	Yes No None N/A	9806030

Form 1293 rev. 06

AIR TOXICS LTD.
AN ENVIRONMENTAL ANALYTICAL LABORATORY

180 BLUE RAVINE ROAD, SUITE B
FOLSOM, CA 95630-4719
(916) 985-1000 FAX: (916) 985-1020

CHAIN-OF-CUSTODY RECORD

Nº 15[illegible]6

Contact Person: KRIS SIMANIAN
Company: DEMENZO & ASSOC
Address: 39701 SCHOOLCRAFT B-10 City: LIVONIA State: MI Zip: 48150
Phone: 313-464-3880 FAX: 313-464-4368
Collected By: Signature: K. Simanian

Project info:
P.O. #: 21978
Project #: 9804002
Project Name: Hull & Assoc

Turn Around Time:
☑ Normal
☐ Rush _____ Specify

Lab I.D.	Field Sample I.D.	Date & Time	Analyses Requested	Canister Pressure / Vacuum Initial	Final	Receipt
10A	Site #1 FRI 22513	5-29-98	TO-14 (24 Hour Sample)	29.5	10.5	9.0"Hg
11A	Site #2 FRI 2993	5-29-98	TO-14 (24 Hour Sample)	28.5	5.5	6.0"Hg
12A	Site #3 FRI 96115	5-29-98	TO-14 (24 Hour Sample)	29	9.5	9.5"Hg
13A	Site #4 FRI 12704	5-29-98	TO-14 (24 Hour Sample)	28.5	7.5	7.5"Hg

Relinquished By: (Signature) Date/Time 6-1-98 Print Name: KRIS SIMANIAN
Relinquished By: (Signature) Date/Time
Received By: (Signature) Date/Time
Relinquished By: (Signature) Date/Time
Received By: (Signature) Date/Time [illegible] ATL 6/2/98 1010

Notes:

Lab Use Only	Shipper Name	Air Bill #	Opened By:	Date/Time	Temp. (°C)	Condition	Custody Seals Intact?	Work Order #
	Fed-X	8035549323 13	TS	6/2/98 1010	Amb	Good	Yes No None N/A	9806030

Form 1293 rev 06

TRIANGLE LABS

CASE NARRATIVE

Analysis of Samples for the Presence of

Semivolatile Analytes by

High-Resolution Gas Chromatography / Low-Resolution Mass Spectrometry

METHOD 8270A Rev. 1 (7/92)

Date :	June 12, 1998
Client ID :	Derenzo and Associates, Inc.
TLI Project Number :	45869

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TRIANGLE LABORATORIES, INC. **June 12, 1998**
CASE NARRATIVE **45869**

Objective: Analysis of twelve Sandwich PUF samples and a PUF filter blank for a client-specified list of polycyclic aromatic hydrocarbons (naphthalene, fluorene, phenanthrene, anthracene, fluoranthene, and benzo(a)pyrene), using Method 8270A.

Method:

Twelve Sandwich PUF samples and a PUF filter blank were received by Triangle Laboratories, Inc. on ice at 9°C and in good condition on June 2, 1998. The samples were stored in a cooler at 4°C prior to extraction. The PUF's used for sampling were pre-spiked with 100 micrograms (ug) of terphenyl-d_{14} prior to sampling. Prior to extraction, each sample was spiked with 100 ug of the surrogate standards pyrene-d_{10} and anthracene-d_{10}. They were then Soxhlet extracted with ethyl ether/hexane for 16 hours. The extracts were brought to a final volume of 1.0 mL for the semivolatile analysis. The analysis is based on the guidelines of Method 8270A Rev.1 (7/92).

The internal standards, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, and perylene-d_{12} were added to the extracts such that the final internal standard concentration was 40 ug/mL immediately prior to analysis by GC/MS.

The GC/MS analysis conditions are listed below:

GC Conditions:

Column:	J&W DB5-625, 30m x .32mm x 1μm
Program:	35C, ramp at 12C/min to 285C, hold for 2 min. ramp at 8.5C/min to 315 C, hold for 6.5 min.
Carrier Gas:	Helium

MS Conditions:

Instrument:	HP MSD, Chemsystem and Target data systems
Scan:	35-550 amu at 1.67 scan/sec
Interface:	Capillary, Injector: 250C, Detector: 275C

Report:

Enclosed with the case narrative are the sample identification index, project summary sheets, client and TLI chain of custody sheets, wet laboratory extraction information sheets, GC/MS tracking forms, and analytical run logs. The sample identification index correlates the client sample name, TLI sample number and the analytical file name for the each sample. The project summary sheets list the amounts of analytes detected in gray and list the estimated detection limits in parentheses for analytes which were not detected.

The data are reported as quantitation reports, chromatograms, interim reports, and spectra of detected target analytes. The quantitation report header lists the TLI project number, analysis method, instrument sample file name, and client sample name. The client project number, TLI sample number, calibration file, dilution factor, and date received, extracted, and analyzed are also listed in the quantitation report header. The response factors used for all calculations are from the

continuing calibration listed in the header. All initial and continuing calibration data are located in the back of the data package. The amount reported for each target analyte detected in the samples is reported in total ug. The retention time (RT) will be listed for all internal standards and analytes which are detected. If a target analyte is not detected, it will be flagged with a "U" and a detection limit will be listed. Estimated detection limits are calculated using an area of 10,000 for all analytes which were not found in the samples. The estimated detection limits reported are the average detection limits achievable over time on an instrument type. The actual detection limit for a given compound on a given day may vary from the estimate reported. The quantitation limit for all analytes is half of the low point of the initial calibration adjusted for dilution when appropriate. Below this point the calibration cannot be considered to be linear. Any amounts reported at a level below the quantitation limit will be flagged with a "J" and should be considered estimated. If a target analyte is found at a level exceeding the upper calibration limit, it will be flagged with an "E" and should also be considered estimated. Any analytes flagged with a "B" on the sample topsheets were detected in the associated laboratory blank. All target analytes are quantitated against the internal standard preceding them on the target analyte list. Surrogate standards are quantitated against the internal standard with the matching internal standard reference number. For example, anthracene-d_{10} has 4 in the IS Ref column and would be quantitated against the internal standard which has IS4 listed in the flag column. If an internal standard area is above or below the quality control limits as defined by the continuing calibration, it will be flagged with "High" or "Low" in the flag column.

Immediately following the quantitation report are two pages which comprise the total ion chromatograms. Labeled internal and surrogate standards present in the sample have their identifications and retention time printed above their peak on the chromatogram. The chromatogram followed by the interim report. On the interim report a $ is indicative of a surrogate standard and a * represents an internal standard. The interim report from the instrument is followed by the target spectra of detected compounds. Four spectral plots are included for each compound: a raw spectrum of the peak, a background subtracted version of the same spectrum, a library spectrum of the compound, and a plot showing the percent difference between the library spectrum and the background subtracted spectrum. Extracted ion current profiles are plotted on the right-hand side of the page showing the quantitation mass and one or two other prominent ions known to be present in target compound as they appear in the sample peak.

Results:

There were discrepancies between the sample identifications listed on the client chain of custody and those listed on the samples which arrived, for samples SITE#3 5-29-98 24hour (given as Site#3 5-27-98 fri24hour on the sample label) and SITE#4 5-29-98 24hour (given as 24 HOUR on the sample label). The identifications on the client chain of custody were used on the topsheet reports and project summary.

All samples were extracted and analyzed within the Method 8270 holding times.

Please note that naphthalene and phenanthrene were found at levels below the quantitation limit in the laboratory blank. These analytes should not be considered truly present in the native samples unless detected at a level at least five times the level in the blank. If the analyte level in the sample

is twenty times the level in the blank, the blank contribution can be considered statistically insignificant.

All internal standard areas were within Method 8270 quality control criteria.

A set of laboratory control spike (LCS)/LCS Duplicate samples was prepared and analyzed along with the samples. Results for the LCS/LCS Dup are reported as a summary that is included in the data package.

The surrogate standard percent recoveries for pyrene-d_{10} in the laboratory blank and one field sample (SITE#4 5-28-98 8hour) were somewhat above the in-house quality control limits. Each of these samples was analyzed twice and exhibited similar results for pyrene-d_{10} recoveries. All other surrogate standard percent recoveries were within in-house quality control limits as defined by the following 95 percent confidence intervals:

Surrogate Standards	Mean	95% confidence interval
anthracene-d_{10}	77	37.45 - 116.75
pyrene-d_{10}	80	33.65 - 126.15
terphenyl-d_{14}	77	18.86 - 135.64

Sample Calculations:

Response Factor, RF = $\frac{\text{Area analyte x Amt Is}}{\text{Area IS x Amt analyte}}$

Amount ug = $\frac{\text{Area analyte x Amt IS x DF}}{\text{Area IS x RF}}$

Where:

Amt IS = amount of internal standard = 40 ug
Amt analyte in the ccal = amount of analyte in the continuing calibration = 50 ug
DF = dilution factor

TRIANGLE LABORATORIES, INC.
CASE NARRATIVE

June 12, 1998
45869

The data reported has been judged to be valid and in compliance with the guidelines of Method 8270A Rev.1 (7/92) except as noted above. Should you have any questions about this project, please feel free to contact our Project Scientist, Rose West, at (919) 544-5729, ext. 270.

For Triangle Laboratories, Inc.,

Report Preparation:

Penny A. Brock

Penny A. Brock
Report Preparation Chemist

Quality Control:

Sarah A. Hubbard

Sarah A. Hubbard
Report Preparation Chemist

The total number of pages in this data package is 182.

Triangle Laboratories, Inc.

Sample Identification Index for Project: 45869

Client Id:	TLI Id:	File Name:
SITE#1 5-27-98 24hour	209-67-9,14	VH277
SITE#1 5-28-98 8hour	209-67-1,5	VH273
SITE#1 5-29-98 24hour	209-67-18,21	VH286
SITE#2 5-27-98 24hour	209-67-10,15	VH278
SITE#2 5-28-98 8hour	209-67-2,6	VH274
SITE#2 5-29-98 24hour	209-67-19,22	VH287
SITE#3 5-27-98 24hour	209-67-11,16	VH279
SITE#3 5-28-98 8hour	209-67-3,7	VH275
SITE#3 5-29-98 24hour	209-67-12,23	VH280
SITE#4 5-27-98 24hour	209-67-13,17	VH290
SITE#4 5-28-98 8hour	209-67-4,8	VH289
SITE#4 5-29-98 24hour	209-67-20,24	VH288
PUF FILTER BLANK	209-67-25	VH272
SBLK060498	SBLK060498	VH271

Triangle Laboratories, Inc.

Proj ct Summary for Project 45869

Client ID:	SITE#1 5-2 7-98 24hour	SITE#1 5-2 8-98 8hour	SITE#1 5-2 9-98 24hour	SITE#2 5-2 7-98 24hour	SITE#2 5-2 8-98 8hour
Filename :	VH277	VH273	VH286	VH278	VH274
TLI Id :	209-67-9,14	209-67-1,5	209-67-18,21	209-67-10,15	209-67-2,6
Matrix :	PUF	PUF	PUF	PUF	PUF
Units :	ug	ug	ug	ug	ug
Naphthalene	8.87	10.82	25.90	19.27	9.06
Fluorene	3.50	1.68	4.85	8.36	6.59
Phenanthrene	14.49	3.37	18.52	22.13	20.58
Anthracene	0.37	0.18	0.77	0.67	0.93
Fluoranthene	2.25	0.67	2.93	2.83	2.67
Benzo(a)pyrene	(1.28)	(1.30)	(1.04)	(1.06)	(0.81)

( )-E timated Detection Limit

Triangle Laboratories, Inc.
801 Capitol Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

Triangle Laboratories, Inc.

Proj :ct Summary for Project 45869

Client ID:	SITE#2 5-2 9-98 24hour	SITE#3 5-2 7-98 24hour	SITE#3 5-2 8-98 8hour	SITE#3 5-2 9-98 24hour	SITE#4 5-2 7-98 24hour
Filename :	VH287	VH279	VH275	VH280	VH290
TLI Id	209-67-19,22	209-67-11,16	209-67-3,7	209-67-12,23	209-67-13,17
Matrix :	PUF	PUF	PUF	PUF	PUF
Units	ug	ug	ug	ug	ug
Naphthalene	58.73	14.36	2.68	73.76	6.73
Fluorene	12.06	7.16	1.54	21.03	4.90
Phenanthrene	40.45	24.79	4.85	65.49	25.96
Anthracene	1.71	0.68	0.25	2.87	0.70
Fluoranthene	5.30	3.26	1.01	8.41	3.65
Benzo(a)pyrene	(1.31)	(1.02)	(0.97)	(1.04)	(1.75)

( )-I stimated Detection Limit

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone (919) 544-5729 • Fax: (919) 544-5491

Triangle Laboratories, Inc.
Project Summary for Project 45869

Client ID:	SITE#4 5-2 8-98 8hour	SITE#4 5-2 9-98 24hour	PUF FILTER BLANK	SBLK060498
Filename :	VH289	VH288	VH272	VH271
TLI Id	209-67-4,8	209-67-20,24	209-67-25	SBLK060498
Matrix	PUF	PUF	PUF	PUF
Units :	ug	ug	ug	ug
Naphthalene	**1.51**	**14.80**	**0.16**	**0.70**
Fluorene	**1.02**	**5.75**	(0.31)	(0.32)
Phenanthrene	**6.86**	**21.28**	(0.27)	**0.15**
Anthracene	**0.23**	**0.71**	(0.29)	(0.29)
Fluoranthene	**1.66**	**3.36**	(0.26)	(0.27)
Benzo(a)pyrene	(1.60)	(1.72)	(1.26)	(1.22)

( )-Estimated Detection Limit

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

1047

CLIENT CHAIN OF CUSTODY **SAMPLER'S SIGNATURE** K. Simonian **Quote 1107000330**

From: Derenzo and Associates, Inc.
39201 Schoolcraft Road
Suite B-10
Livonia, MI 48150

Project: Hull & Assoc

Contact: Kris Simonian
Phone: 313-464-3880
Fax: 313-464-4368
P.O.#: 21974

(DER01 /RW)

If the data will be reported to a state agency, which one? OHIO/USEPA

Analysis Wanted: As Hoc / ATI Acc(ed) / Lester

Total Number of Containers: 4 plugs 4 Filters

Sample Name	Date	Time	Comp	Grab	Matrix	Sample Amt.						Remarks
Site 1 8 Hour	5-28-98		✓		PUF/XAD							
Site 2 8 Hour	5-28-98		✓		PUF/XAD							
Site 3 8 Hour	5-28-98		✓		PUF/XAD							
Site 4 8 Hour	5-28-98		✓		PUF/XAD							
Site 1 8 Hour	5-28-98		✓		Filter							(Filters to BE
Site 2 8 Hour	5-28-98		✓		Filter							DESORBED with
Site 3 8 Hour	5-28-98		✓		Filter							corresponding
Site 4 8 Hour	5-28-98		✓		Filter							PUF/XAD plug)

Relinquished By/Sign.	Date/Time	Received By/Sign.	Relinquished By/Sign.	Date/Time	Received By/Sign.
K. Simonian	6-1-98 9:00am				
Relinquished By/Sign.	**Date/Time**	**Received By/Sign.**	**Relinquished By/Sign.**	**Date/Time**	**Received By/Sign.**

Received for Laboratory By/Signature	Date/Time	Send Samples to:
Bill Hunt	6/2/98 1000	Triangle Laboratories, Inc. Attn: Sample Custodian 801 Capitola Drive Durham, NC 27713 USA Phone: (919) 544-5729 • Fax: (919) 544-5491

CLIENT CHAIN OF CUSTODY **SAMPLER'S SIGNATURE** K. Simonian **Quote 1107000330**

From: Derenzo and Associates, Inc.
39201 Schoolcraft Road
Suite B-10
Livonia, MI 48150

Project: Hull & Assoc.

Contact: Kris Simonian
Phone: 313-464-3880
Fax: 313-464-4368
P.O.#: 21974

(DER01 /RW)

If the data will be reported to a state agency, which one? OHIO/USEPA

Analysis Wanted: As per Attached letter

Total Number of Containers: 4 plugs, 4 Filters

Sample Name	Date	Time	Comp	Grab	Matrix	Sample Amt.						Remarks
✓ SITE 1 24 HOUR	5-27-98		✓		PUF/XAD							
✓ SITE 2 24 HOUR	5-27-98		✓		PUF/XAD							
✓ SITE 3 24 HOUR	5-27-98		✓		PUF/XAD							
✓ SITE 4 24 HOUR	5-27-98		✓		PUF/XAD							
✓ SITE 1 24-HOUR	5-27-98		✓		Filter							(Filters to be
SITE 2 24-HOUR	5-27-98		✓		Filter							Desorbed with
SITE 3 24-HOUR	5-27-98		✓		Filter							corresponding
SITE 4 24-HOUR	5-27-98		✓		Filter							PUF/XAD plug)
Filter Blank	5-27-98		✓									

Relinquished By/Sign.	Date/Time	Received By/Sign.	Relinquished By/Sign.	Date/Time	Received By/Sign.
K. Simonian	6-1-98 9:01 am				
Relinquished By/Sign.	**Date/Time**	**Received By/Sign.**	**Relinquished By/Sign.**	**Date/Time**	**Received By/Sign.**

Received for Laboratory By/Signature	Date/Time	Send Samples to:
Bill Hunt	6/2/98 1 DIP	Triangle Laboratories, Inc. Attn: Sample Custodian 801 Capitola Drive Durham, NC 27713 USA Phone: (919) 544-5729 • Fax: (919) 544-5491

3 of 3

CLIENT CHAIN OF CUSTODY **SAMPLER'S SIGNATURE** [signature] **Quote 1107000330**

From: Derenzo and Associates, Inc.
39201 Schoolcraft Road
Suite B-10
Livonia, MI 48150

Project: Null Assoc.

Contact: Kris Simonian
Phone: 313-464-3880
Fax: 313-464-4368
P.O.#: 21974

(DER01 /RW) If the data will be reported to a state agency, which one? ONIC/USEPA

Analysis Wanted: AS PER ATTACHED LETTER

Total Number of Containers: 4 plugs 4 Filters

Sample Name	Date	Time	Comp	Grab	Matrix	Sample Amt.						Remarks
✓ Site 1 24 Hour	5-29-98		✓		PUF/XAD							
Site 2 24 Hour	5-29-98		✓		PUF/XAD							
✓ Site 3 24 Hour	5-29-98		✓		PUF/XAD							
✓ Site 4 24 Hour	5-29-98		✓		PUF/XAD							
Site 1 24 Hour	5-29-98		✓		Filter							(Filters to be
Site 2 24 Hour	5-29-98		✓		Filter							Desorbed with
Site 3 24 Hour	5-29-98		✓		Filter							corresponding
Site 4 24 Hour	5-29-98		✓		Filter							PUF/XAD plug)

Relinquished By/Sign.	Date/Time	Received By/Sign.	Relinquished By/Sign.	Date/Time	Received By/Sign.
[signature]	6-1-98 9:00am				
Relinquished By/Sign.	Date/Time	Received By/Sign.	Relinquished By/Sign.	Date/Time	Received By/Sign.

Received for Laboratory By/Signature	Date/Time	Send Samples to:
[signature]	6/2/98 1800	Triangle Laboratories, Inc. Attn: Sample Custodian 801 Capitola Drive Durham, NC 27713 USA Phone: (919) 544-5729 • Fax: (919) 544-5491

TRIANGLE LABORATORIES, INC.
Air Quality Preparation Work Order Form

ORDER INFORMATION

TLI Project.#: 04296
Company Name.: Derenzo and Associates, Inc.
Client Proj..:
Client P.O.#.: 21974
Quote Number.: 1107000330

Due Date..: 05/05/98
Order Date: 04/27/98
Cust.ID...: DER01
Proj.Sci..: RW

MATERIALS NEEDED

___ Filters
[_] 8.26cm
[_] 8.5cm
[_] 3 1/2"/9.0cm
[_] 4"/10.16cm
[_] 4 1/2"/11.0cm
[_] 5"/12.5cm
[_] Other _____

___ XAD traps
[_] Ball and Socket
[_] Socket and Socket
[_] Ball and Ball
[_] Ball and Stem
[_] Screw Cap
[_] Other _____

PUFs..... 5
Sandwich PUF: yes / no
Ship in: jars / sleeves
Glassware.....
[_] Client [✓] TLI [_] Rent____
Sleeves.....
[_] Client [✓] TLI

Filter Material: ___ Quartz ___ Glass fiber Other _____
Ship filter in: ___ 1 petri dish ___ separate petri dishes

SAMPLE PREPARATION INFORMATION

Preparer.....: Andrew Ch
# of Lab Blanks.: 1
XAD Resin Batch#: P1112
Pre-Spike req...: yes / no

Date....: 5 / 4 / 98
Supplier: Supelco

Spiker/Obs'er Inits: JLE / AC
Spiking Date: 5/4/98 / __ / __
Spiking Sol'n ID #: ~~NUSF-C-8~~ 5841A
Volumn Spiked (uL): 1000
Nominal Conc.: 100 ug/mL
Expiration Date: 7 / 31 / 98

..Method..	.Spike.
PCDD/PCDF	____ng
HR-PCB	____ug
LR-SVoa	100 ug
Other _____	____

5841A terphenyl D-14 EXP 7/31/98

SHIPPING INSTRUCTIONS

Ship Attn: Kris Simonian
c/o: Derenzo and Associates, Inc.
Address: 39201 Schoolcraft Road
Suite B-10
CStZip: Livonia, MI 48150
USA
Phone Num: 313-464-3880

SPECIAL INSTRUCTIONS

Form Prepared By: John Guenth Date: 4 / 27 / 98

Form Reviewed By: SA Date: 4 / 27 / 98

Return this form to TRIANGLE LABS, INC. with the samples.

TRIANGLE LABORATORIES, INC.
Air Quality Preparation Work Order Form

ORDER INFORMATION

TLI Project.#: 04325
Company Name.: Derenzo and Associates, Inc.
Client Proj..:
Client P.O.#.: 21974
Quote Number.: 1107000330

Due Date..: 05/27/98
Order Date: 05/21/98
Cust.ID...: DER01
Proj.Sci..: RW

MATERIALS NEEDED

___ Filters
[_] 8.26cm
[_] 8.5cm
[_] 3 1/2"/9.0cm
[_] 4"/10.16cm
[_] 4 1/2"/11.0cm
[_] 5"/12.5cm
[_] Other _____

___ XAD traps
[_] Ball and Socket
[_] Socket and Socket
[_] Ball and Ball
[_] Ball and Stem
[_] Screw Cap
[_] Other _____

PUFs..... 7
Sandwich PUF: yes / no
Ship in: jars / sleeves
Glassware.....
[_] Client [✓] TLI [7] Rent____
Sleeves.....
[_] Client [✓] TLI

Filter Material: ___ Quartz ___ Glass fiber Other _____
Ship filter in: ___ 1 petri dish ___ separate petri dishes

SAMPLE PREPARATION INFORMATION

Preparer.....: Andrew Cho
# of Lab Blanks.: 1
XAD Resin Batch#: P1131
Pre-Spike req...: yes / no

Date....: 5/26/98
Supplier: Supelco

Spiker/Obs'er Inits: SBM / [illegible]
Spiking Date: 5/26/98
Spiking Sol'n ID #: ~~USF-C-S~~ 5841A
Volumn Spiked (uL): Terphenyl Bulk 1000
Nominal Conc.: 100 ug/mL
Expiration Date: 7/31/98

..Method.. .Spike.
PCDD/PCDF _____ng
HR-PCB _____ug
LR-SVoa 100 ug terphenyl D-14
Other _____ _____

SHIPPING INSTRUCTIONS

Ship Attn: Kris Simonian
c/o: Derenzo and Associates, Inc.
Address: 39201 Schoolcraft Road
Suite B-10
CStZip: Livonia, MI 48150
USA
Phone Num: 313-464-3880

SPECIAL INSTRUCTIONS

Client needs to receive PUFs by Wednesday Morning, 5/27/98.
J Guenth 5/21/98

Form Prepared By: J Guenth Date: 5/21/98

Form Reviewed By: Rose V. West Date: 5/21/98

Return this form to TRIANGLE LABS, INC. with the samples.

TRIA GLE LABORATORIES, INC.

LIST OF CER IFICATIONS AND ACCREDITATIONS

ENVIRONMENTAL

American Association for Labora ory Accreditation. Accredidation pending. Certificate Number 0226-01. Accreditation fo technical competence in Environmental Testing. (Including Waste Water, Sol/Haz Waste, Pulp/ aper, and Air Matrices) Parameters are AOX/TOX, and Dioxin/Furan. Method 1613 for Dr king Water **Pending New Certificate.**.

State of Alabama, Department of Environmental Management. Expires December 31, 1998. Laboratory I.D. # 40950. Dioxin in drinking water.

State of Alaska, Department of E vironmental Conservation. Expired December 21, 1997. Certificate number OS-00397. Dio in in drinking water. **Pending New Certificate**

State of Arizona, Department of I ealth Services. Expires May 26, 1998. Certificate #AZ0423. Drinking Water for Dioxi , Dioxin in WW and S/H Waste. **Pending New Certificate**

State of Arkansas, Department of Pollution Control and Ecology. Expires February 19, 1999. Pulp/paper, soil, water, and H zardous Waste for Dioxin/Furan; AOX/TOX, Volatiles, Semi-volatiles, and Metals.

State of California, Department o Health Services. Expires August 31, 1999. Certificate #1922. Selected Metals in Waste W ter; Volatiles, Semi-volatiles, and Dioxin/furan in WW and Sol/Haz Waste. Dioxin in drinking v ater. **Pending New Certificate**

State of Connecticut, Department of Health Services. Expires September 30, 1999. Registration
PH-0117. Dioxin in drinking wate .

Delaware Health and Social Servi es. Expires December 31, 1998. Certificate #NC 140. Dioxin in drinking water.

Florida Department of Health anc Rehabilitative Services. Expires June 30, 1998. Dioxin in DW. Drinking Water ID HRS# 874 4. Metals, Extractable Organics (GC/MS), Pesticides/PCB's

Triangle Laboratories, Inc.
801 Capitola Drive
Durham, NC 27713-4411
919-544-5729
P.O. Box 13485
Research Triangle Park, NC 27709-3485
Fax # 919-544-5491

(GC) and Volatiles (GC/MS) in Environmental Samples. Environmental water ID HRS# E87411.

Hawaii Department of Health. Expires March 1, 1999. Dioxin in drinking water. "Accepted" status for regulatory purposes .

Idaho Department of Health and Welfare. Expires December 31, 1998. Dioxin in drinking water

State of Kansas, Department of Health and Environment. Expires January 31, 1999. Method 1613 for drinking water. ID #'s - Drinking water and/or pollution control - E-10215. Solid or Hazardous Waste - E-10129.

Commonwealth of Kentucky, Department for Environmental Protection. Expires December 31, 1998. ID#90060. Dioxin in drinking water.

Maryland Department of Health and Mental Hygiene. Expires September 30, 1998. Certification #235 Drinking water by Method 1613A.

State of Michigan, Department of Public Health. Expires June 30, 1998. Drinking water by Method 1613.

Mississippi State Department of Health. No expiration date. Dioxin in drinking water.

Montana Department of Health and Environmental Services. Expires December 31, 1998. Dioxin in drinking water.

State of New Jersey, Department of Environmental Protection and Energy. Expires June 30, 1998. ID #67851. BNAs and Volatiles. Dioxin in drinking water.

State of New Mexico, Environmental Department. Still certified, awaiting information from A2LA Dioxin in drinking water. **Pending New Certificate**

New York State Department of Health. Expires June 30, 1998. ID #11026. Environmental Analyses of non-potable Water, Soil and Hazardous Waste. Method 1613 in DW **Pending New Certificate.**

State of North Carolina, Department of Environment Health and Natural Resources
Expires
August 31, 1998. Certificate # 37751 Dioxin in drinking water.

Revised 5/21/98 dj
y:certific\certlist.mem

State of North Carolina, Departm nt of Environment, Health, and Natural Resources, Division of Environmental Manag ment. Expires December 31, 2000. Certificate # 485. Metals, pesticides & PCBs, semi-vol tiles and volatiles; TCLP.

North Dakota State Department o Health and Consolidated Laboratories. Expires December 31, 1998. Certificate # R- 76. Effective October 4, 1993. Dioxin in drinking water.

Oklahoma Department of Enviror mental Quality. Expires August 31, 1998. Laboratory #9612. Dioxin by 1613A, 8290 and 280.

State of South Carolina, Departm nt of Health and Environmental Control. Expires June 30, 1998. Certificate number # 9040001 (drinking water). Expires August 31, 1999. Certificate number #99040002 (othe parameters). Dioxin/Furans, BNA, Volatiles, and PCBs/pesticides under Clean Water ct, 2,3,7,8-TCDD for Drinking Water, and Organic extractables for Solid and Hazardous Waste.

State of Tennessee. Department of Environment and Conservation. Expires February 5, 1999. ID #02992. Metl od 1613 Drinking water only.

U.S. Department of Agriculture S il Permit. Expires September 30, 2001. Permit No. S-3790. Under the authority of the Federal Pl nt Pest Act, permission is granted to receive foreign soil samples for use in laboratory analysis.

U.S. Army Corps of Engineers. E pires October 19, 1999. Validated to perform analyses for the Fort Belvoir, VA (Contract Num er DACA31-97-D-0029), Vint Hill Farms Station, Vint Hill, VA (Contract Number DACA31-95- D-0083), and Selma Pressure Treating Superfund Site, Selma, CA (Contract number DACW 45-94-D-0054).

U.S. EPA Region V. Expires Nove nber 14, 1999. Dioxin in drinking water.

U.S. EPA Region VIII, for the Sta e of Wyoming. Expires November 12, 1998. Dioxin in drinking water.

State of Utah, Department of Heal h. Expired May 30,1998.. Certificate Number E-166. Certification for the following param ters: Semi-Volatiles and Volatiles under RCRA; Volatiles under Clean Water Act; Dioxin/furar s by Method 8280; Drinking water for Dioxin by Method 1613; Metals including Mercury and Microwave Digestion. **Pending New Certificate**

Commonwealth of Virginia, Depa tment of General Services, Division of Consolidated Laboratory Services. Expires June 30, 1998. ID # 00341. Dioxin in drinking water.

Revised 5/21/98 dj
y:certific\certlist.mem

State of Washington, Departmei t of Ecology. Expires September 11, 1998. Lab Accreditation Number C067. Scope of Accredita ion applies to water analyses for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Diben: ofurans, BNA Extr (Semivolatile) Organics and Purgeable (Volatile) Organics.
State of Washington, Departmer t of Health. Expired April 30, 1998. Dioxin in drinking water.
Lab I.D. 129. **Pending New Cert: ficate.**

State of West Virginia, Departm nt of Health. Expires December 31, 1998. Certificate No. 9923(C). Dioxin in drinking water.

State of Wisconsin, Department f Natural Resources. Expires August 31, 1998. Laboratory ID Number 999869530. Certificati n for the following categories of Organics: Purgeable, Base/Neutral, Acid, PCBs, and Dio din. Expires November 14, 1999. Laboratory ID 999869530. Dioxin in drinking water.

PHARMACEUTICAL

Drug Enforcement Agency (DEA . Expires November 30, 1998. Registration number RT01195835. Controlled substance registration for schedules 1,2,3,3N,4,5.

N.C. Department of Human Reso rces. Expires October 31, 1998. Registration number NC-PT 0000 0031. North Carolina :ontrolled substances registration. Application submitted for renewal.

Food & Drug Administration (FI A) Registration. Expires June 1998. ID #'s 001500 1053481. Annual registration of dru g establishment. Annual registration of drug establishment.

OTHER

Clinical Laboratory Improvemen Amendments (CLIA) Registration. Expires May 30, 1999.
ID # 34D0705123. Department of Health & Human Services, Health Care Financing Administration.

U.S. EPA Large Quantity Hazard us Waste Generator. No expiration date. EPA ID #NCD982156879. Permit indicates hat the laboratory is a large generator of hazardous waste.

North Carolina General License f r Radiation Protection. No. expiration date. License No. 032-875-OG. The general license a plies only to radioactive material contained in devices which have been manufactured and labeled n accordance with specific requirements.

Revised 5/21/98 dj
y:certific\certlist.mem

DOCUMENT CONTROL

Triangle Laboratories, Inc.
801 Capitola Drive
Durham, NC 27713-4411
919-544-5729
P.O. Box 13485
Research Triangle Park, NC 27709-348
Fax # 919-544-5491

TRIANGLE LABORATORIES, INC. -- LOG IN RECORD/CHAIN OF CUSTODY

Custody Seal : Absent
Chain of Custody : Present
Sample Tags : Absent
Sample Tag Numbers: Not Listed on Chain of Custody
SMO Forms : N/A

Sample Seals: Absent
Container...: Intact

TLI Project Number 45869
Client: DER01 - Derenzo and Associates, Inc.

Date Received 06/02/98 By Mark O'Brien

Carrier and Number FedEx/803859893302

Book 209 Page 67

Ice Chest ICE PACKS Temp 9.0 C

TLI Number.. mR/H:CPM.	....Client Sample ID..... .....Client COC ID.....*	.....Matrix Location.....	To LAB Date/Init	To STORAGE Date/Init	To LAB Date/Init	To STORAGE Date/Init	To LAB Date/Init	To STORAGE Date/Init	To LAB Date/Init	To STORAGE Date/Init	DISPOSED Date/Init
209-67-1	Site#1 5-28-98 8hour Site#1 5-28-98 8hour	PUF/XAD C02	6/4/98 JSY	USED							
209-67-2	Site#2 5-28-98 8hour Site#2 5-28-98 8hour	PUF/XAD C02									
209-67-3	Site#3 5-28-98 8hour Site#3 5-28-98 8hour	PUF/XAD C02									
209-67-4	Site#4 5-28-98 8hour Site#4 5-28-98 8hour	PUF/XAD C02									
209-67-5	Toledo#1 5-28-98 8hour Site 1 5-28-98 8HOUR *	FILTER C02									
209-67-6	Toledo#2 5-28-98 8hour Site 2 5-28-98 8HOUR *	FILTER C02									
209-67-7	Toledo#3 5-28-98 8hour Site 3 5-28-98 8HOUR *	FILTER C02									
209-67-8	Site #4 5-28 8hour Site 4 5-28-98 8HOUR *	FILTER C02									
209-67-9	Site #1 5-27-98 24hour Site 1 5-27-98 24hour *	PUF/XAD C02									
209-67-10	Site #2 5-27-98 24hour Site 2 5-27-98 24hour *	PUF/XAD C02									
209-67-11	Site #3 5-27-98 24hour Site 3 5-27-98 24Hour *	PUF/XAD C02									
209-67-12	Site #3 5-27-98 fri24hour Site 3 5-29-98 24hour *	PUF/XAD C02									
209-67-13	Site #4 5-27-98 24Hour Site 4 5-27-98 24hour *	PUF/XAD C02									
209-67-14	Toledo #1 5-27-98 Site 1 5-27-98 24hour *	FILTER C02	6/4/98 JSY	USED							

Receiving Remarks: No site or date on puf ID from Site 4 5/29/98. Puf with ID: Site 3 PUF 5/27/98 Fri 24hour should have been 5/29/98 according to faxed COC. Added Dates to Client's COC ID to help distinguish b/w samples; changed sample ID for #8 after initial log-in MAK 6-2-98

Archive Remarks:

Form Revised 05/27/1997 -- Page 1 OF 2

TRIANGLE LABORATORIES, INC. -- LOG IN RECORD/CHAIN OF CUSTODY

Custody Seal : Absent	Sample Seals: Absent	TLI Project Number 45869	Book
Chain of Custody : Present	Container...: Intact	Client: DER01 - Derenzo and Associates, Inc.	209
Sample Tags : Absent			
Sample Tag Numbers: Not Listed on Chain of Custody			
SMO Forms : N/A		Date Received 06/02/98 By Mark O'Hanna	Page
Ice Chest ICE PACKS Temp 9.0 C		Carrier and Number FedEx/803859893302	67

TLI Number.. mR/H:CPM.	....Client Sample ID..... .....Client COC ID.....*	.....Matrix Location.....	To LAB Date/Init	To STORAGE Date/Init	To LAB Date/Init	To STORAGE Date/Init	To LAB Date/Init	To STORAGE Date/Init	To LAB Date/Init	To STORAGE Date/Init	DISPOSED Date/Init
209-67-15	Toledo #2 5-27-98 Site 2 5-27-98 24hour *	FILTER C02	6/4/98 JSY	USED							
209-67-16	Toledo #3 5-27-98 Site 3 5-27-98 24hour *	FILTER C02									
209-67-17	Toledo #4 5-27-98 Site 4 5-27-98 24hour *	FILTER C02									
209-67-18	SITE#1 5/29/98 24 HOUR Site 1 5-29-98 24hour *	PUF/XAD C02									
209-67-19	SITE#2 5/29/98 24 HOUR Site 2 5-29-98 24hour *	PUF/XAD C02									
209-67-20	24 HOUR Site 4 5-29-98 24hour *	PUF/XAD C02									
209-67-21	Toledo #1 5/29/96 34 OFF Site 1 5-29-98 24hour *	FILTER C02									
209-67-22	Toledo #2 5/29/96 36 OFF Site 2 5-29-98 24hour *	FILTER C02									
209-67-23	Toledo #3 5/29/96 34 OFF Site 3 5-29-98 24hour *	FILTER C02									
209-67-24	Toledo #4 5/29/96 38 OFF Site 4 5-29-98 24hour *	FILTER C02									
209-67-25	PUF FILTER BLANK FILTER BLANK *	FILTER C02	6/4/98 JSY	USED							

Receiving Remarks: No site or date on puf ID from Site 4 5/29/98. Puf with ID: Site 3 PUF 5/27/98 Fri 24hour should have been 5/29/98 according to faxed COC.

Archive Remarks:

TRIANGLE LABORATORIES, INC.

Sample Preparation Tracking & Management Form

Project: 45869 Client: Derenzo and Associates, Inc. (DER01)

Sample Information

Method: 8270 for PAH Extraction Date: 06/04/98

Solvent/Acids(): 10% Et. Ether/HCl Lot: 971716-12/973460 (i.e.) JSY 6/4/98

Sample # crd	TLI SAMPLE ID / CLIENT SAMPLE ID	Sample Wgt / Vol ~~g / ml~~ train	Final Volume ml	5857A Surrg. cp. X exp. 07/08/98 06/04/98 100 µg/ml 1000 µl	5833B ~~5834A~~ Surrg. cp. W exp. 07/08/98 06/04/98 100 µg/ml 1000 µl	__/__/__	__/__/__	__/__/__	__/__/__
000	TLI Blank / TLI PUF Blank	1	1	JLE JSY	JLE JSY				
001	209-67-1,5 / Site#1 5-28-98 8hour	1	1	JLE JSY	JLE JSY				
002	209-67-2,6 / Site#2 5-28-98 8hour	1	1	JLE JSY	JLE JSY				
003	209-67-3,7 / Site#3 5-28-98 8hour	1	1	JLE JSY	JLE JSY				
004	209-67-4,8 / Site#4 5-28-98 8hour	1	1	JLE JSY	JLE JSY				
005	209-67-9,14 / Site #1 5-27-98 24hour	1	1	JLE JSY	JLE JSY				
006	209-67-10,15 / Site #2 5-27-98 24hour	1	1	JLE JSY	JLE JSY				
007	209-67-11,16 / Site #3 5-27-98 24hour	1	1	JLE JSY	JLE JSY				
008	209-67-12,23 / Site #3 5-27-98 fri24hour	1	1	JLE JSY	JLE JSY				
009	209-67-13,17 / Site #4 5-27-98 24Hour	1	1	JLE JSY	JLE JSY				
010	209-67-18,21 / SITE#1 5/29/98 24 HOUR	1	1	JLE JSY	JLE JSY				
011	209-67-19,22 / SITE#2 5/29/98 24 HOUR	1	1	JLE JSY	JLE JSY				
012	209-67-20,24 / 24 HOUR	1	1	JLE JSY	JLE JSY				

Comments: Pre-spiked blank (prep. #04325) was used as TLI blank; pre-sol'd pufs were used for LCS, LCSD. JSY 6/4/98

REV 07/26/93 (PSTMF 7)

TRIANGLE LABORATORIES, INC.

Sample Preparation Tracking & Management Form

Project: 45869 Client: Derenzo and Associates, Inc. (DER01)

Sample Information (i.e.) 734 6/4/98

Method: 8270 for PAH Extraction Date: 06/04/98

Solvent/Acids(): 10% Et. Ether/Hex Lot: 971716-12 / 973460 58332

734 6/4/98 (i.e.)

Sample # crd	TLI SAMPLE ID / CLIENT SAMPLE ID	Sample Wgt / Vol ~~g / ml~~ train	Final Volume ml	5857A Surrg. sp. X exp. 07/08/98 06/04/98 100 µg/ml 1000 µl	~~5834A~~ Surrg. sp. W exp. 07/08/98 06/04/98 100 µg/ml 1000 µl	6064 8270 Acid QC check. sol. exp. 10/16/98 06/04/98 100 µg/ml 1000 µl	6059 8270 BN QC check. sol. exp. 08/26/98 06/04/98 100 µg/ml 1000 µl	~~5841A~~ p-Terphenyl/-d14 surr. sp. exp. 07/31/98 06/04/98 100 µg/ml 1000 µl	___/___/___
013	209-67-25 PUF FILTER BLANK	1	1	JLE JSY	JLE JSY				
014	TLI LCS TLI LCS	1	1	JLE JSY	JLE JSY	x JLE JSY	x JLE JSY	x	
015	TLI LCSD TLI LCSD	1	1	JLE JSY	JLE JSY	x JLE JSY	x JLE JSY	x	

Comments:

REV 07/26/93 (PSTMF 7)

45869

OWL SAMPLE EXTRACTION AND CLEANUP TRACKING FORM
PUF'S-FOR 680/8270 ANALYSIS

		STEP DESCRIPTION		
All	OWL-165	Prep glassware JSY 6/4/92		
"	OWL-165	Place filter and PUF into thimble holder or thimble	JLE/JSY	G
"	DSP-128	Spike appropriate internal standards and/or surrogates	JSY	G
"	DSP-128	Spike aprropriate QC solutions, if needed		
"	OWL-165	Extract 16 hours with 10 % ethyl ether/hexane	JSY	G
"	OWL-111	Filter extract through NaSO4		
"	OWL-111	K/D to about 10 mL	JSY	G
"	OWL-111	N2 -> 1 mL	JSY	G
"	OWL-111	Transfer to 1/2 dram vial, rinse 2 X 0.5 mL hexane	JSY	G
"	OWL-111	N2 -> 1 mL	JLE/JSY	G
"	---	Relinquish to LRGC/MS	JLE	G
			JLE	G

TRIANGLE LABORATORIES, INC.
Wet Lab Project Communication Tracking Form
Project: 45869 Company: Derenzo and Associates, Inc. (DER01)

Use this form to record all exchanges of information between production units when computer access is not available, or as specified by SOP.

Date	Initials	Comment / Decision / Resolution / Action / Observation
6/4/98	JLE, JSY	Triangle Laboratories, Inc. TLI PROJECT #04325 DATE: 05-26-98 TLI BLANK SPIKE: 100ug TERPHENYL D-14 SPIKER SBM PREPED BY AC DERENZO AND ASSOCIATES, INC (4)
		2 puf yellow, xad off white
		DER01-Derenzo and Associates, Inc. Toledo#1 5-28-98 8hour Project:45869 209-67-5 (1)
		Triangle Laboratories, Inc. TLI PROJECT #04325 DATE: 05-26-98 SPIKE: 100ug TERPHENYL D-14 SPIKER SBM PREPED BY AC DERENZO AND ASSOCIATES, INC (1)
		2 puf yellow, xad white off 1 filter gray
		DER01-Derenzo and Associates, Inc. Toledo#2 5-28-98 8hour Project:45869 209-67-6 (2)
		1 filter light gray xad - off white 2 pufs yellow
		Triangle Laboratories, Inc. TLI PROJECT #04325 DATE: 05-26-98 SPIKE: 100ug TERPHENYL D-14 SPIKER SBM PREPED BY AC DERENZO AND ASSOCIATES, INC (2)

Revised 04/(

TRIANGLE LABORATORIES, INC.
Wet Lab Project Communication Tracking Form
Project: 45869 Company: Derenzo and Associates, Inc. (DER01)

Use this form to record all exchanges of information between production units when computer access is not available, or as specified by SOP.

Date	Initials	Comment / Decision / Resolution / Action / Observation
6/4/98	JSY	DER01-Derenzo and Associates, Inc. Toledo#3 5-28-98 8hour Project:45869 209-67-7 (3) Triangle Laboratories, Inc. TLI PROJECT #04325 DATE: 05-26-98 SPIKE: 100ug TERPHENYL D-14 SPIKER SBM PREPED BY AC DERENZO AND ASSOCIATES, INC (3)
		1 filter - lt. g 2 pufs - yell XAD - white
		DER01-Derenzo and Associates, Inc. Site #4 5-28 8hour Project:45869 209-67-8 (4) Triangle Laboratories, Inc. TLI PROJECT #04325 DATE: 05-26-98 SPIKE: 100ug TERPHENYL D-14 SPIKER SBM PREPED BY AC DERENZO AND ASSOCIATES, INC (4)
		1 filter - lt. gra 2 pufs - yellow XAD - white
		DER01-Derenzo and Associates, Inc. Toledo #1 5-27-98 Project:45869 209-67-14 (5) Triangle Laboratories, Inc. TLI PROJECT# 04296 DATE: 05-04-98 SPIKE: 100ug TERPHENYL-D14 SPIKER JLE PREPED BY AC DERENZO AND ASSOCIATES, INC. (5)
		1 filter - gray 2 pufs - yel XAD - white

Revised 04/08/98 (PREPCO)

JSY 6/4/98

TRIANGLE LABORATORIES, INC.
Wet Lab Project Communication Tracking Form
Project: 45869 Company: Derenzo and Associates, Inc. (DER01)

Use this form to record all exchanges of information between production units when computer access is not available, or as specified by SOP.

Date	Initials	Comment / Decision / Resolution / Action / Observation
6/4/98	JSY	DER01-Derenzo and Associates, Inc. Toledo #2 5-27-98 Project: 45869 209-67-15 (6) — 1 filter - gray
		Triangle Laboratories, Inc. TLI PROJECT# 04296 DATE: 05-04-98 SPIKE: 100ug TERPHENYL-D14 SPIKER JLE PREPED BY AC DERENZO AND ASSOCIATES, INC. (6) — 2 pufs - yel XAD - whit
		DER01-Derenzo and Associates, Inc. Toledo #3 5-27-98 Project: 45869 209-67-16 (7) — 1 filter - gray
		Triangle Laboratories, Inc. TLI PROJECT# 04296 DATE: 05-04-98 SPIKE: 100ug TERPHENYL-D14 SPIKER JLE PREPED BY AC DERENZO AND ASSOCIATES, INC. (7) — 2 pufs - yellow XAD - white
		DER01-Derenzo and Associates, Inc. Toledo #3 5/29/96 134 OFF Project: 45869 209-67-23 (8) — 1 filter - lt. gray
		Triangle Laboratories, Inc. TLI PROJECT #04325 DATE: 05-26-98 SPIKE: 100ug TERPHENYL D-14 SPIKER SBM PREPED BY AC DERENZO AND ASSOCIATES, INC (8) — 2 pufs - yello XAD - white

4/08/98 (PREPCO)-

JSY 6/4/98

TRIANGLE LABORATORIES, INC.
Wet Lab Project Communication Tracking Form
Project: 45869 Company: Derenzo and Associates, Inc. (DER01)

Use this form to record all exchanges of information between production units when computer access is not available, or as specified by SOP.

Date	Initials	Comment / Decision / Resolution / Action / Observation	
6/4/98	JSY	DER01-Derenzo and Associates, Inc. Toledo #4 5-27-98 Project: 45869 209-67-17 (9)	1 filter-gray
		Triangle Laboratories, Inc. TLI PROJECT# 04296 DATE: 05-04-98 SPIKE: 100ug TERPHENYL-D14 SPIKER JLE PREPED BY AC DERENZO AND ASSOCIATES, INC. (9)	2 pufs-yellow XAD-white
		DER01-Derenzo and Associates, Inc. Toledo #1 5/29/96 34 OFF Project: 45869 209-67-21 (10)	1 filter-lt-gra
		Triangle Laboratories, Inc. TLI PROJECT #04325 DATE: 05-26-98 SPIKE: 100ug TERPHENYL D-14 SPIKER SSR PREPED BY AC DERENZO AND ASSOCIATES, INC (10)	2 pufs-yellow XAD-white
		DER01-Derenzo and Associates, Inc. Toledo #2 5/29/96 36 OFF Project: 45869 209-67-22 (11)	1 filter-lt-gray
		Triangle Laboratories, Inc. TLI PROJECT# 04296 DATE: 05-04-98 SPIKE: 100ug TERPHENYL-D14 SPIKER JLE PREPED BY AC DERENZO AND ASSOCIATES, INC. (11)	2 pufs-yellow XAD-white

Revised 04/08/98 (PREPCO)

JSY 6/4/98

TRIANGLE LABORATORIES, INC.
Wet Lab Project Communication Tracking Form
Project: 45869 Company: Derenzo and Associates, Inc. (DER01)

Use this form to record all exchanges of information between production units when computer access is not available, or as specified by SOP.

Date	Initials	Comment / Decision / Resolution / Action / Observation
6/4/98	JSY	DER01-Derenzo and Associates, Inc. Toledo #4 5/29/96 38 OFF Project: 45869 209-67-24 (12)
		1 filter - gray
		Triangle Laboratories, Inc. TLI PROJECT #04325 DATE: 05-26-98 SPIKE: 100ug TERPHENYL D-14 SPIKER Skin PREPED BY AC DERENZO AND ASSOCIATES, INC (12)
		2 pufs - yellow XAD - white
		DER01-Derenzo and Associates, Inc. PUF FILTER BLANK Project: 45869 (13) 209-67-25
		1 large (like a sheet) filter - white
		JSY 6/4/98

Revised 04/08/98 (PREPCO)

TRIANGLE LABORATORIES, INC.
Transfer Chain-of-Custody Form
Project 45869

Transfer From: OWLS5 To: OGCPS

	Initials..	Date.....	Time...
Released by:	JLE	6/6/98	8:50
Accepted by:	DD	6/8/	4:20

MILES.ID.....		TLI_No.....	Cust.Id.....
45869-	-000	TLI Blank	TLI PUF Blank
45869-	-001	209-67-1,5	Site#1 5-28-98 8hour
45869-	-002	209-67-2,6	Site#2 5-28-98 8hour
45869-	-003	209-67-3,7	Site#3 5-28-98 8hour
45869-	-004	209-67-4,8	Site#4 5-28-98 8hour
45869-	-005	209-67-9,14	Site #1 5-27-98 24hour
45869-	-006	209-67-10,15	Site #2 5-27-98 24hour
45869-	-007	209-67-11,16	Site #3 5-27-98 24hour
45869-	-008	209-67-12,23	Site #3 5-27-98 fri24hour
45869-	-009	209-67-13,17	Site #4 5-27-98 24Hour
45869-	-010	209-67-18,21	SITE#1 5/29/98 24 HOUR
45869-	-011	209-67-19,22	SITE#2 5/29/98 24 HOUR
45869-	-012	209-67-20,24	24 HOUR
45869-	-013	209-67-25	PUF FILTER BLANK
45869-	-014	TLI LCS	TLI LCS
45869-	-015	TLI LCSD	TLI LCSD

XfrCOC (Rev 11/01/94)

Additional comments or instructions:

SEMIVOLATILE GC/MS TRACKING FORM

TL-RTP Project #: 458169

MATRIX: MM5 (PUF) Water Soil Other _____

METHOD: (8270) 625 CLP

ANALYTE LIST: Table 2 TCLP 625 CLP CAA/PAP (PAH) CB/CP Other _____

ICAL Name: ICAL 050698

Date	Time	TLRTP ID	Sample ID	File Name	Comments
6/8/98	1343	5793	DFTPP	VH266	
6/8/98	1417	6002	SSTD050	VH267	
6/8/98	1508	N/A	LCS	VH268	
6/8/98	1547	N/A	LCS Dup	VH 269	
6/8/98	1725	N/A	SBLK 060498	VH271	pyrene-d10 high
6/8/98	1815	209-67-25	PUF Filter Blank	VH272	
6/8/98	1904	209-67-1,5	Site#1 5-28-98 8hr	VH273	
6/8/98	1954	209-67-2,6	Site#2 5-28-98 8hr	VH274	
6/8/98	2043	209-67-3,7	Site#3 5-28-98 8hr	VH275	
6/8/98	2132	209-67-4,8	Site#4 5-28-98 8hr	VH276	not reported PAB 6/12/ pyrene-d10 high CPH 6-1
6/8/98	2221	209-67-9,14	Site#1 5-28-98 24hr	VH277	
6/8/98	2310	209-67-10,15	Site#2 5-27-98 24hr	VH278	
6/8/98	2358	209-67-11,16	Site#3 5-27-98 24hr	VH279	
6/9/98	0047	209-67-12,23	Site#3 5-27-98 Fri 24hr	VH280	client COC says: Site #3 5-29-98 24hour PAB 6/12/98
6/9/98	0135	209-67-13,17	Site#4 5-27-98 24-hr	VH281	pyrene-d10 high CPH 6
6/9/98	0935	5793	DFTPP	VH282	
6/9/98	1031	6002	SStd050	VH284	
6/9/98	1307	209-67-18,21	Site#1 5-29-98 24hr	VH286	
6/9/98	1355	209-67-19,22	Site#2 5-29-98 24hr	VH287	
6/9/98	1442	209-67-20,24	24 hour	VH288	client COC says: Site #4 5-29-98 24 hour PAB 6/12/98
6/9/98	1529	209-67-4,8	Site#4 5-28-98 8hr	VH289	pyrene-d10 high CPH 6
6/9/98	1616	209-67-13,17	Site#4 5-27-98 24hr	VH290	
6/9/98	1751	N/A	SBLK 060498	VH292	not reported PAB 6/12/ pyrene-d10 high CPH 6-11

RIANGLE LABORATORIES OF RTP, INC.

Triangle Laboratories, Inc.
Run Log

COPY

CPH 6-11-98

Column Type	Column #	Analysis*	Acquisition Method	GC Method*	Find DBs*	Other*
DB5-625	5672811	—	cclist.m	—	—	—

Standards		
Internal / Surrogate / Recovery	Internal / Surrogate / Recovery	Analyte
6013C Exp 9.15.98 5µl @ 834 µg/ml	N/A	N/A

Extract / Sample volume 100 µL mL (Circle unit)

DD 5/6/98
Signature Date

Date**	Time**	Project	Sample#	Client ID	Filename	pH*	Operator/Date	Backup*	Proc	Comments***
5/6/98	1037	N/A	5999	SSTD160	VH165		DD 5/6/98		DD	poor chrom peaks sat'd DD 5/6/98
5/6/98	1135	N/A	5999	SSTD160	VH166		DD 5/6/98		DD	poor chrom DD 5/6/98
5/6/98	1514	N/A	5793	DFTPP	VH167		DD 5/6/98		DD	autotuned - changed polarity DD 5/6/98
5/6/98	1533	N/A	5999	SSTD160	VH168		DD 5/6/98		DD	
5/6/98	1640	N/A	6001	SSTD080	VH169		DD 5/6/98		DD	
5/6/98	1717	N/A	6000	SSTD120	VH170		DD 5/6/98		DD	
5/6/98	1805	N/A	6002	SSTD050	VH171		DD 5/6/98		DD	
5/6/98	1853	N/A	6003	SSTD020	VH172		DD 5/6/98		DD	
5/6/98	1941	N/A	6004	SSTD010	VH173		DD 5/6/98		DD	
5/6/98	2029	9160ID @ad 5/6/98 N/A	N/A	SBLK 041498	VH174		DD 5/6/98		DD	

* Volatile Data Only ** Transcribed Data *** Dated Signature/Initials Required

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COPY
CPH 6-11-98

Triangle Laboratories, Inc.
Run Log

Column Type	Column #	Analysis*	Acquisition Method	GC Method*	Find DBs*	Other*
DB5-625	5672811		CClist.m			

Standards		
Internal / Surrogate / Recovery	Internal / Surrogate / Recovery	Analyte
6013C Exp: 9/15/98 5µl @ 834 µg/ml	N/A	N/A

Extract / Sample volume 100 µl (Circle unit) mL

CPH — Signature
6-11-98 — Date

Date**	Time**	Project	Sample#	Client ID	Filename	pH*	Operator/Date	Backup*	Proc	Comments***
6/8/98	1317	N/A	5793	DFTPP	VH265		MD 6/8/98		MD	poor chrom. no 6/8/98
6/8/98	1343	N/A	5793	DFTPP	VH266		MD 6/8/98		MD	pruned MD 6/8/98
6/8/98	1417	N/A	6002	SStd050	VH267		MD 6/8/98		MD	pruned MD 6/8/98
6/8/98	1508	45869	N/A	LCS	VH268		MD 6/8/98		MD	
6/8/98	1547	45869	N/A	LCSDup	VH269		MD 6/8/98		MD	
6/8/98	1636	N/A	N/A	Solvent Blank	VH270		MD 6/8/98		MD	
6/8/98	1725	45869	N/A	SBlk 060498	VH271		MD 6/8/98		MD	
6/8/98	1815	45869	209-67-25	Puf Filter Blk	VH272		MD 6/8/98		MD	
6/8/98	1904	45869	209-67-1,5	Site#1 5-28-98 8hour	VH273		MD 6/8/98		MD	
6/8/98	1954	45869	209-67-2,6	Site#2 5-28-98 8hour	VH274		MD 6/8/98		MD	

* Volatile Data Only ** Transcribed Data *** Dated Signature/Initials Required

SRVR1\QAFORMS\LRRUNLOG.DOC (1/23/97)

Triangle Laboratories, Inc.
Run Log

COPY
CPH 6-11-98

Column Type	Column #	Analysis*	Acquisition Method	GC Method*	Find DBs*	Other*
DB5-625	5672811		cclist.m			

Standards		
Internal / Surrogate / Recovery	Internal / Surrogate / Recovery	Analyte
6013C Exp: 9/5/98 5µl @ 834 µg/ml	N/A	N/A

Extract / Sample volume 100 (µL) mL (Circle unit)

CPH — Signature; 6-11-98 — Date

Date**	Time**	Project	Sample#	Client ID	Filename	pH*	Operator/Date	Backup*	Proc	Comments***
6/8/98	2043	45869	209-67-3,7	Site# 3 5-28-98 8hour	VH275		MD 6/8/98		MD	
6/8/98	2132	45869	209-67-4,8	Site#4 5-28-98 8hour	VH276		MD 6/8/98		MD	
6/8/98	2221	45869	209-67-9,14	Site#1 5-28-98 24hour @CPH 6-11-98	VH277		MD 6/8/98		MD	
6/8/98	2310	45869	209-67-10,15	Site#2 5-27-98 24hour	VH278		MD 6/8/98		MD	
6/8/98	2358	45869	209-67-11,16	Site#3 5-27-98 24hour	VH279		MD 6/8/98		MD	
6/9/98	0047	45869	209-67-12,23	Site#3 5-27-98 Fri 24hr	VH280		MD 6/8/98		MD	
6/9/98	0135	45869	209-67-13,17	Site#4 5-27-98 24hr	VH281		MD 6/8/98		MD	
6/9/98	0224	45869	209-67-18,21	Site#1 5-29-98 24hour	VH282		MD 6/8/98		MD	Off clock MD 6/9/98
6/9/98	0925	N/A	5793	DFTPP	VH283		MD 6/9/98		MD	passed MD 6/9/98
6/9/98	1031	N/A	6002	SStd050	VH284		MD 6/9/98		MD	passed MD 6/9/98

* Volatile Data Only ** Transcribed Data *** Dated Signature/Initials Required

SRVR1\QAFORMS\LRRUNLOG.DOC (1/23/97)

Triangle Laboratories, Inc.
Run Log

CPH 6-11-98

Column Type	Column #	Analysis*	Acquisition Method	GC Method*	Find DBs*	Other*
DB5-625	5672811		cclist.m			

Standards		
Internal / Surrogate / Recovery	Internal / Surrogate / Recovery	Analyte
60132 Exp: 9/15/98 5 µl @834 µg/ml	N/A	N/A

Extract / Sample volume 100 µL (Circle unit) mL

CPH — Signature; 6-11-98 — Date

Date**	Time**	Project	Sample#	Client ID	Filename	pH*	Operator/Date	Backup*	Proc	Comments***
6/9/98	1230	N/A	N/A	Solvent Blank	VH285		DD 6/9/98		DD	
6/9/98	1307	45869	209-67-18,21	Site#1 5-27-98 24 hour	VH286		DD 6/9/98		DD	
6/9/98	1355	45869	209-67-19,22	Site#2 5-27-98 24 hour	VH287		DD 6/9/98		DD	
6/9/98	1442	45869	209-67-20,24	@CPH 6-11-98 Site# 24 hour	VH288		DD 6/9/98		DD	
6/9/98	1529	45869	209-67-4,8	@CPH 6-11-98 Site#4 5-27-98 8 hour	VH289		DD 6/9/98		DD	
6/9/98	1616	45869	209-67-13,17	Site#4 5-27-98 24 hour	VH290		DD 6/9/98		DD	
6/9/98	1705	N/A	N/A	Solvent Blank	VH291		DD 6/9/98		DD	
6/9/98	1751	45869	N/A	SBLK 060498	VH292		DD 6/9/98		DD	

* Volatile Data Only ** Transcribed Data *** Dated Signature/Initials Required

SRVR1\QAFORMS\LRRUNLOG.DOC (1/23/97)

Triangle Laboratories, Inc.
LCS Summary of Results

LCS File : VH268	LCS DUP File: VH269
Date : 06/12/98	

Compound	LCS %REC	DUP %REC	RPD
Naphthalene	75	76	1
Fluorene	94	91	3
Phenanthrene	92	92	0
Anthracene	91	88	3
Fluoranthene	89	83	7
Benzo(a)pyrene	87	87	0

Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH268

Method 8270A PUF
Sample ID: LCS

Client Project: Hull & Assoc
TLI ID: LCS

Date Received: / /
Date Extracted: 06/04/98
Date Analyzed : 06/08/98

Response File: VH267

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.55		
Naphthalene	75.33	B	12.59		10
Acenaphthene-d_{10}		IS 3	16.18		
Fluorene	93.68		17.33		10
Phenanthrene-d_{10}		IS 4	19.28		
Phenanthrene	92.14	B	19.34		10
Anthracene	91.17		19.45		10
Fluoranthene	88.63		21.82		10
Chrysene-d_{12}		IS 5	24.90		
Perylene-d_{12}		IS 6	28.95		
Benzo(a)pyrene	87.25		28.81		10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	97.02	19.41	4	97
Pyrene-d_{10}	110.51	22.26	5	111

Reviewed by PAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

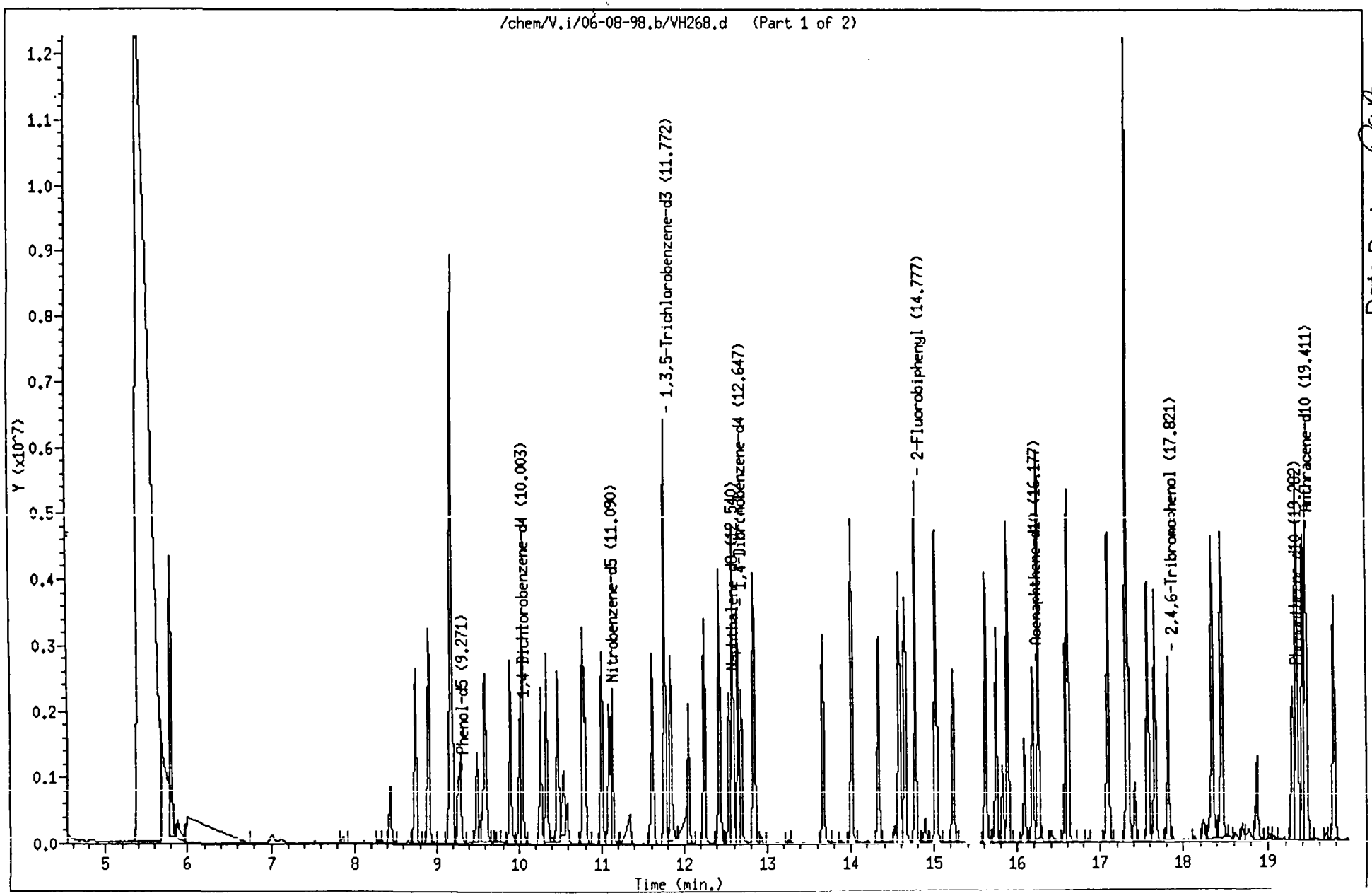
Savar v3.7
Printed: 15:26 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH268.d
Date : 08-JUN-1998 15:08
Instrument : V.i
Sample ID : LCS
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

38

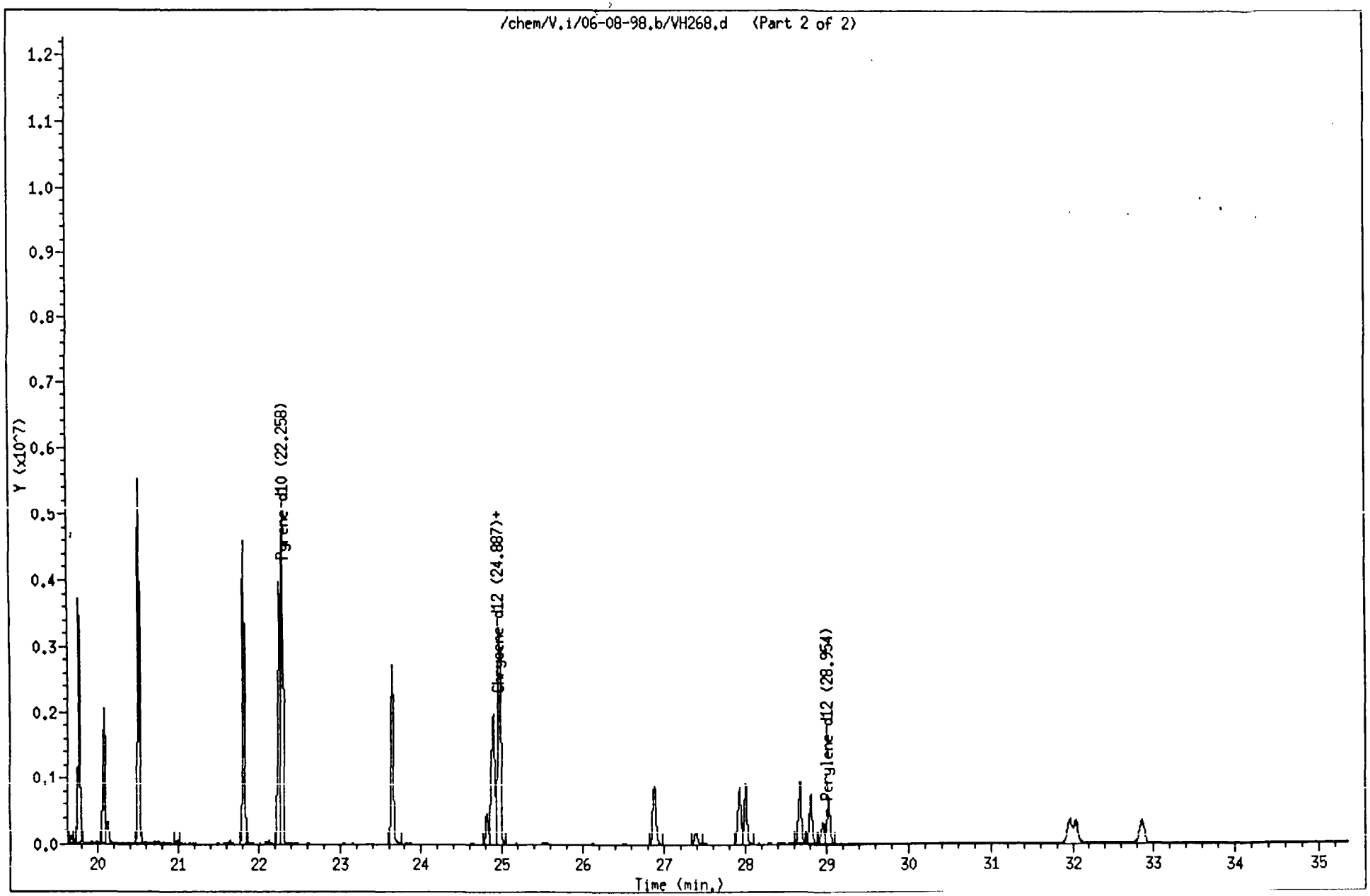


Data Review: PaB
Date: 6/12/98

Data File: /chem/V.i/06-08-98.b/VH268.d
Date : 08-JUN-1998 15:08
Instrument : V.i
Sample ID : LCS
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH268.d
Lab. Id. : 45869
Inj Date : 08-JUN-1998 15:08 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : LCS
Misc Info :
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 1
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	10.003(1.000)	508056	40.00	
*	2 Naphthalene-d8	136.00	12.547(1.000)	1796270	40.00	
*	3 Acenaphthene-d10	164.00	16.177(1.000)	999600	40.00	
*	4 Phenanthrene-d10	188.00	19.282(1.000)	1657474	40.00	
*	5 Chrysene-d12	240.00	24.901(1.000)	595576	40.00	
*	6 Perylene-d12	264.00	28.954(1.000)	333308	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.271(0.927)	470948	30.70	30.70
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.090(0.884)	1096229	71.85	71.85
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.772(0.938)	1198118	77.26	77.26
$	13 1,4-Dibromobenzene-d4	240.00	12.647(1.264)	968066	84.62	84.62
$	14 2-Fluorobiphenyl	172.00	14.777(0.913)	2489772	86.76	86.76
$	15 2,4,6-Tribromophenol	329.80	17.821(1.102)	461941	80.48	80.48
$	16 Anthracene-d10	188.00	19.411(1.007)	3304619	97.02	97.02
$	17 Pyrene-d10	212.00	22.258(0.894)	3007977	110.50	110.50
$	18 Terphenyl-d14	Compound Not Detected.				
	42 Naphthalene	128.00	12.590(1.003)	3254330	75.36	75.36
	46 2-Methylnaphthalene	142.00	14.016(1.117)	2249567	84.34	84.34
	50 2-Chloronaphthalene	162.00	15.026(0.929)	2106735	84.66	84.66
	56 Acenaphthylene	152.00	15.886(0.982)	3340675	80.08	80.08
	58 Acenaphthene	154.00	16.249(1.004)	2090414	87.24	87.24
	65 Fluorene	166.00	17.330(1.071)	2984821	93.69	93.69
	73 Phenanthrene	178.00	19.339(1.003)	3501069	92.17	92.17
	74 Anthracene	178.00	19.447(1.009)	3230113	91.15	91.15
	77 Fluoranthene	202.00	21.818(1.132)	3437384	88.64	88.64
	78 Pyrene	202.00	22.301(0.896)	3498687	101.15	101.15
	83 Benzo(a)anthracene	228.00	24.880(0.999)	1595852	84.19	84.19
	84 Chrysene	228.00	24.974(1.003)	1477885	91.40	91.40
	86 Benzo(b)fluoranthene	252.00	27.932(0.965)	949386	81.69	81.69

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	252.00	28.011(0.967)	851420	87.52	87.52
88 Benzo(e)pyrene	252.00	28.670(0.990)	943569	96.47	96.47
89 Benzo(a)pyrene	252.00	28.812(0.995)	717580	87.25	87.25
90 Perylene	252.00	29.026(1.002)	686634	96.22	96.22
91 Indeno(1,2,3-cd)pyrene	276.00	31.959(1.104)	624517	91.71	91.71(M)
92 Dibenz(a,h)anthracene	278.00	32.045(1.107)	430703	89.33	89.33
93 Benzo(g,h,i)perylene	276.00	32.861(1.135)	575521	96.03	96.03

QC Flag Legend

M - Compound response manually integrated.

Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH269

Method 8270A PUF
Sample ID: LCS Dup

Client Project: Hull & Assoc
TLI ID: LCSDup

Date Received: / /
Date Extracted: 06/04/98
Date Analyzed : 06/08/98

Response File: VH267

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.53		
Naphthalene	75.85	B	12.58		10
Acenaphthene-d_{10}		IS 3	16.16		
Fluorene	91.03		17.32		10
Phenanthrene-d_{10}		IS 4	19.27		
Phenanthrene	92.34	B	19.33		10
Anthracene	87.80		19.43		10
Fluoranthene	83.21		21.80		10
Chrysene-d_{12}		IS 5	24.89		
Perylene-d_{12}		IS 6	28.93		
Benzo(a)pyrene	86.89		28.79		10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	93.73	19.39	4	94
Pyrene-d_{10}	21.67	22.24	5	122

Reviewed by PAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

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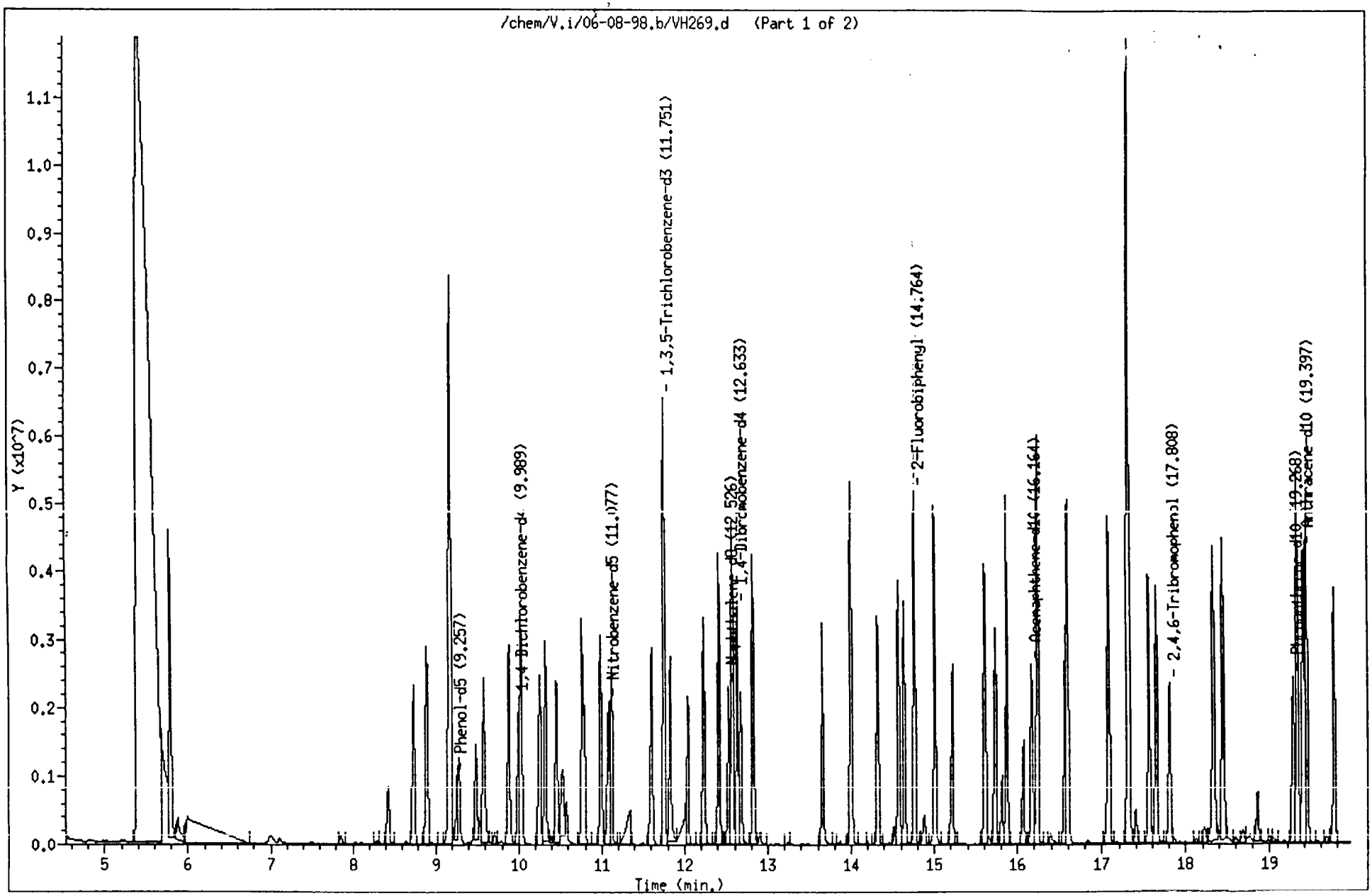
Savar v3.7
Printed: 15:26 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH269.d
Date : 08-JUN-1998 15:47
Instrument : V.i
Sample ID : LCS DUP
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

43



Data Review: PAB
Date: 6/12/98

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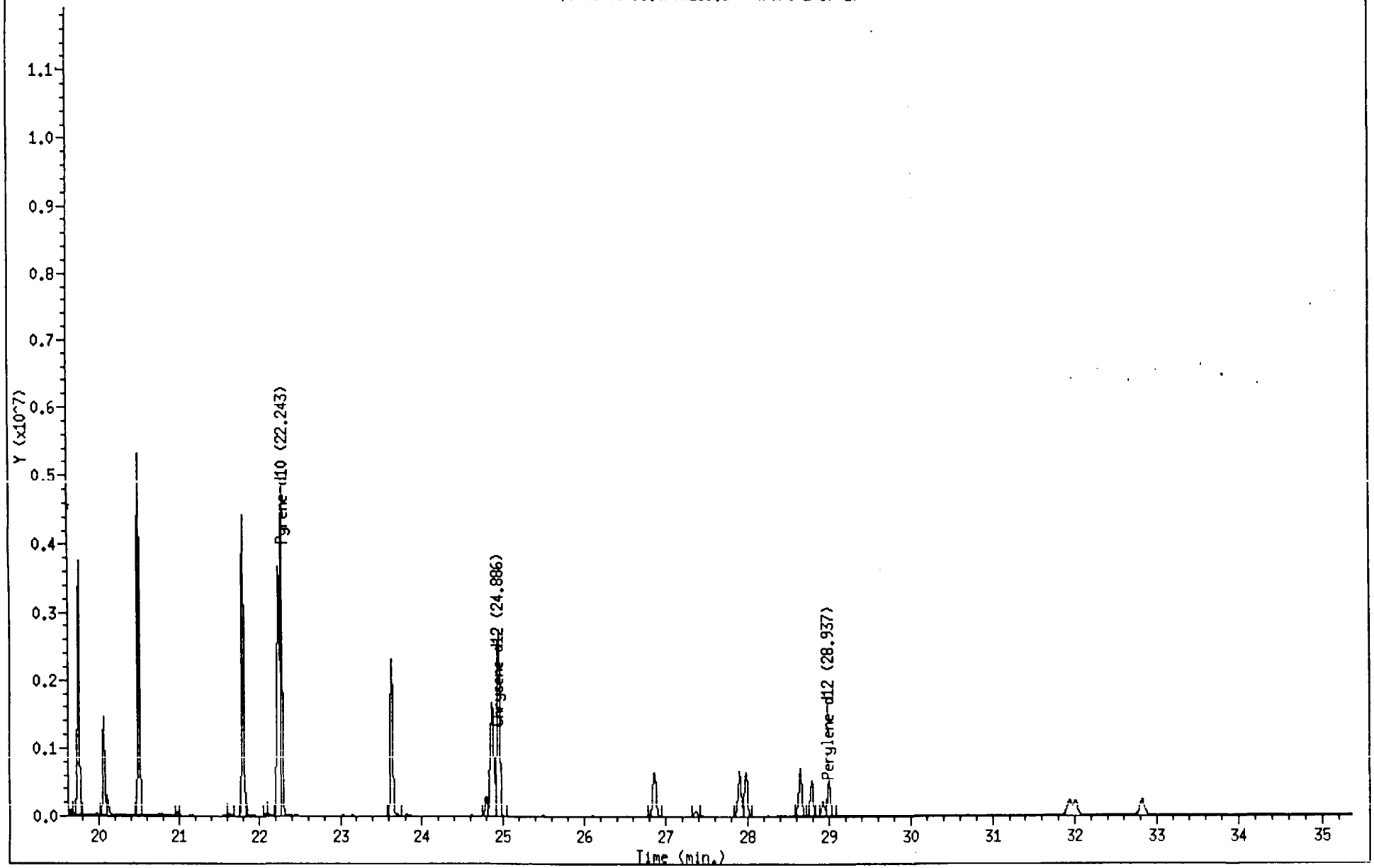
Data File: /chem/V.i/06-08-98.b/VH269.d
Date : 08-JUN-1998 15:47
Instrument : V.i
Sample ID : LCS DUP
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

/chem/V.i/06-08-98.b/VH269.d (Part 2 of 2)



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH269.d
Lab. Id. : 45869
Inj Date : 08-JUN-1998 15:47 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : LCS DUP
Misc Info :
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 2
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.989(1.000)	522979	40.00	
*	2 Naphthalene-d8	136.00	12.526(1.000)	1828233	40.00	
*	3 Acenaphthene-d10	164.00	16.164(1.000)	1041347	40.00	
*	4 Phenanthrene-d10	188.00	19.268(1.000)	1678361	40.00	
*	5 Chrysene-d12	240.00	24.886(1.000)	496489	40.00	
*	6 Perylene-d12	264.00	28.930(1.000)	241493	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.257(0.927)	468272	29.66	29.66
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.077(0.884)	1109613	71.45	71.45
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.758(0.939)	1219328	77.26	77.26
$	13 1,4-Dibromobenzene-d4	240.00	12.633(1.265)	992103	84.24	84.24
$	14 2-Fluorobiphenyl	172.00	14.764(0.913)	2502001	83.69	83.69
$	15 2,4,6-Tribromophenol	329.80	17.808(1.102)	413973	69.23	69.23
$	16 Anthracene-d10	188.00	19.390(1.006)	3232673	93.73	93.73
$	17 Pyrene-d10	212.00	22.243(0.894)	2760536	121.65	121.65(R)
$	18 Terphenyl-d14	Compound Not Detected.				
	42 Naphthalene	128.00	12.576(1.004)	3334871	75.88	75.88
	46 2-Methylnaphthalene	142.00	13.995(1.117)	2317829	85.38	85.38
	50 2-Chloronaphthalene	162.00	15.006(0.928)	2184878	84.28	84.28
	56 Acenaphthylene	152.00	15.873(0.982)	3438179	79.11	79.11
	58 Acenaphthene	154.00	16.236(1.004)	2150954	86.16	86.16
	65 Fluorene	166.00	17.317(1.071)	3021415	91.04	91.04
	73 Phenanthrene	178.00	19.326(1.003)	3552840	92.37	92.37
	74 Anthracene	178.00	19.433(1.009)	3149777	87.77	87.77
	77 Fluoranthene	202.00	21.803(1.132)	3268154	83.22	83.22
	78 Pyrene	202.00	22.287(0.896)	3245853	112.57	112.57
	83 Benzo(a)anthracene	228.00	24.857(0.999)	1304494	82.55	82.55
	84 Chrysene	228.00	24.951(1.003)	1187935	88.13	88.13
	86 Benzo(b)fluoranthene	252.00	27.901(0.964)	709574	84.27	84.27

Data Review: PaB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	252.00	27.980(0.967)	627226	88.98	88.98
88 Benzo(e)pyrene	252.00	28.653(0.990)	699301	98.68	98.68
89 Benzo(a)pyrene	252.00	28.788(0.995)	517745	86.88	86.88
90 Perylene	252.00	29.001(1.002)	487507	94.29	94.29
91 Indeno(1,2,3-cd)pyrene	276.00	31.934(1.104)	414153	83.94	83.94(M)
92 Dibenz(a,h)anthracene	278.00	32.005(1.106)	286947	82.14	82.14
93 Benzo(g,h,i)perylene	276.00	32.821(1.134)	394645	90.88	90.88

QC Flag Legend

R - Spike/Surrogate failed recovery limits.
M - Compound response manually integrated.

Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH277

Method 8270A PUF
Sample ID: SITE#1 5-27-98 24hour

Client Project: Hull & Assoc	Date Received: 06/02/98	Response File: VH267
TLI ID: 209-67-9,14	Date Extracted: 06/04/98	
	Date Analyzed : 06/08/98	
	Dilution Factor: 1.00	

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	8.87	BJ	12.56		10
Acenaphthene-d_{10}		IS 3	16.15		
Fluorene	3.50	J	17.29		10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene	14.49	B	19.31		10
Anthracene	0.37	J	19.42		10
Fluoranthene	2.25	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.28	10

Surrogate Summary	Amount (ug)	RT	IS Ref.	%REC
Anthracene-d_{10}	91.21	19.38	4	91
Pyrene-d_{10}	119.52	22.23	5	120
Terphenyl-d_{14}	102.66	22.60	5	103

Reviewed by PAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

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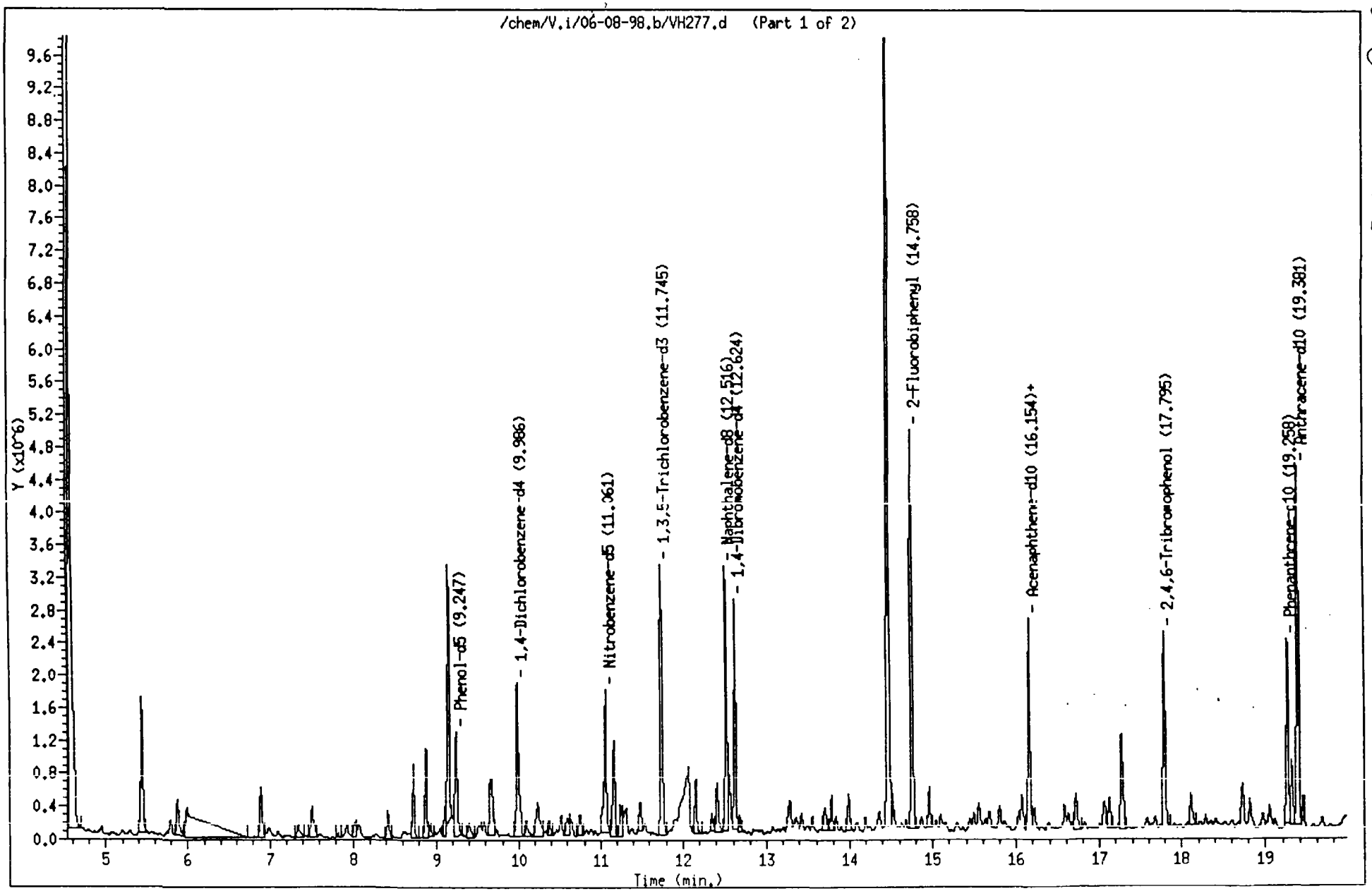
Savar v3.7
Printed: 15:15 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH277.d
Date : 08-JUN-1998 22:21
Instrument : V.i
Sample ID : SITE#1 5-27-98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

48

Column diameter : 0.32



Data Review: PAB
Date: 6/12/98

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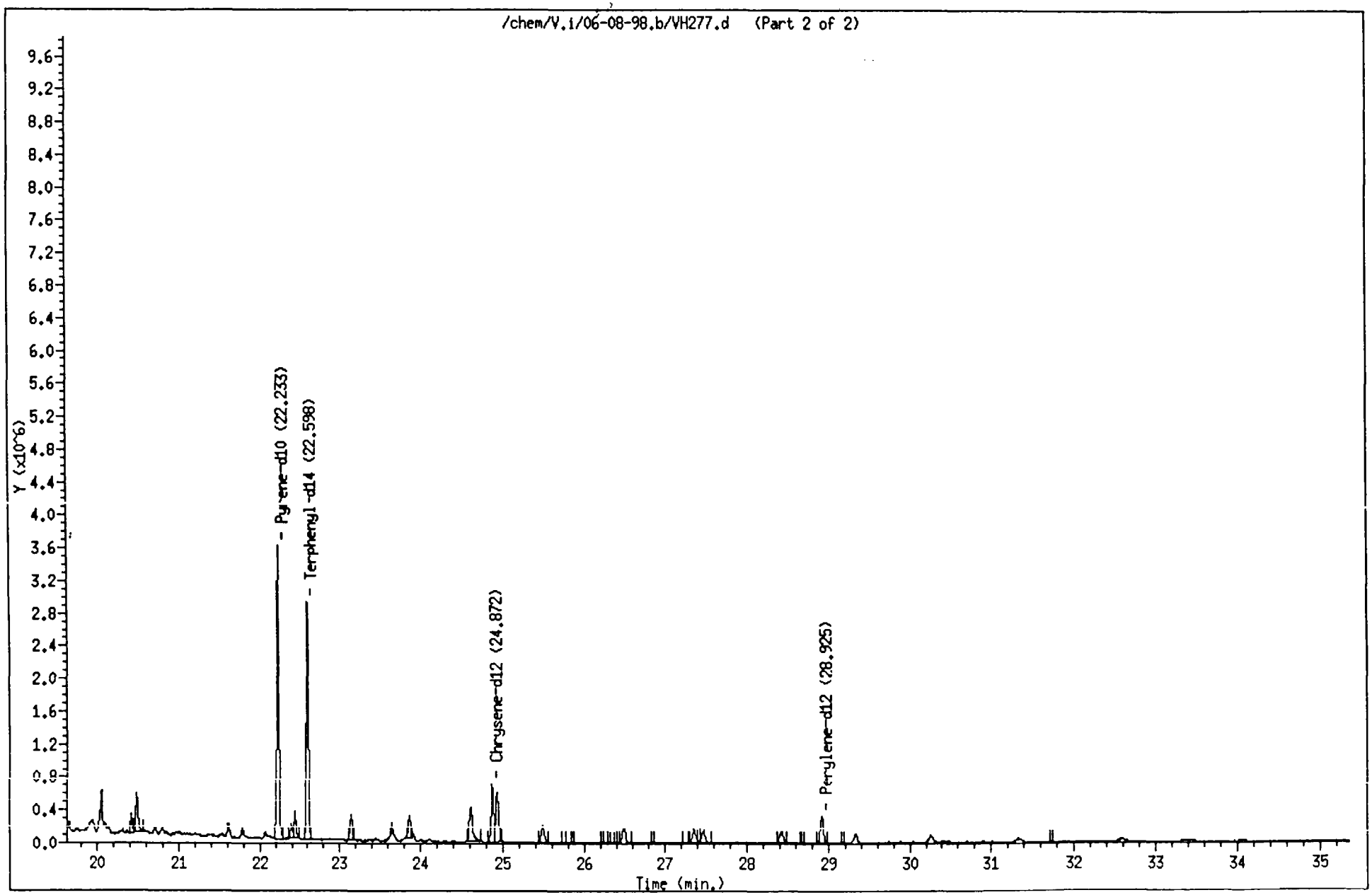
Data File: /chem/V.i/06-08-98.b/VH277.d
Date : 08-JUN-1998 22:21
Instrument : V.i
Sample ID : SITE#1 5-27-98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories
of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH277.d
Lab. Id. : 45869
Inj Date : 08-JUN-1998 22:21 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#1 5-27-98 24HOUR
Misc Info : 209-67-9,14
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 10
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

		Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1	1,4-Dichlorobenzene-d4	152.00	9.986(1.000)	473324	40.00	
*	2	Naphthalene-d8	136.00	12.516(1.000)	1764091	40.00	
*	3	Acenaphthene-d10	164.00	16.154(1.000)	911878	40.00	
*	4	Phenanthrene-d10	188.00	19.258(1.000)	1477404	40.00	
*	5	Chrysene-d12	240.00	24.872(1.000)	422002	40.00	
*	6	Perylene-d12	264.00	28.925(1.000)	315723	40.00	
$	7	2-Fluorophenol	Compound Not Detected.				
$	8	Phenol-d5	99.00	9.247(0.926)	510772	35.74	35.74
$	9	2-Chlorophenol-d4	Compound Not Detected.				
$	10	1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11	Nitrobenzene-d5	82.00	11.061(0.884)	818154	54.60	54.60
$	12	1,3,5-Trichlorobenzene-d3	185.00	11.745(0.938)	942468	61.89	61.89
$	13	1,4-Dibromobenzene-d4	240.00	12.624(1.264)	758631	71.18	71.18
$	14	2-Fluorobiphenyl	172.00	14.758(0.914)	2164130	82.67	82.67
$	15	2,4,6-Tribromophenol	329.80	17.795(1.102)	377751	72.14	72.14
$	16	Anthracene-d10	188.00	19.381(1.006)	2769142	91.21	91.21
$	17	Pyrene-d10	212.00	22.233(0.894)	2305059	119.50	119.50
$	18	Terphenyl-d14	244.00	22.605(0.909)	1242314	102.69	102.69
	42	Naphthalene	128.00	12.559(1.003)	376319	8.87	8.87(a)
	46	2-Methylnaphthalene	142.00	13.986(1.117)	168519	6.43	6.43(a)
	50	2-Chloronaphthalene	Compound Not Detected.				
	56	Acenaphthylene	152.00	~~16.140(0.999)~~	~~7036~~	~~0.18~~	~~0.18(a)~~ JP PAB
	58	Acenaphthene	154.00	16.219(1.004)	58501	2.68	2.68(a)
	65	Fluorene	166.00	17.294(1.071)	101826	3.50	3.50(a)
	73	Phenanthrene	178.00	19.309(1.003)	490601	14.49	14.49
	74	Anthracene	178.00	19.42 ~~19.309(1.003)~~	~~490601~~ 117861	~~15.53~~	~~15.53~~ (4) PAB
	77	Fluoranthene	202.00	21.792(1.132)	77743	2.25	2.25(a)
	78	Pyrene	Compound Not Detected.				
	83	Benzo(a)anthracene	Compound Not Detected.				
	84	Chrysene	Compound Not Detected.				
	86	Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-08-98.b/VH277.d

Date : 08-JUN-1998 22:21

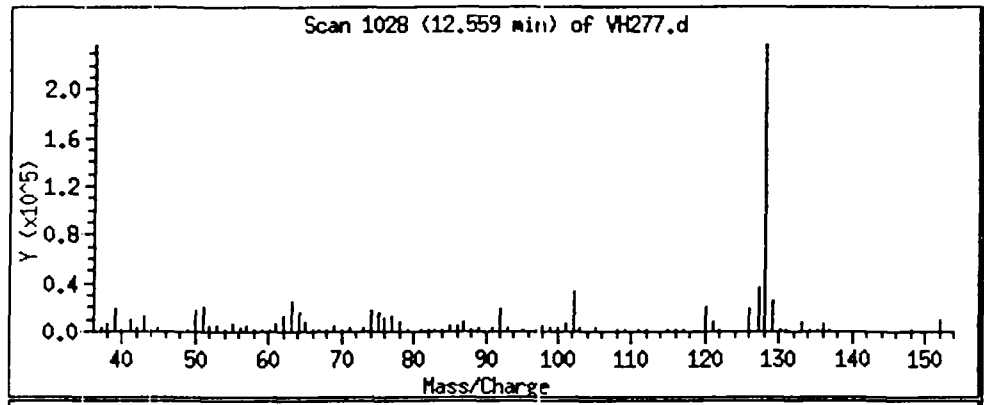
Instrument : V.i

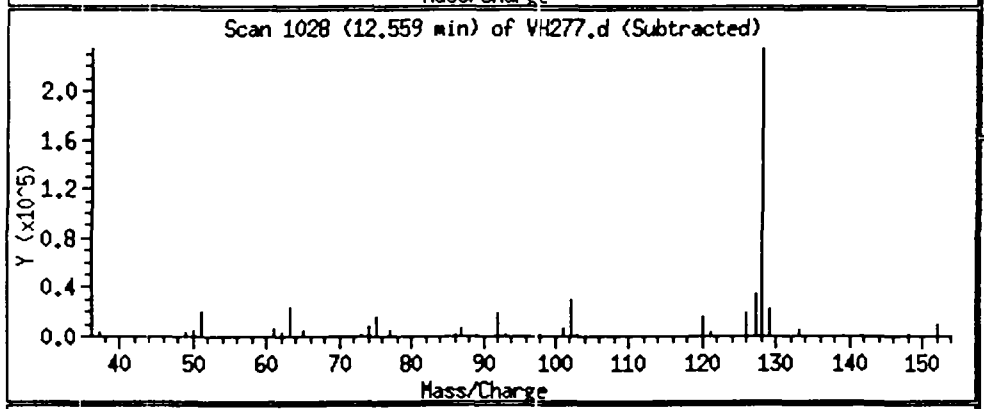
Sample ID : SITE#1 5-27-98 24HR

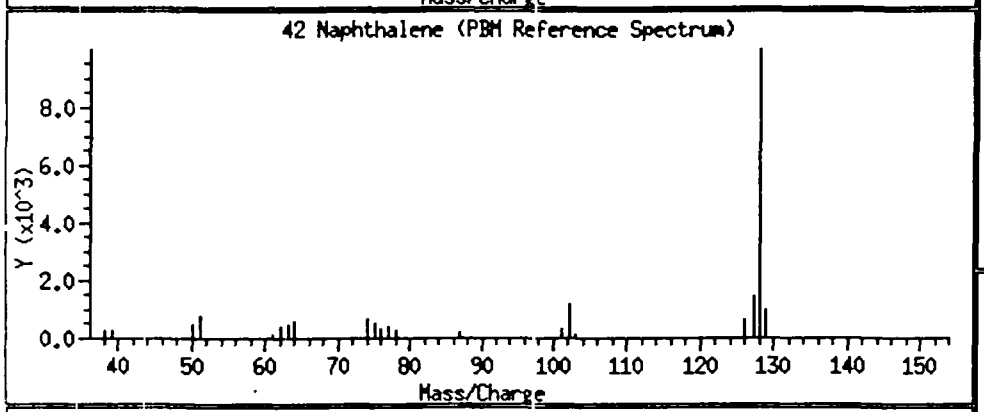
Column phase : DB-5 Column diameter : 0.32

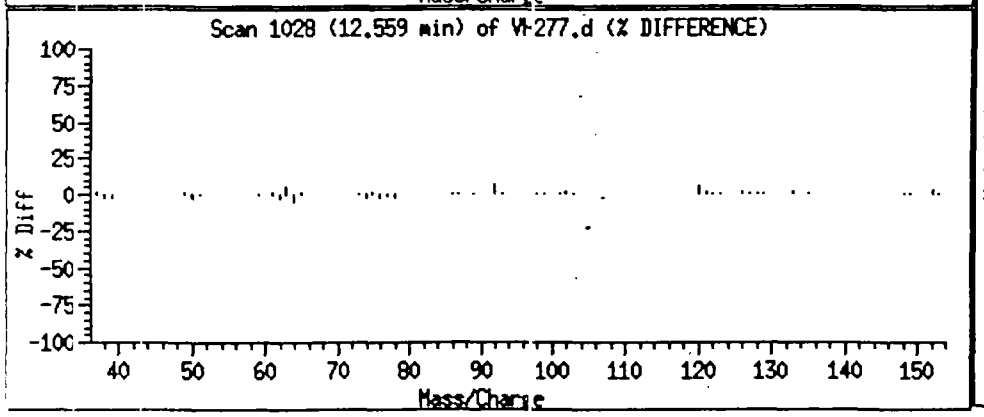
Volume Injected (uL) : 1.0

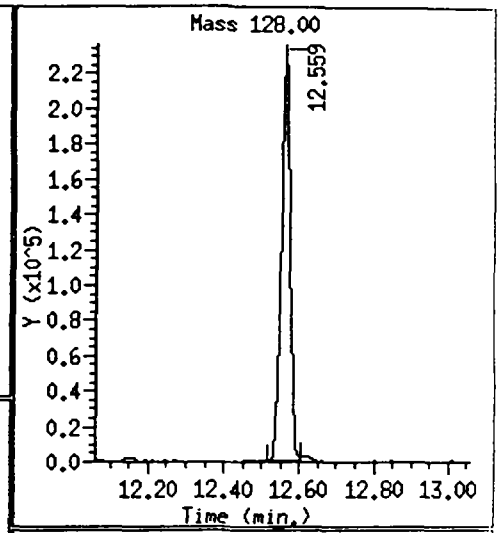
42 Naphthalene

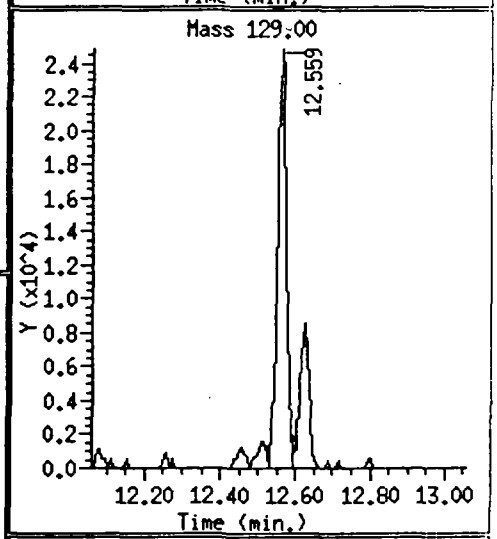


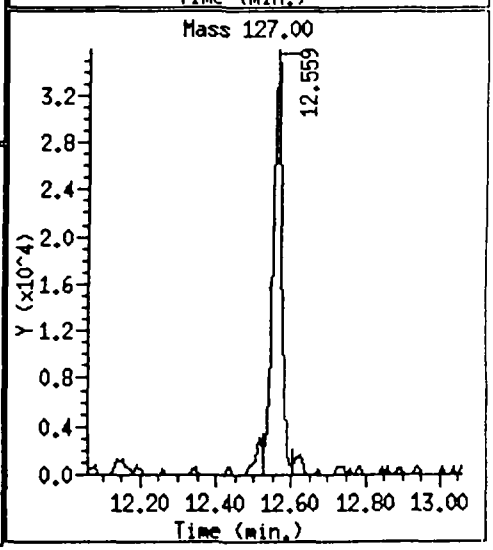












Data File: /chem/V.i/06-08-98.b/VH277.d

Date : 08-JUN-1998 22:21

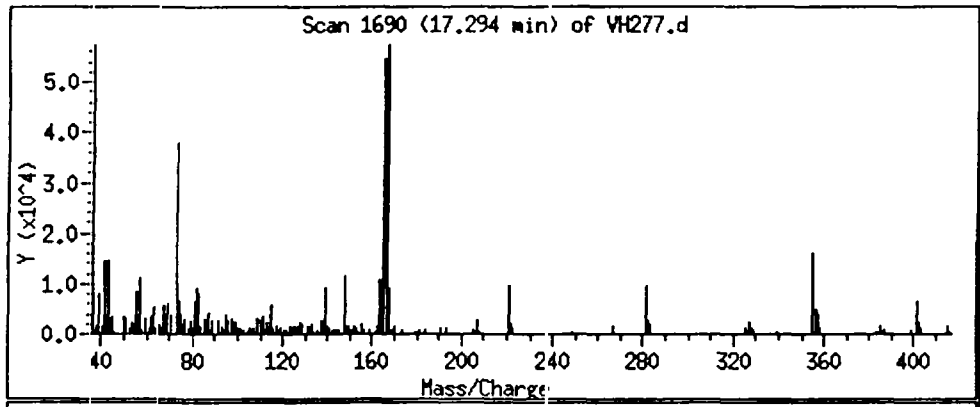
Instrument : V.i

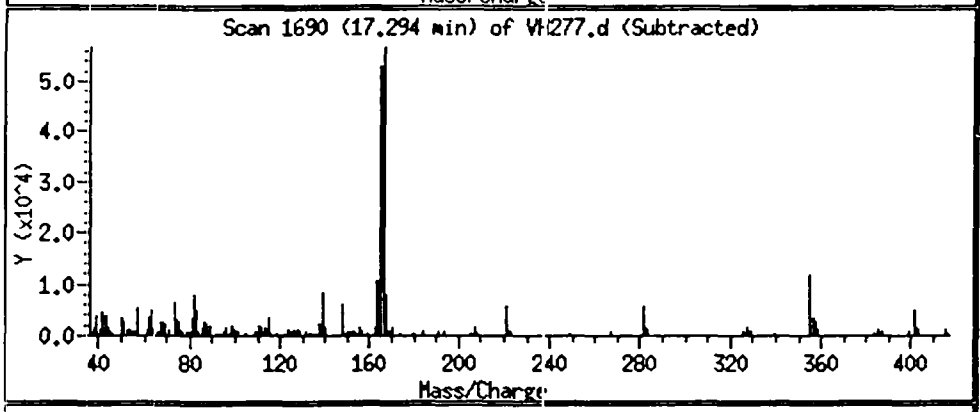
Sample ID : SITE#1 5-27-98 24HR

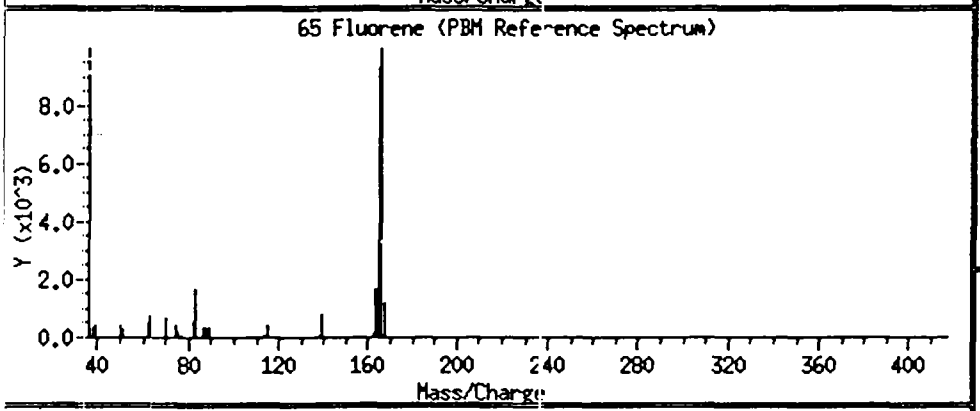
Column phase : DB-5 Column diameter : 0.32

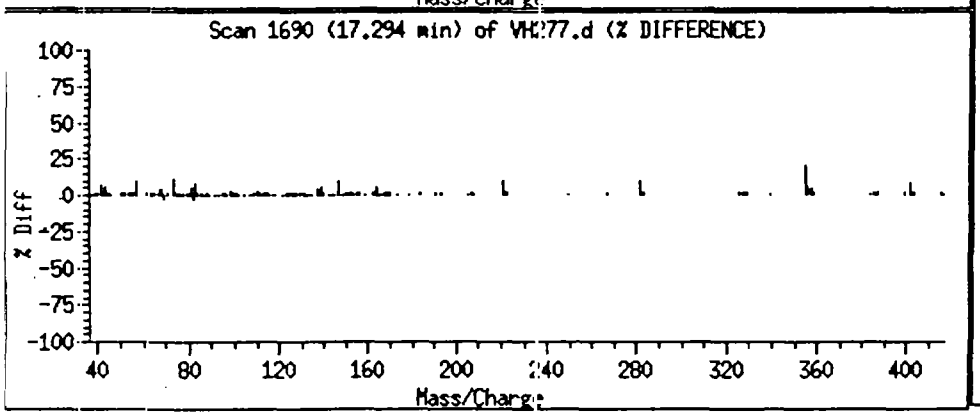
Volume Injected (uL) : 1.0

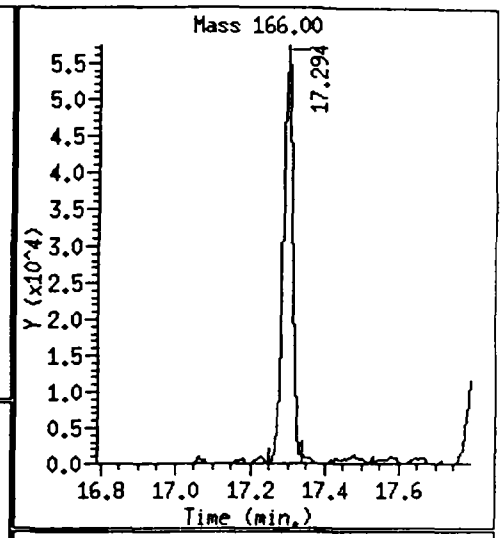
65 Fluorere

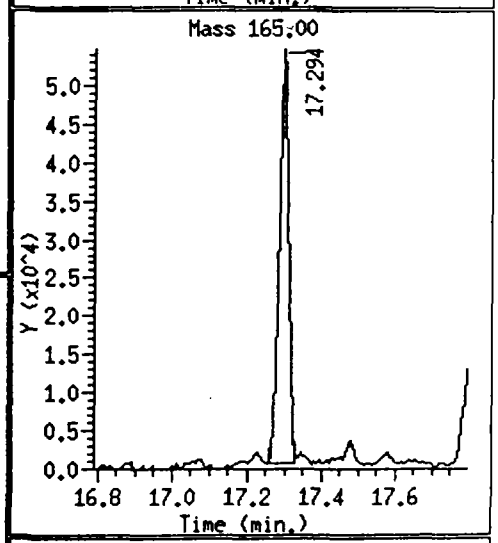


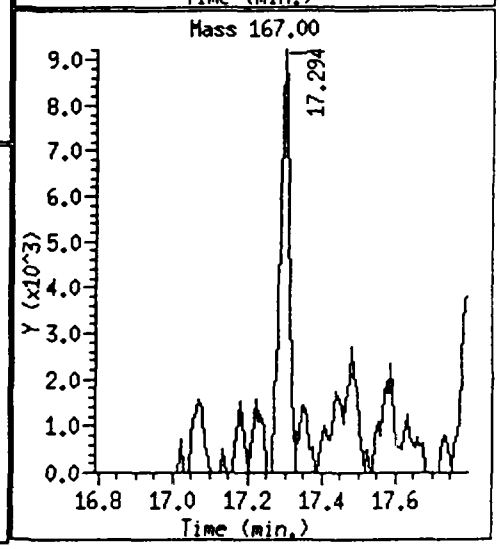












Data File: /chem/V.i/06-08-98.b/VH277.d

Date : 08-JUN-1998 22:21

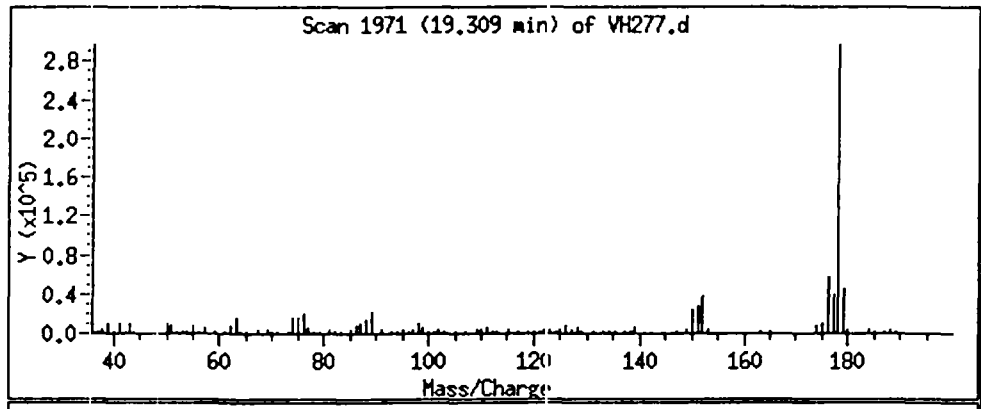
Instrument : V.i

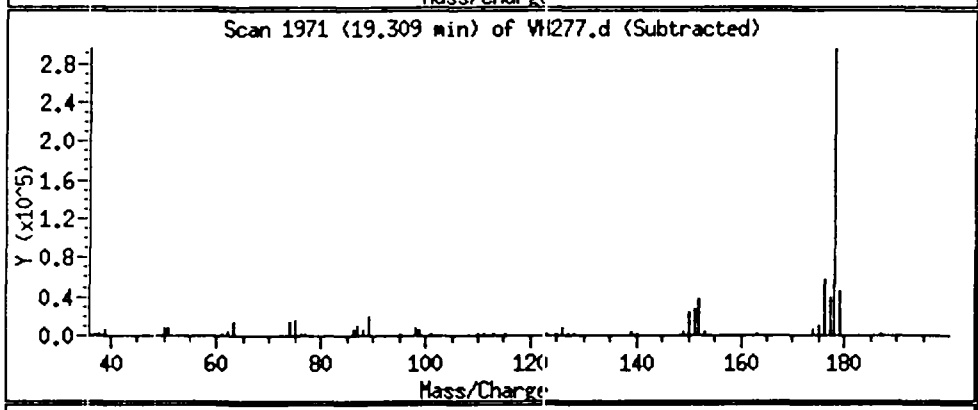
Sample ID : SITE#1 5-27-98 24HR

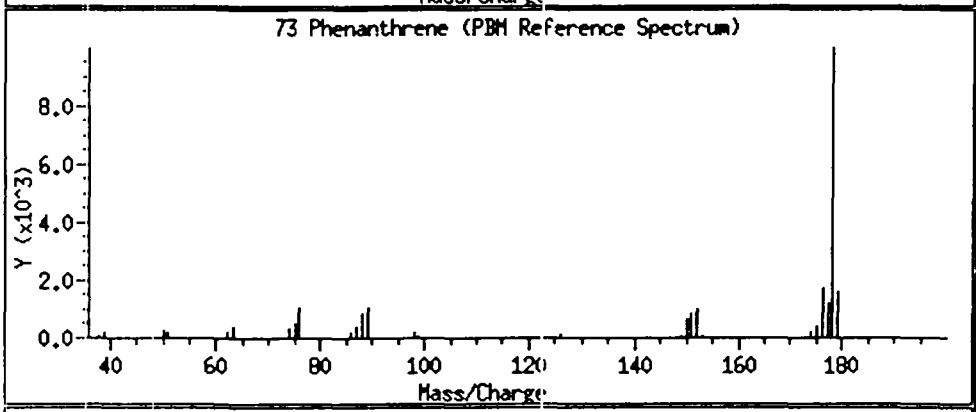
Column phase : DB-5 Column diameter : 0.32

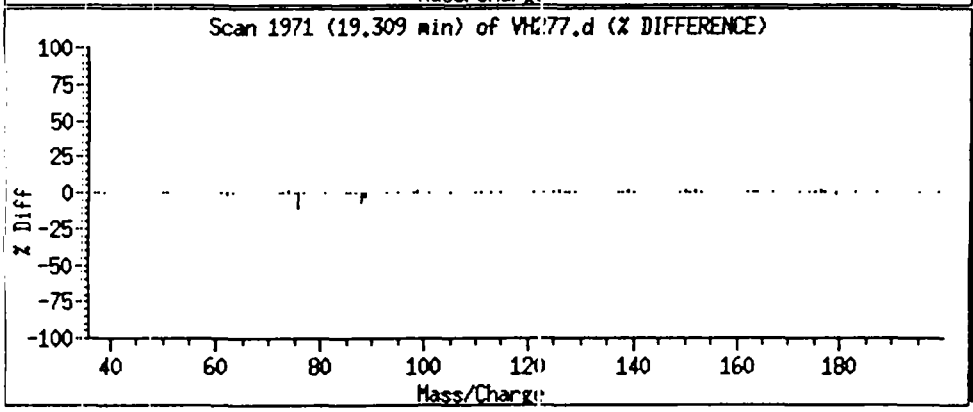
Volume Injected (uL) : 1.0

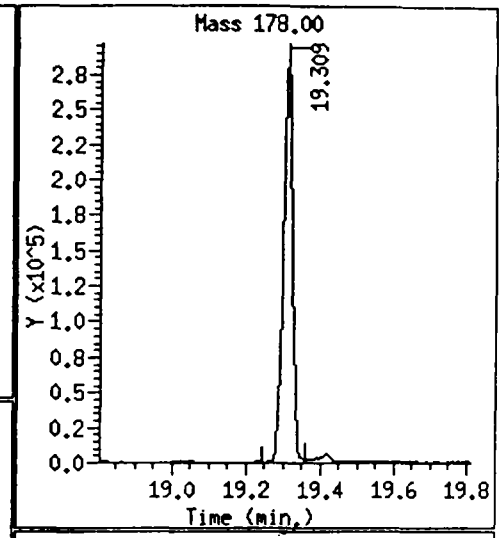
73 Phenanthrene

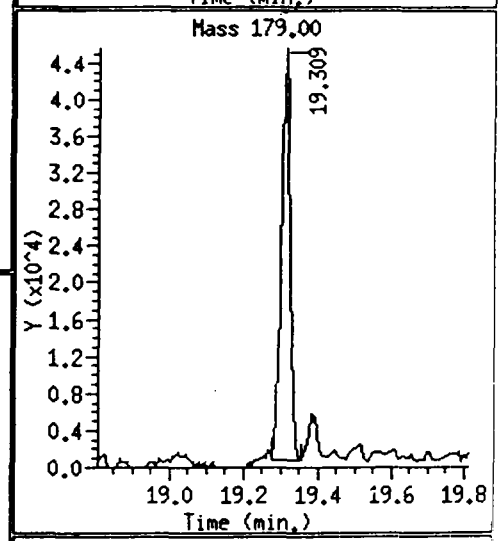


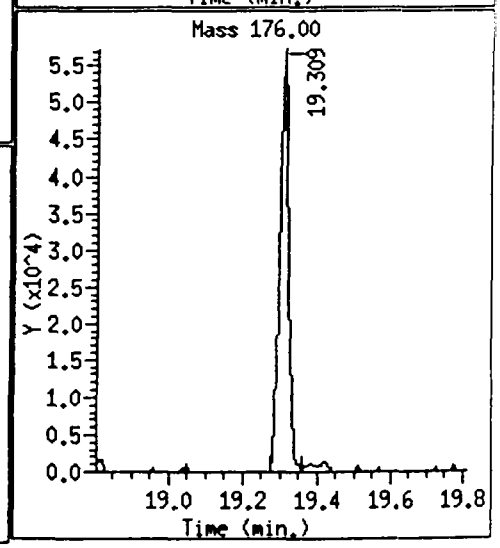












Data File: /chem/V.i/06-08-98.b/VH277.d

Date : 08-JUN-1998 22:21

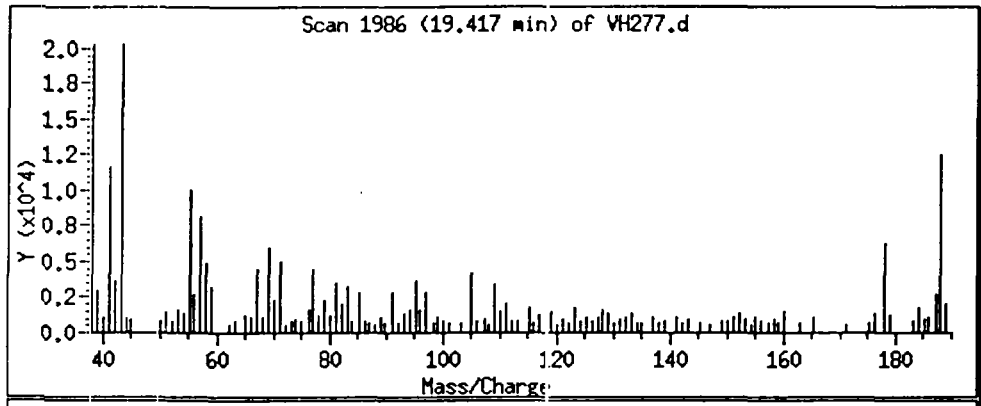
Instrument : V.i

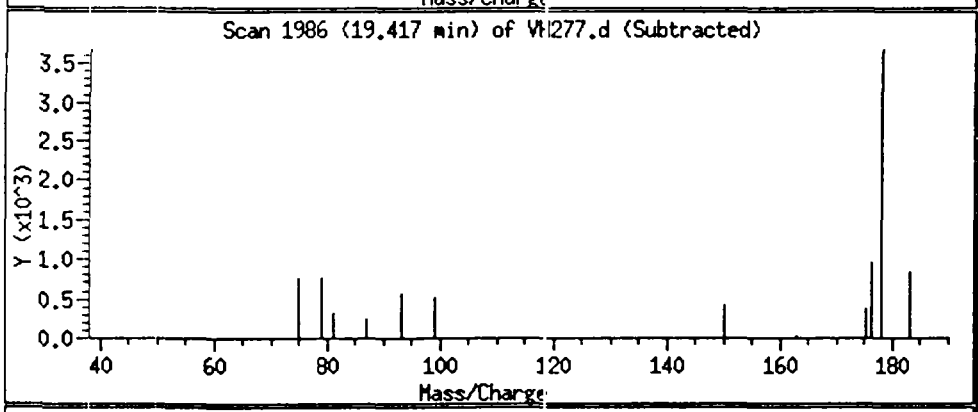
Sample ID : SITE#1 5-27-98 24HR

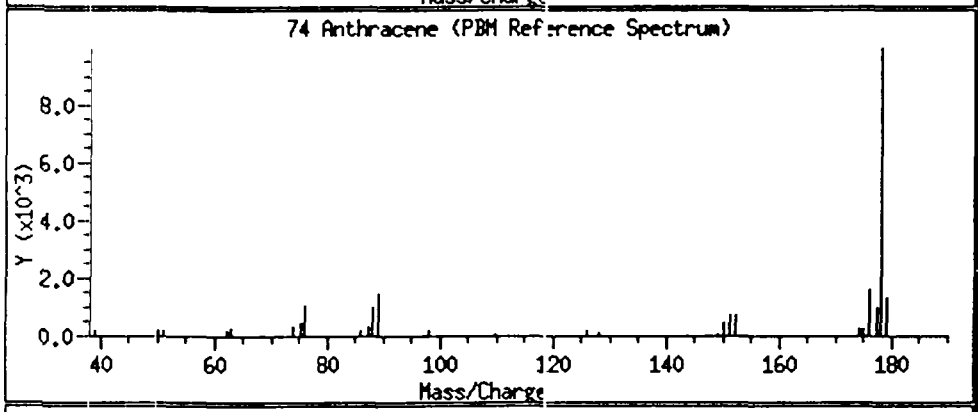
Column phase : DB-5 Column diameter : 0.32

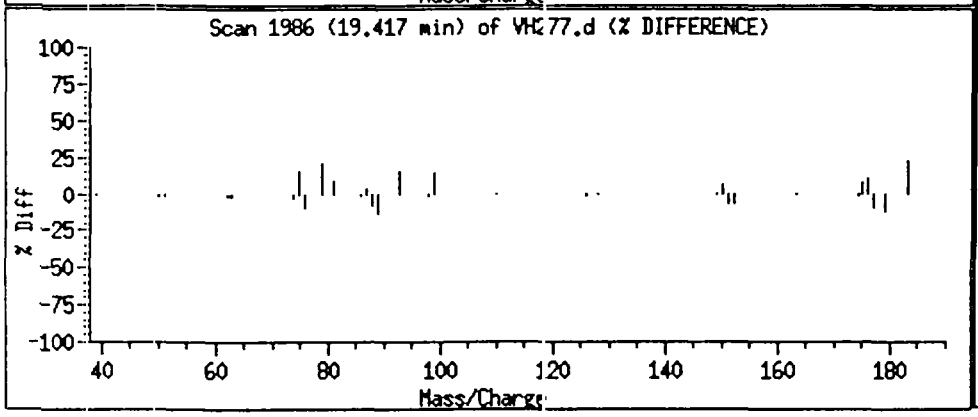
Volume Injected (uL) : 1.0

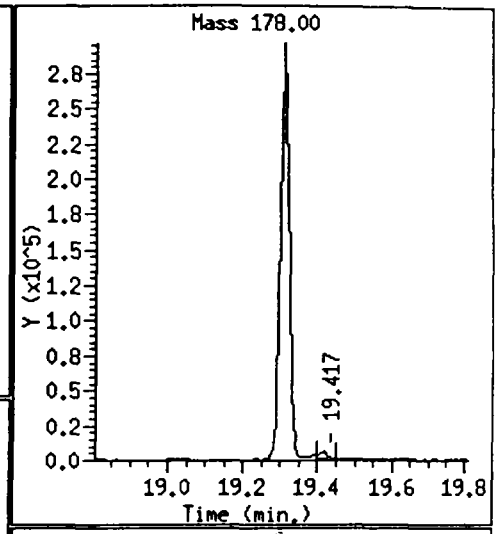
74 Anthracene

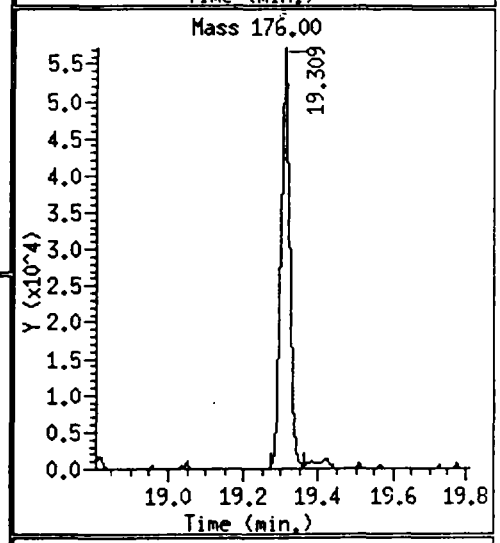


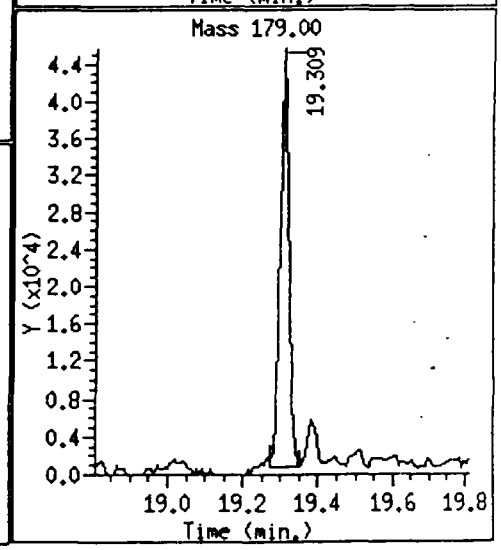












Data File: /chem/V.i/06-08-98.b/VH277.d

Date : 08-JUN-1998 22:21

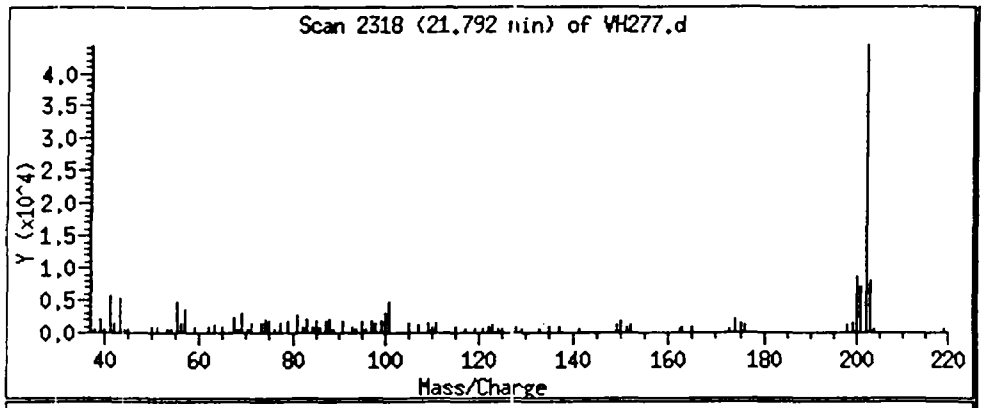
Instrument : V.i

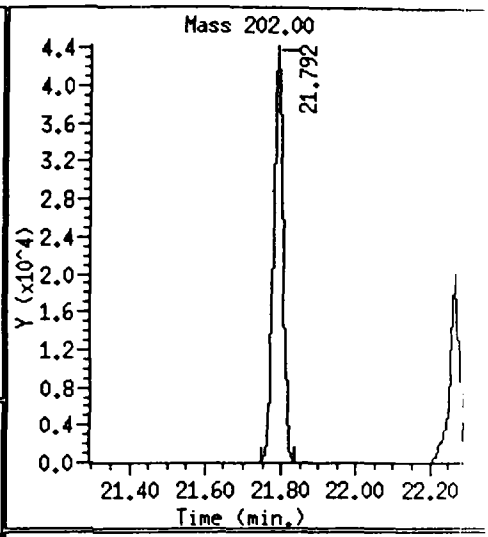
Sample ID : SITE#1 5-27-98 24HR

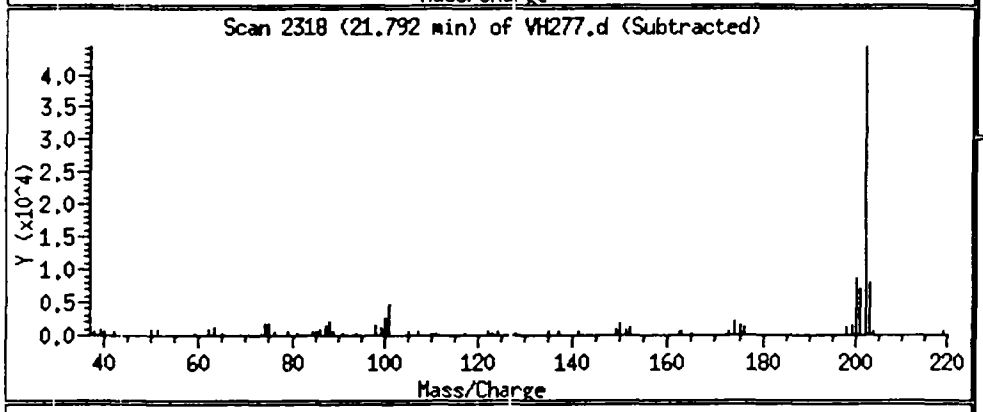
Column phase : DB-5 Column diameter : 0.32

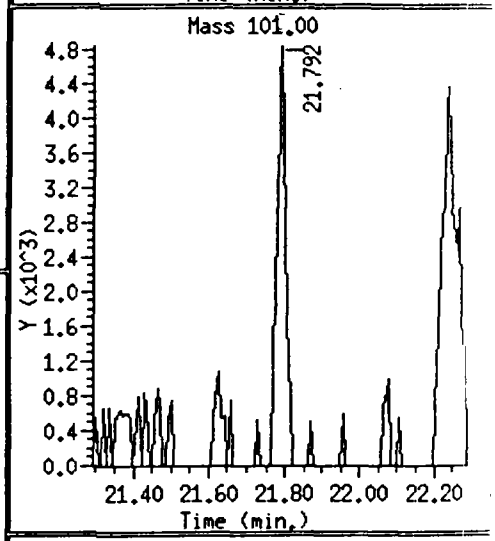
Volume Injected (uL) : 1.0

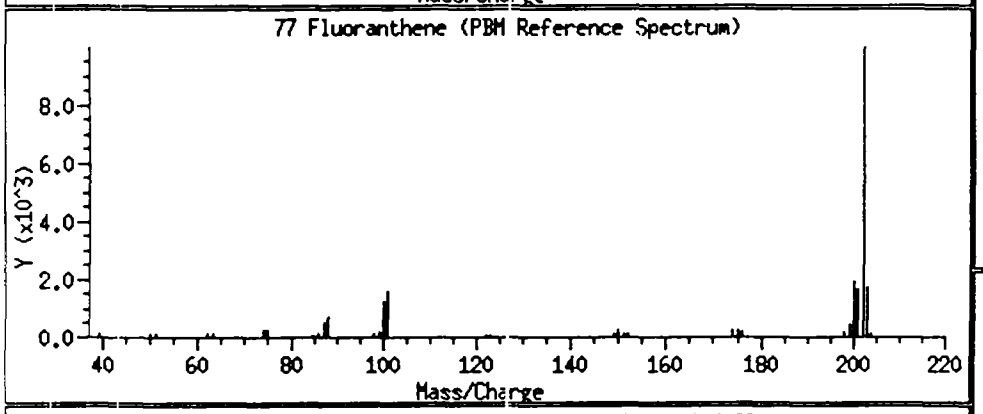
77 Fluoranthene

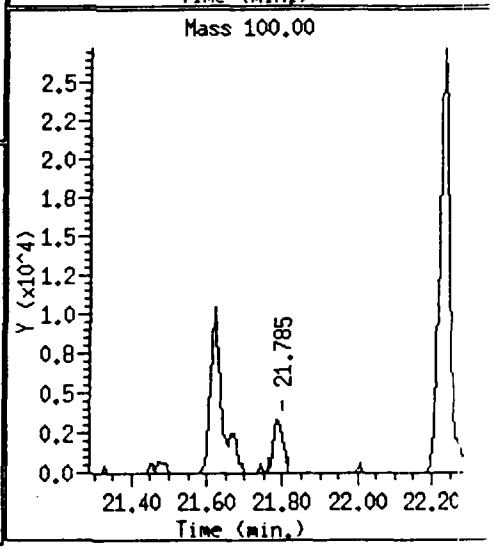


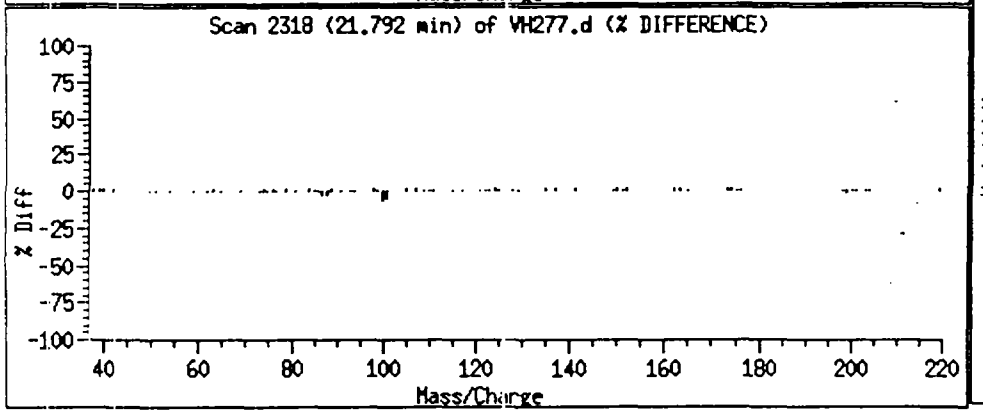












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH273

Method 8270A PUF
Sample ID: SITE#1 5-28-98 8hour

Client Project: Hull & Assoc
TLI ID: 209-67-1,5

Date Received: 06/02/98
Date Extracted: 06/04/98
Date Analyzed : 06/08/98

Response File: VH267

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.51		
Naphthalene	10.82	B	12.56		10
Acenaphthene-d_{10}		IS 3	16.15		
Fluorene	1.68	J	17.29		10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene	3.37	BJ	19.30		10
Anthracene	0.18	J	19.42		10
Fluoranthene	0.67	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.30	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	81.06	19.38	4	81
Pyrene-d_{10}	114.17	22.23	5	114
Terphenyl-d_{14}	98.45	22.60	5	98

Reviewed by PaB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

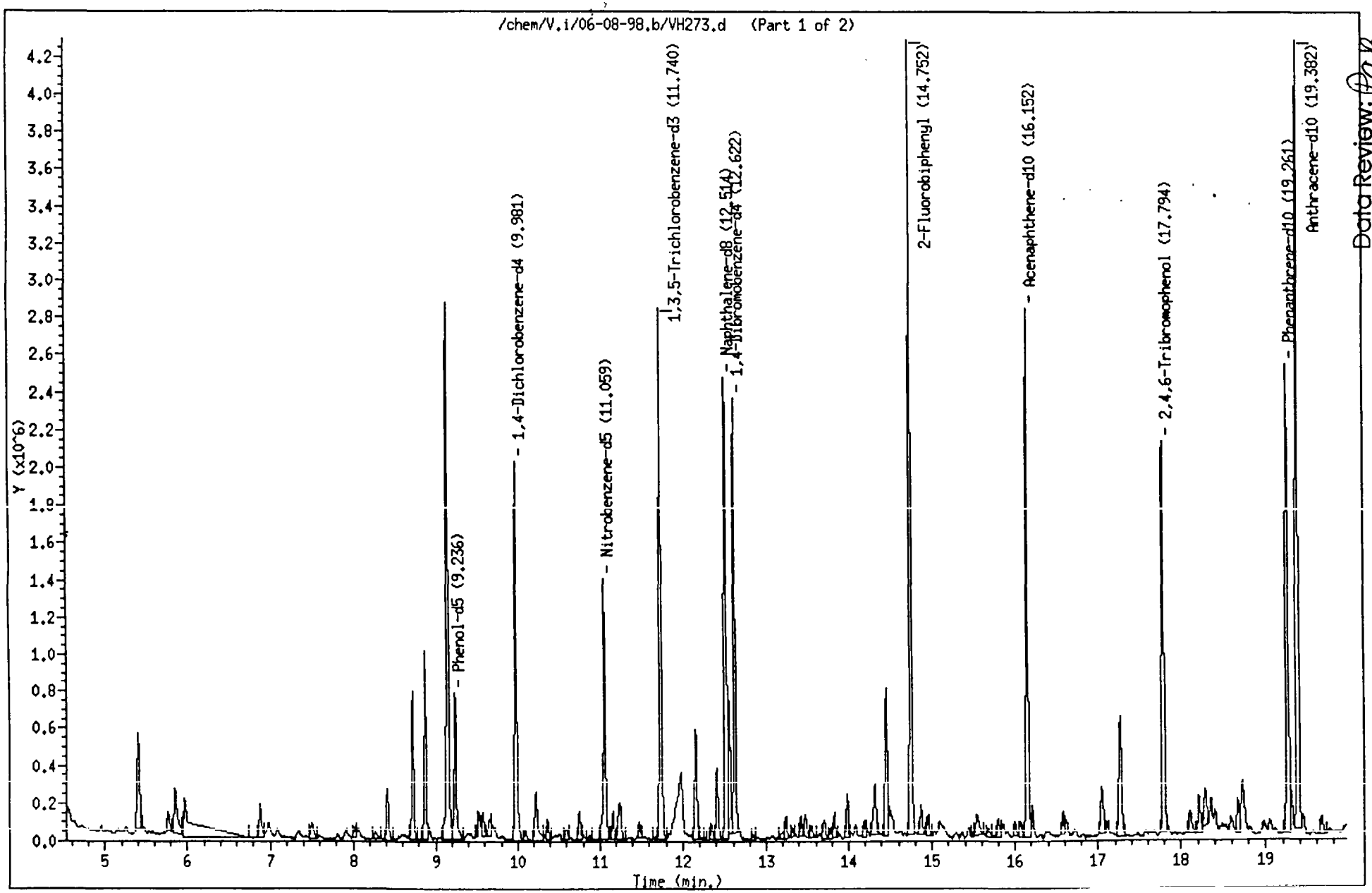
Savar v3.7
Printed: 15:14 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH273.d
Date : 08-JUN-1998 19:04
Instrument : V.i
Sample ID : SITE#1 5-28-98 8HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

58

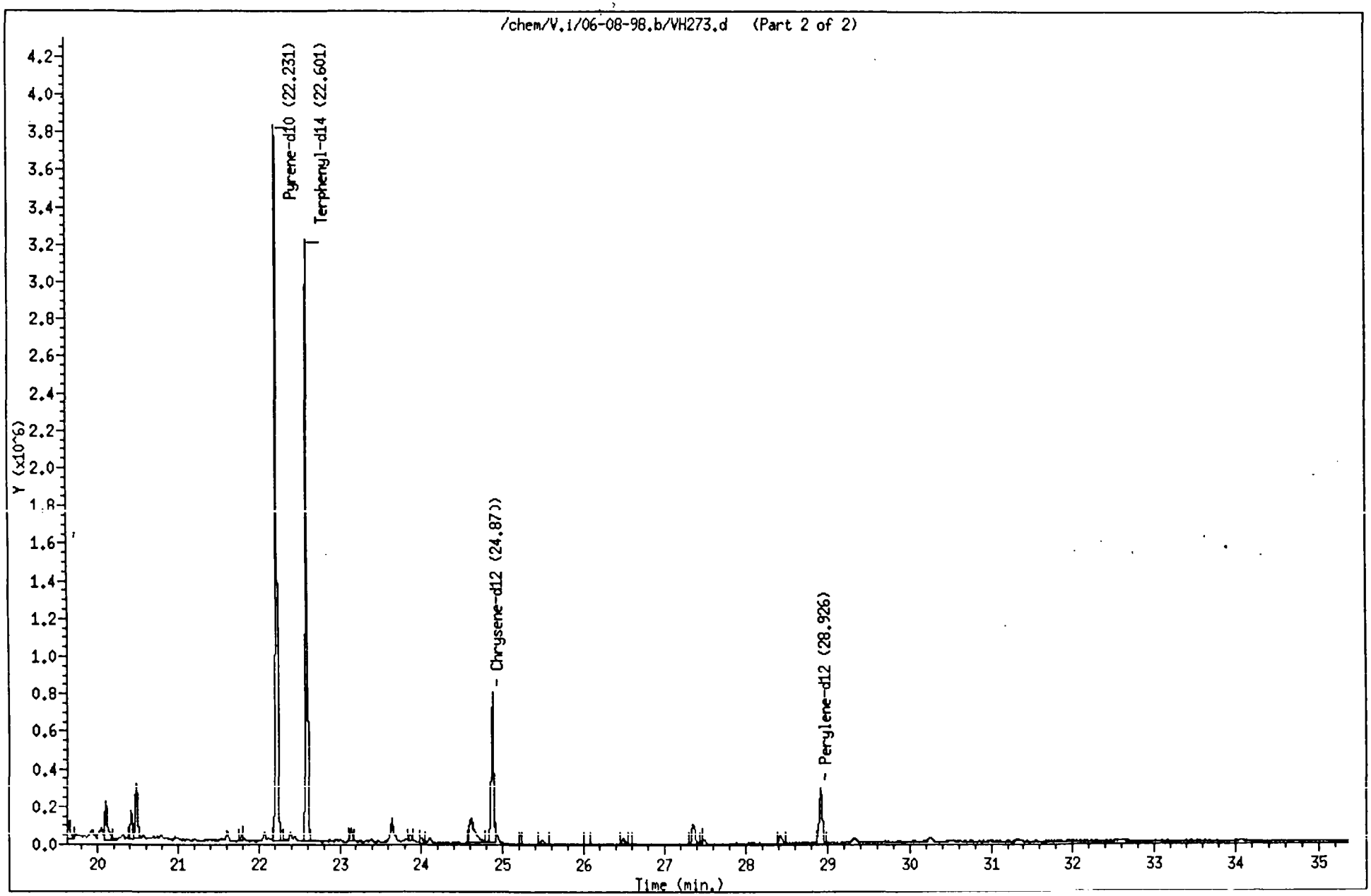


Data Review: PAB
Date: 6/12/98

Data File: /chem/V.i/06-08-98.b/VH273.d
Date : 08-JUN-1998 19:04
Instrument : V.i
Sample ID : SITE#1 5-28-98 8HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH273.d
Lab. Id. : 45869
Inj Date : 08-JUN-1998 19:04 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#1 5-28-98 8HOUR
Misc Info : 209-67-1,5
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 6
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.981(1.000)	502887	40.00	
*	2 Naphthalene-d8	136.00	12.514(1.000)	1794248	40.00	
*	3 Acenaphthene-d10	164.00	16.152(1.000)	995766	40.00	
*	4 Phenanthrene-d10	188.00	19.261(1.000)	1586870	40.00	
*	5 Chrysene-d12	240.00	24.870(1.000)	484133	40.00	
*	6 Perylene-d12	264.00	28.919(1.000)	311078	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.236(0.925)	360237	23.73	23.73
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.059(0.884)	642909	42.18	42.18
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.740(0.938)	792151	51.14	51.14
$	13 1,4-Dibromobenzene-d4	240.00	12.622(1.265)	639706	56.49	56.49
$	14 2-Fluorobiphenyl	172.00	14.752(0.913)	1945359	68.05	68.05
$	15 2,4,6-Tribromophenol	329.80	17.794(1.102)	359443	62.86	62.86
$	16 Anthracene-d10	188.00	19.382(1.006)	2643498	81.06	81.06
$	17 Pyrene-d10	212.00	22.231(0.894)	2526004	114.15	114.15
$	18 Terphenyl-d14	244.00	22.601(0.909)	1366792	98.48	98.48
	42 Naphthalene	128.00	12.557(1.003)	466866	10.82	10.82
	46 2-Methylnaphthalene	142.00	13.984(1.117)	82758	3.11	3.11(a)
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	154.00	16.217(1.004)	44756	1.87	1.87(a)
	65 Fluorene	166.00	17.290(1.070)	53361	1.68	1.68(a)
	73 Phenanthrene	178.00	19.304(1.002)	122719	3.37	3.37(a)
	74 Anthracene	178.00	19.42 ~~19.304(1.002)~~	122719	5959 ~~3.62~~	~~3.62(a)~~ (M) PAB
	77 Fluoranthene	~~Compound Not Detected.~~	21.791		24804	(M) PAB
	78 Pyrene	Compound Not Detected.				
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIM MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-08-98.b/VH273.d

Date : 08-JUN-1998 19:04

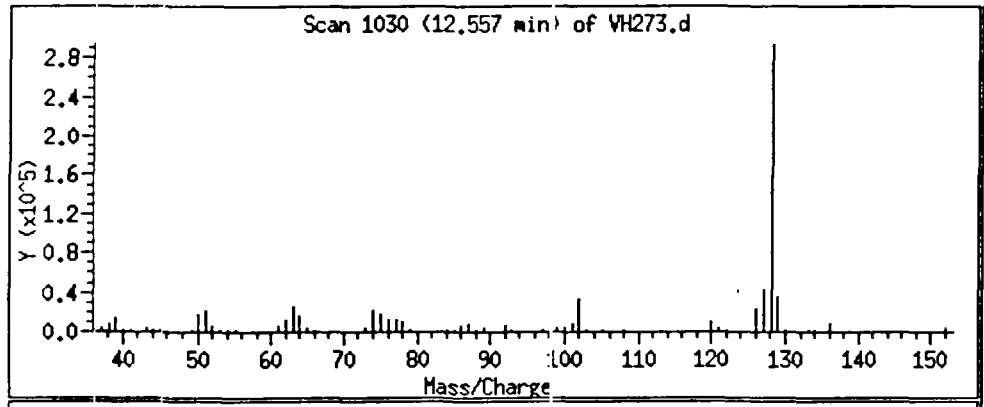
Instrument : V.i

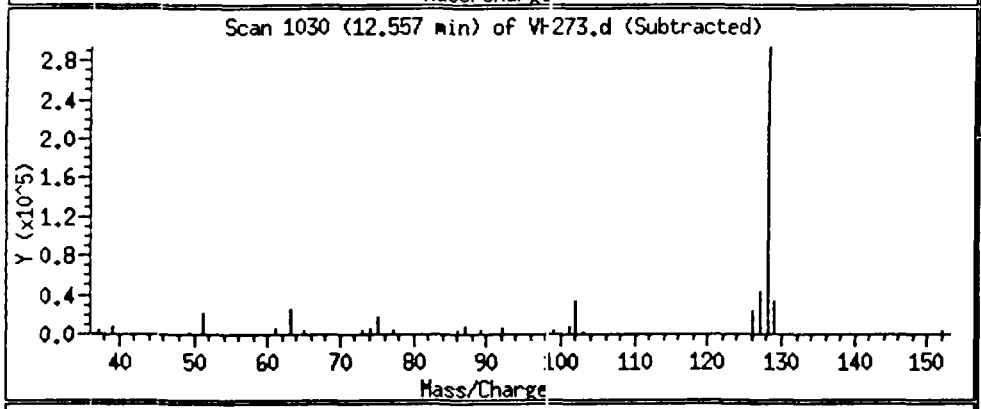
Sample ID : SITE#1 5-28-98 8HR

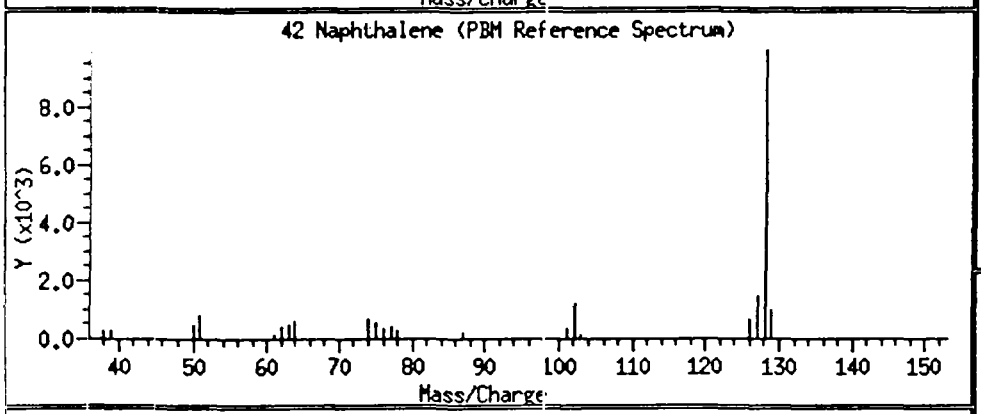
Column phase : DB-5 Column diameter : 0.32

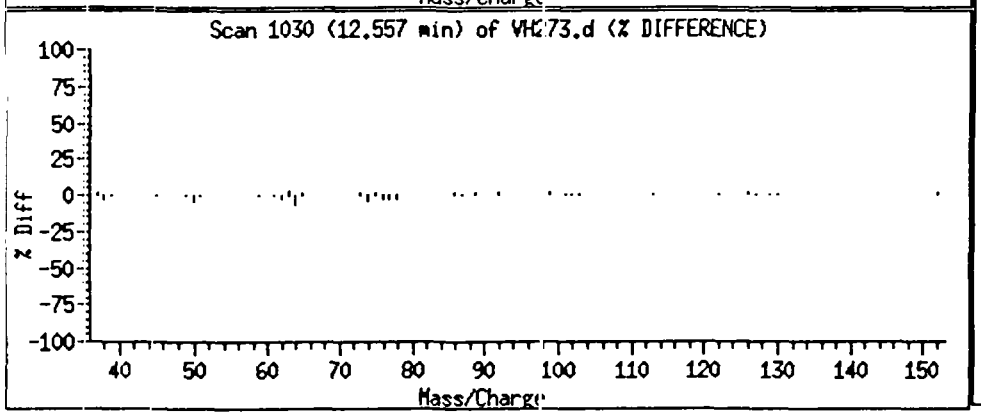
Volume Injected (uL) : 1.0

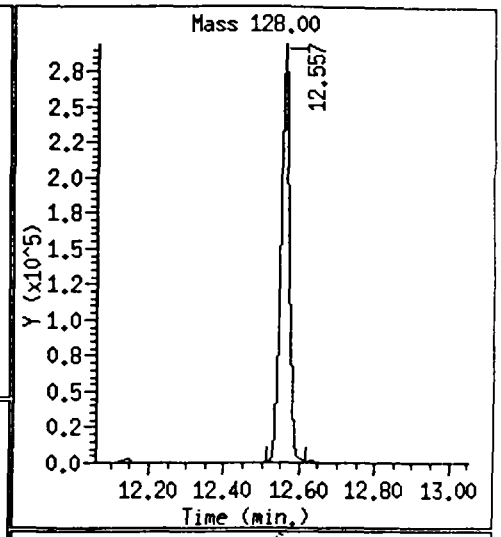
42 Naphthalene

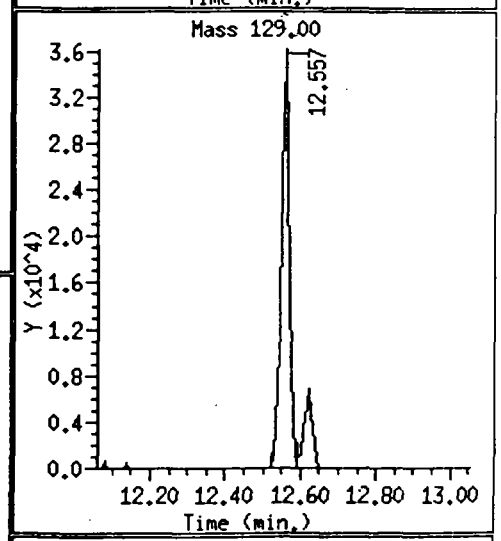


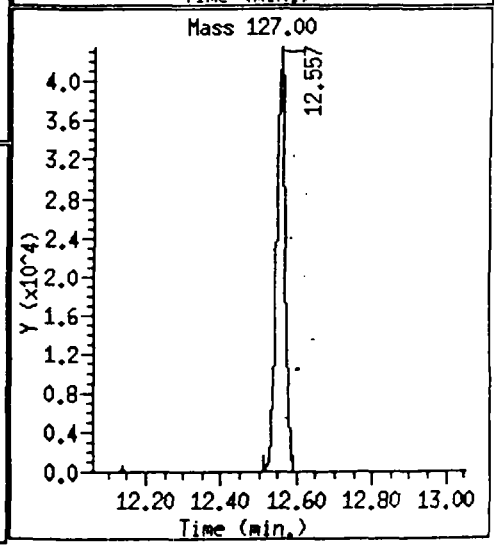












Data File: /chem/V.i/06-08-98.b/VH273.d

Date : 08-JUN-1998 19:04

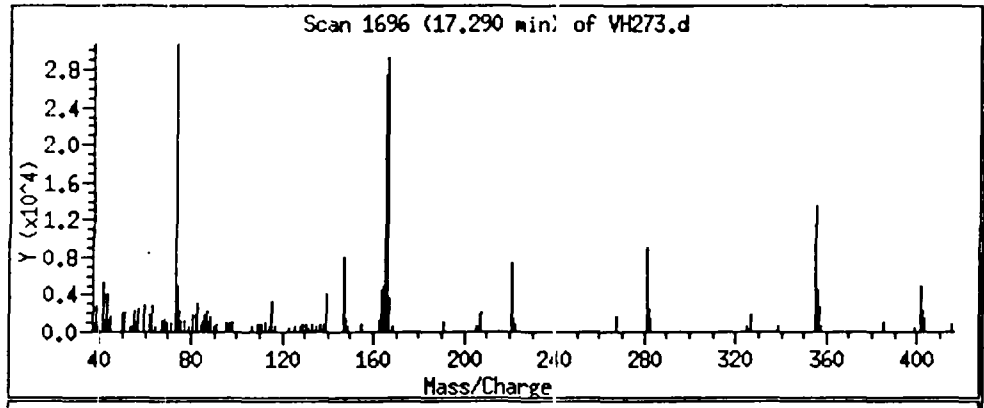
Instrument : V.i

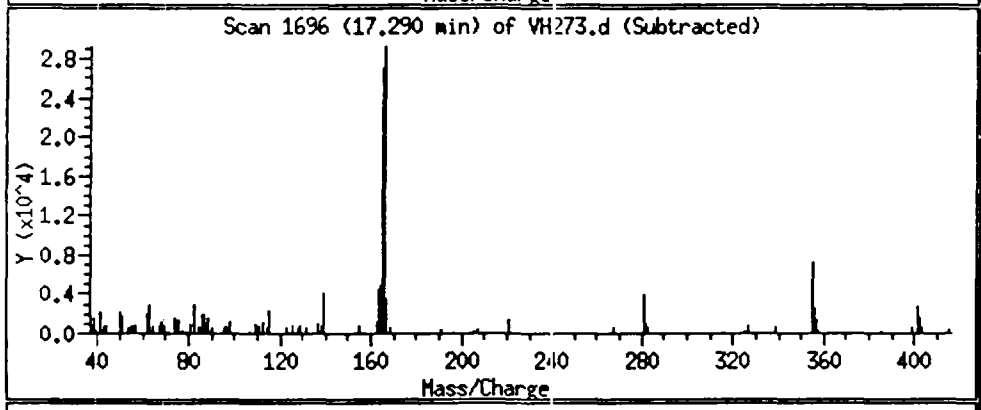
Sample ID : SITE#1 5-28-98 8HR

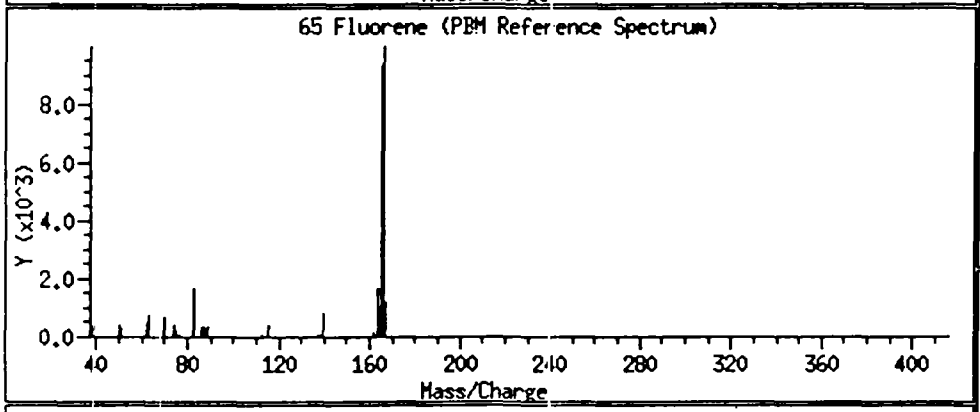
Column phase : DB-5 Column diameter : 0.32

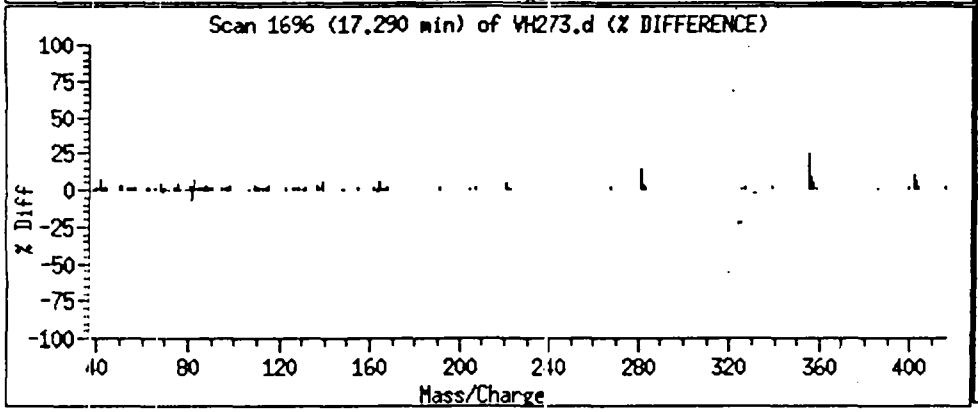
Volume Injected (uL) : 1.0

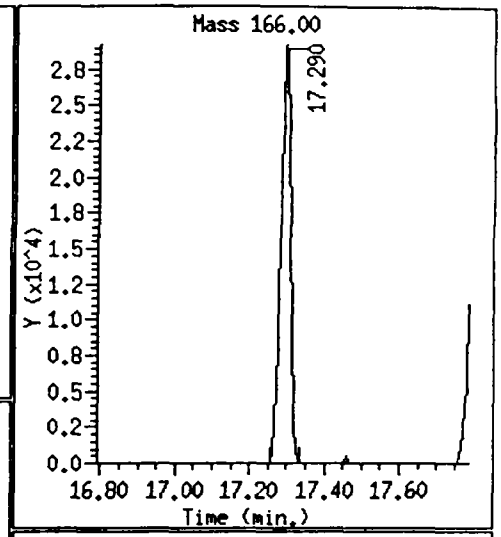
65 Fluorene

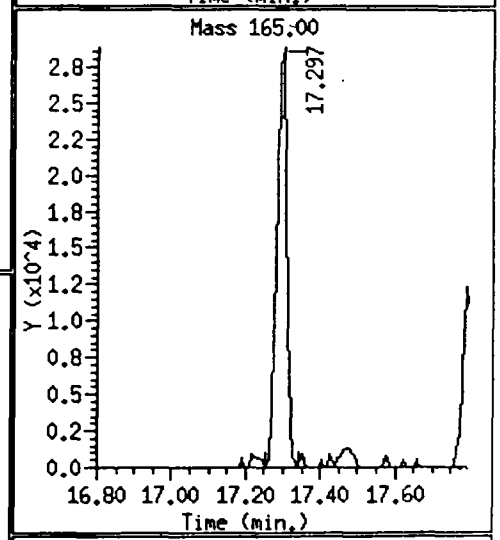


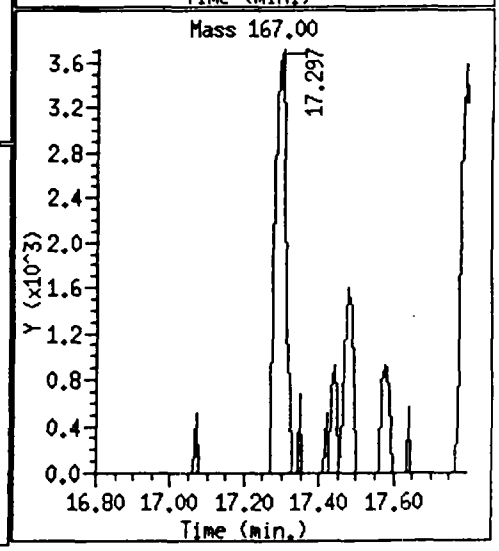












Data File: /chem/V.i/06-08-98.b/VH273.d

Date : 08-JUN-1998 19:04

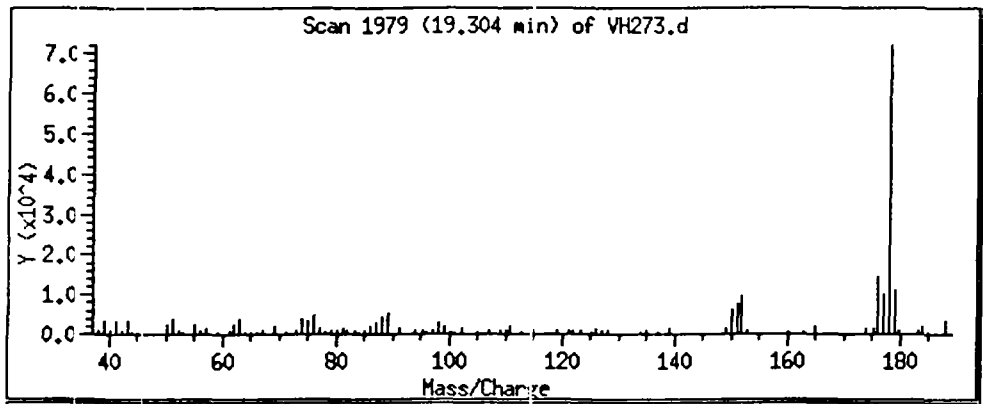
Instrument : V.i

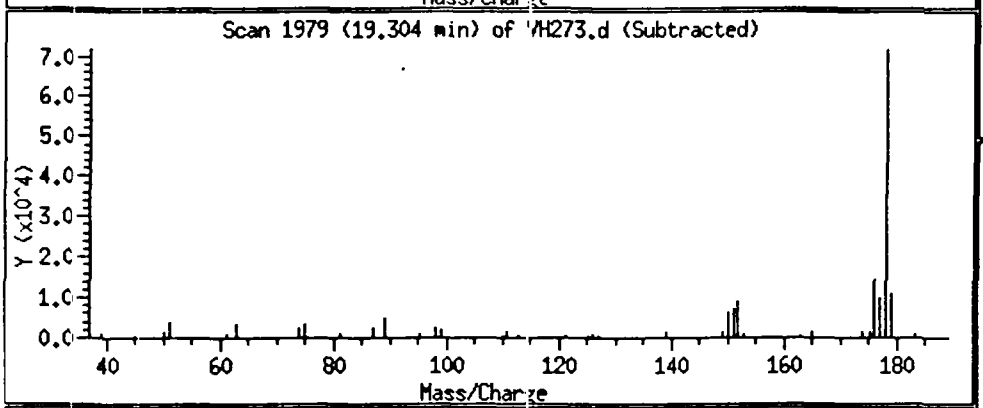
Sample ID : SITE#1 5-28-98 8HR

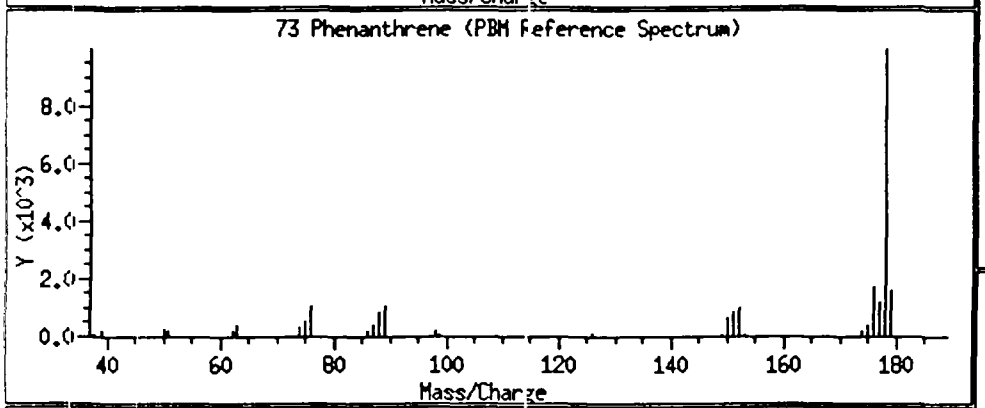
Column phase : DB-5 Column diameter : 0.32

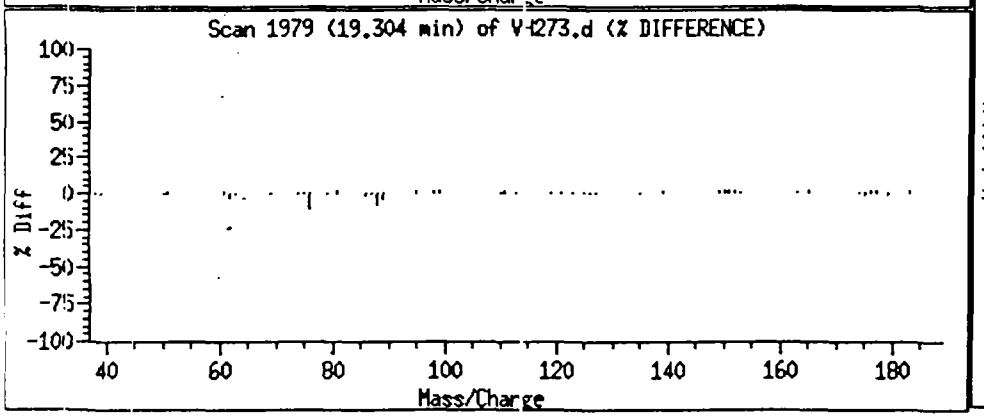
Volume Injected (uL) : 1.0

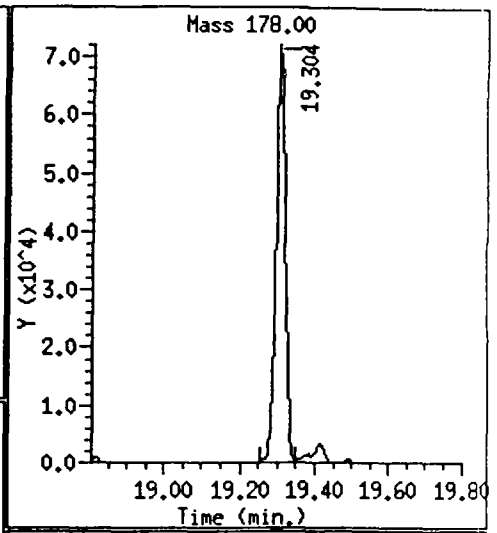
73 Phenanthrene

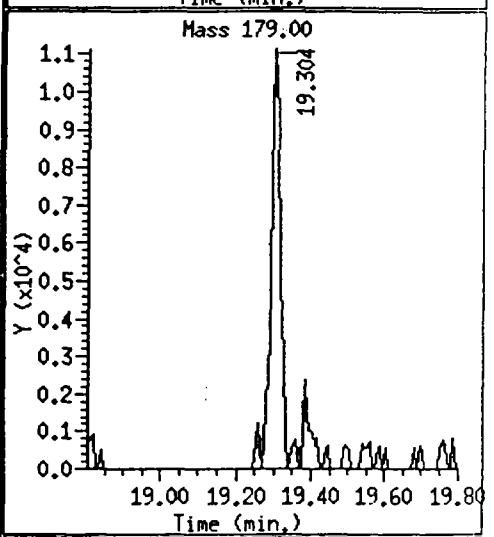


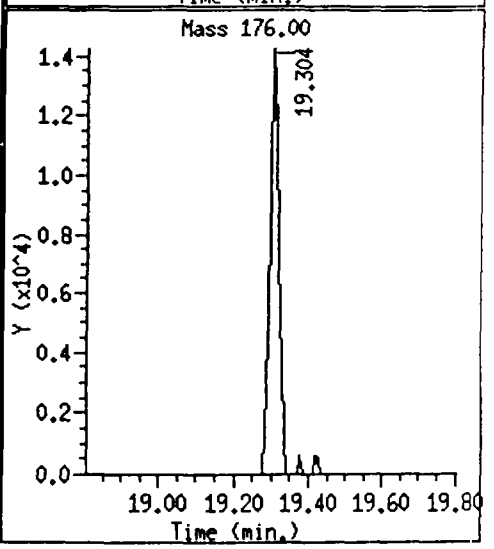












Data File: /chem/V.i/06-08-98.b/VH273.d

Date : 08-JUN-1998 19:04

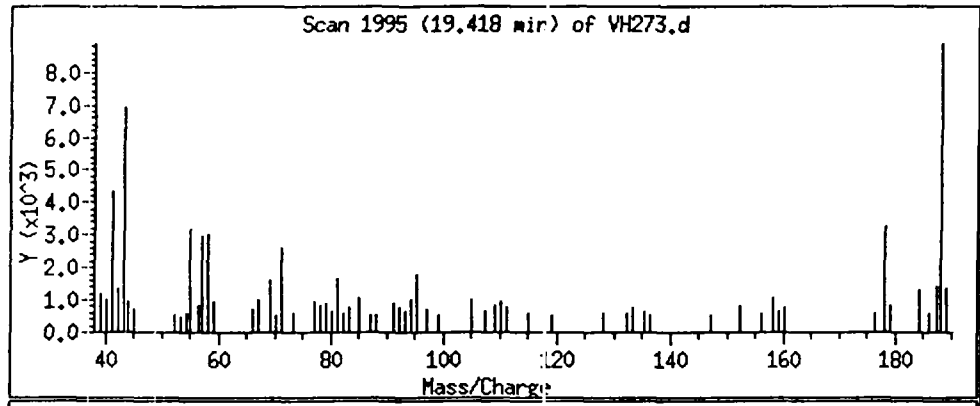
Instrument : V.i

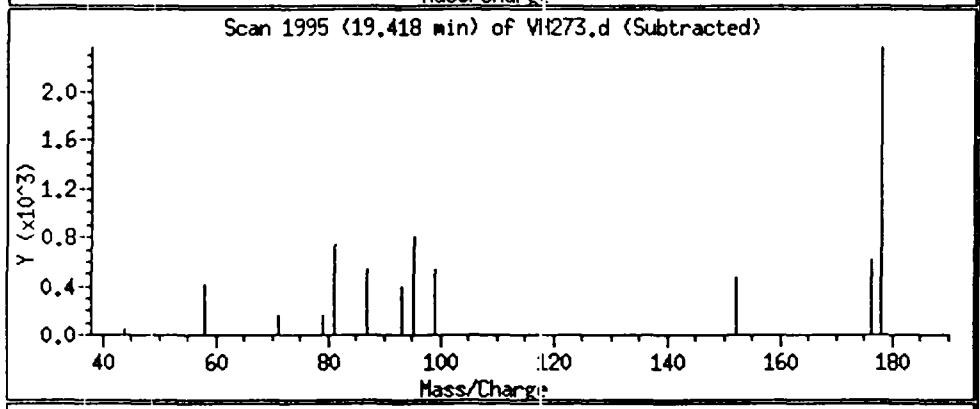
Sample ID : SITE#1 5-28-98 BHR

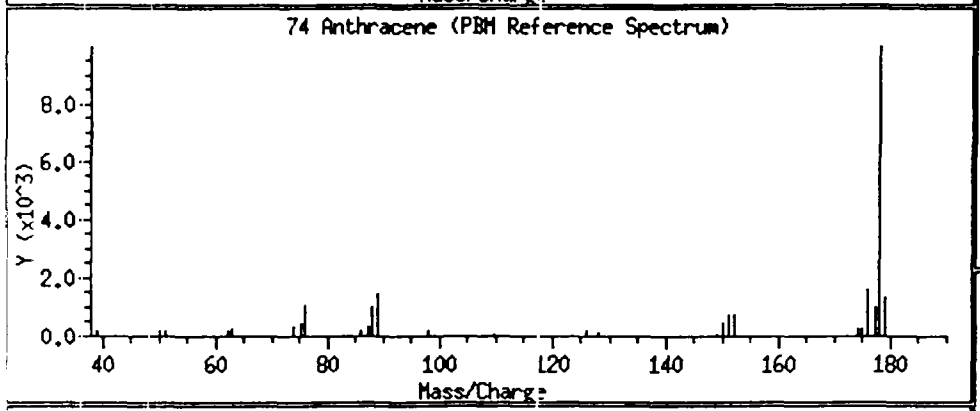
Column phase : DB-5 Column diameter : 0.32

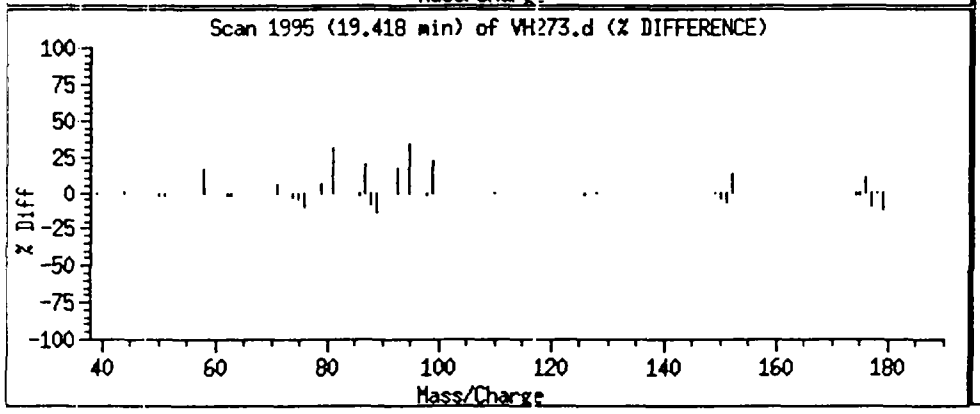
Volume Injected (uL) : 1.0

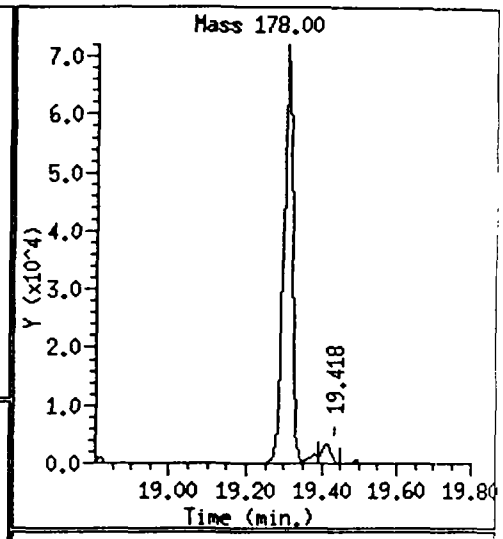
74 Anthracene

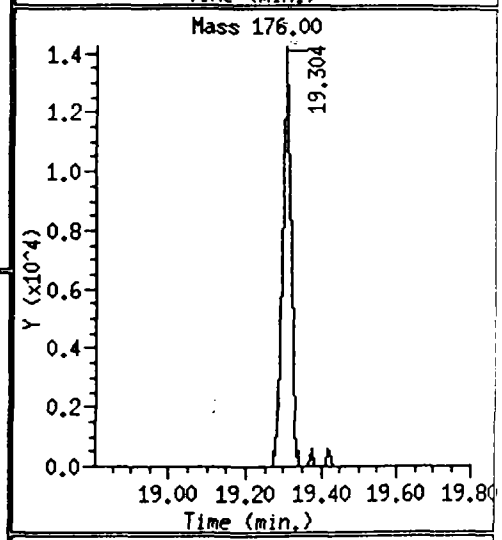


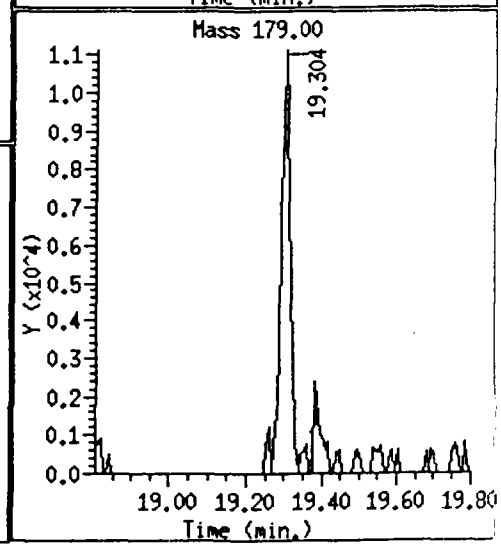












Data File: /chem/V.i/06-08-98.b/VH273.d

Date : 08-JUN-1998 19:04

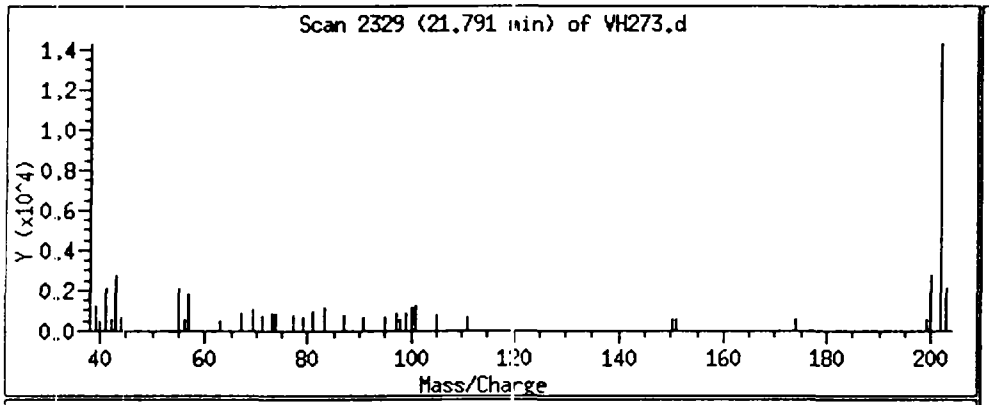
Instrument : V.i

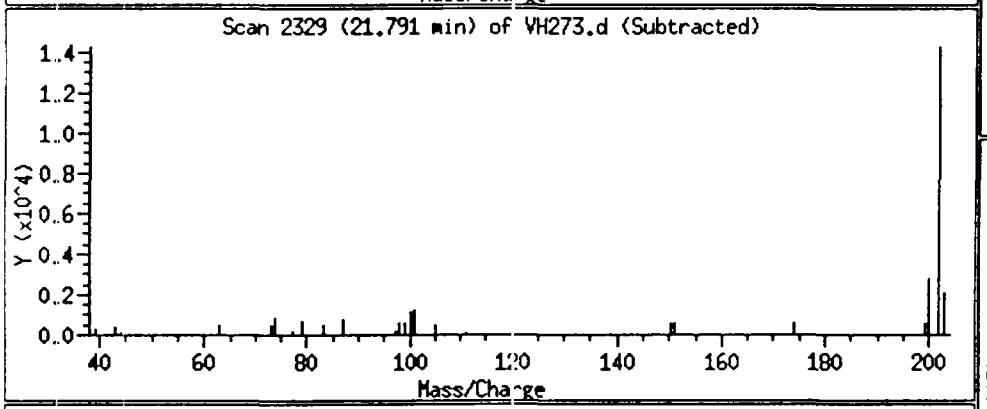
Sample ID : SITE#1 5-28-98 8HR

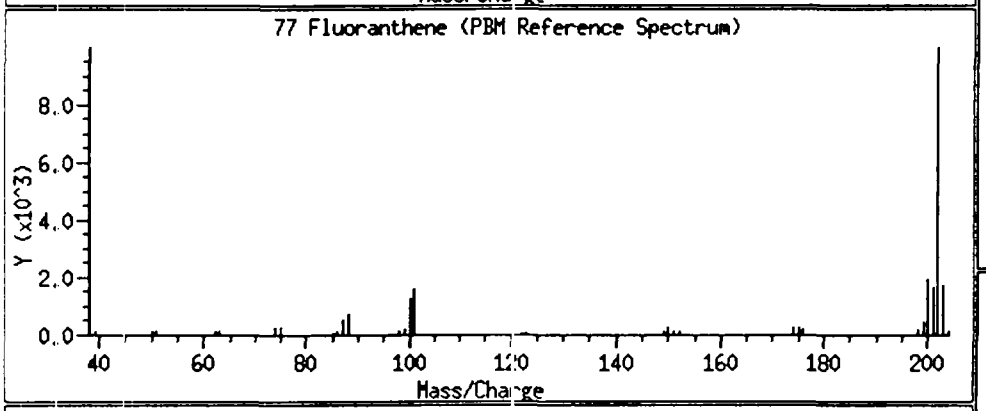
Column phase : DB-5 Column diameter : 0.32

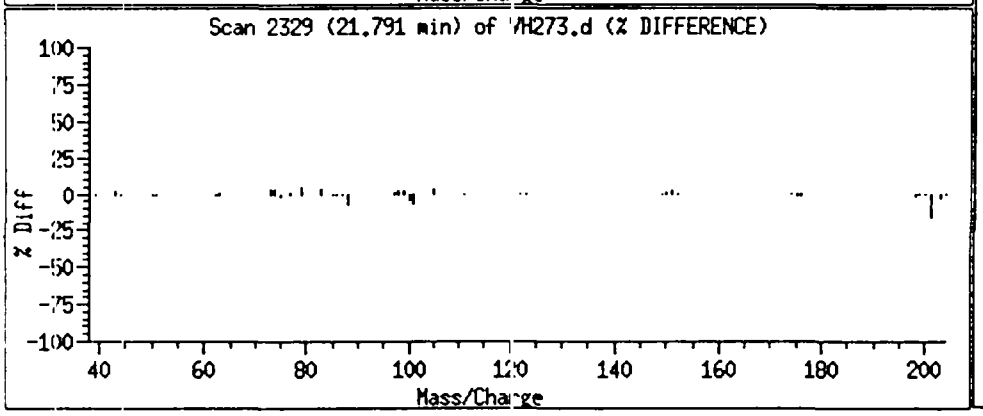
Volume Injected (uL) : 1.0

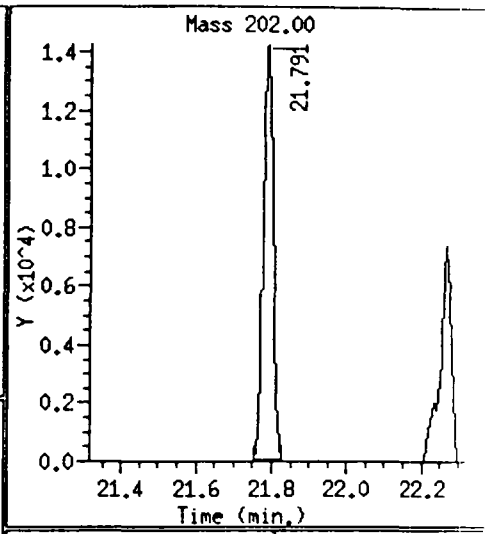
77 Fluoranthene

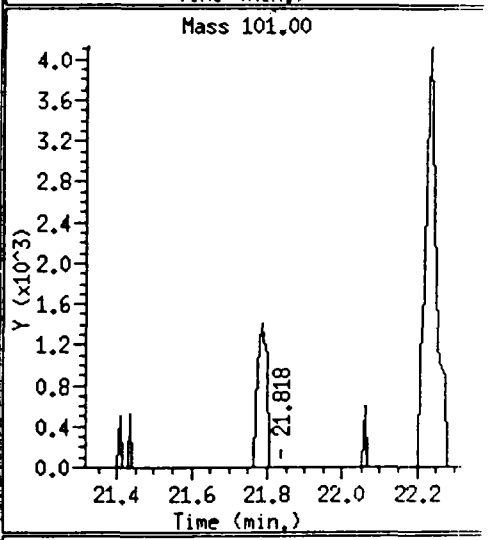


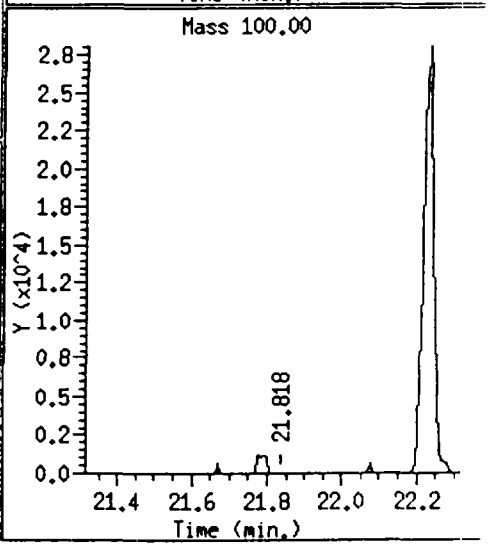












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH286

Method 8270A PUF
Sample ID: SITE#1 5-29-98 24hour

Client Project: Hull & Assoc
TLI ID: 209-67-18,21

Date Received: 06/02/98
Date Extracted: 06/04/98
Date Analyzed : 06/09/98

Response File: VH284

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.53		
Naphthalene	25.90	B	12.57		10
Acenaphthene-d_{10}		IS 3	16.17		
Fluorene	4.85	J	17.31		10
Phenanthrene-d_{10}		IS 4	19.27		
Phenanthrene	18.52	B	19.32		10
Anthracene	0.77	J	19.42		10
Fluoranthene	2.93	J	21.80		10
Chrysene-d_{12}		IS 5	24.88		
Perylene-d_{12}		IS 6	28.93		
Benzo(a)pyrene		U		1.04	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	91.78	19.39	4	92
Pyrene-d_{10}	107.42	22.25	5	107
Terphenyl-d_{14}	100.58	22.61	5	101

Reviewed by RaB Date 10/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

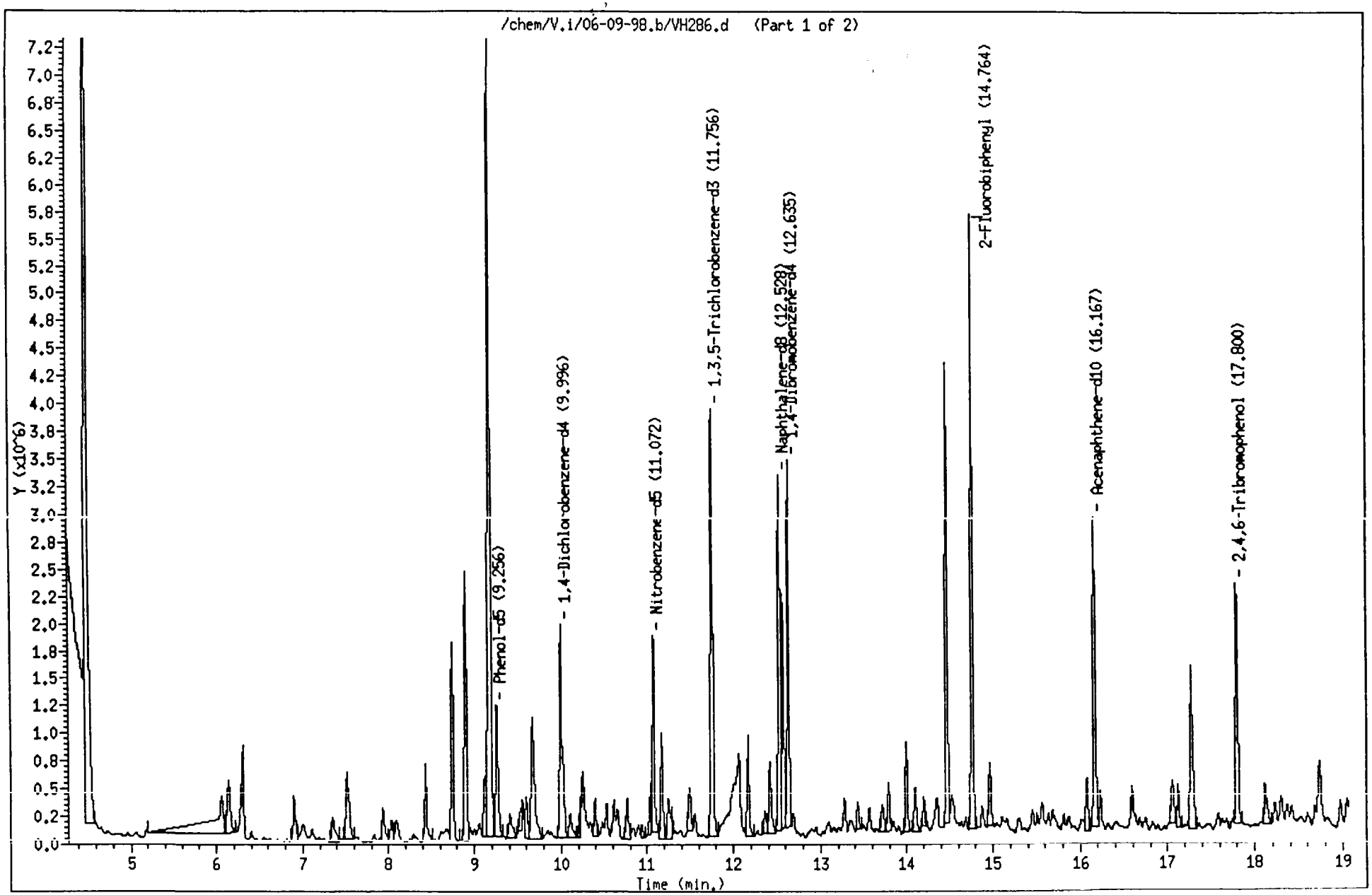
Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

Savar v3.7
Printed: 15:16 06/12/1998

Data File: /chem/V.i/06-09-98.b/VH286.d
Date : 09-JUN-1998 13:07
Instrument : V.i
Sample ID : SITE#1 5/29/98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



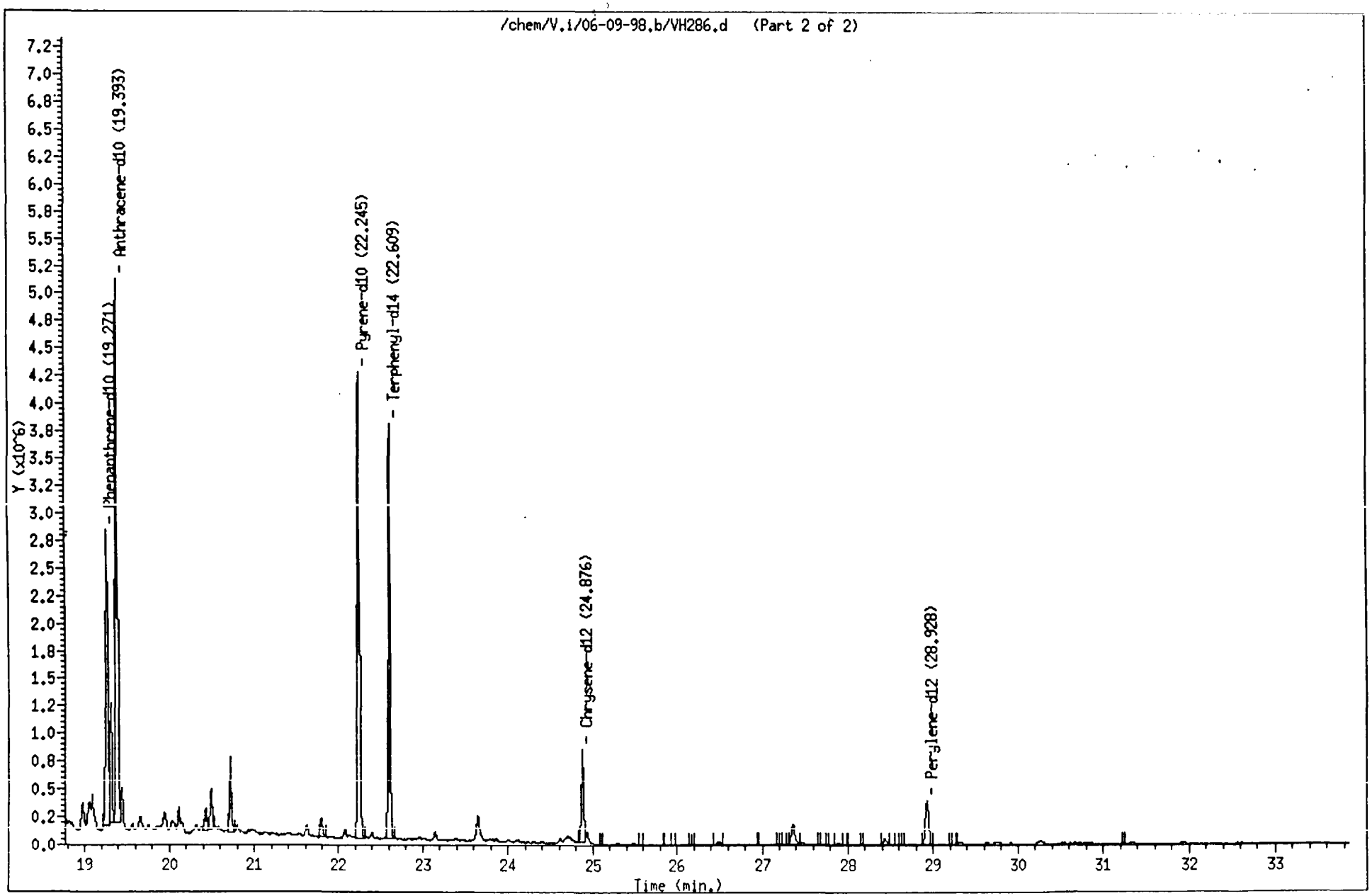
Data Review: [illegible]
Date: 6/12/98

69

Data File: /chem/V.i/06-09-98.b/VH286.d
Date : 09-JUN-1998 13:07
Instrument : V.i
Sample ID : SITE#1 5/29/98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-09-98.b/VH286.d
Lab. Id. : 45869
Inj Date : 09-JUN-1998 13:07 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#1 5/29/98 24HOUR
Misc Info : 209-67-18,21
Comment :
Method : /chem/V.i/06-09-98.b/cclist.m
Meth Date : 09-Jun-1998 12:27 target
Cal Date : 09-JUN-1998 10:31 Cal File: VH284.d
Als bottle: 2
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

		Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1	1,4-Dichlorobenzene-d4	152.00	9.996(1.000)	445076	40.00	
*	2	Naphthalene-d8	136.00	12.528(1.000)	1807145	40.00	
*	3	Acenaphthene-d10	164.00	16.167(1.000)	981998	40.00	
*	4	Phenanthrene-d10	188.00	19.271(1.000)	1642578	40.00	
*	5	Chrysene-d12	240.00	24.876(1.000)	490168	40.00	
*	6	Perylene-d12	264.00	28.928(1.000)	386279	40.00	
$	7	2-Fluorophenol	Compound Not Detected.				
$	8	Phenol-d5	99.00	9.263(0.927)	416374	30.34	30.34
$	9	2-Chlorophenol-d4	Compound Not Detected.				
$	10	1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11	Nitrobenzene-d5	82.00	11.072(0.884)	845624	55.62	55.62
$	12	1,3,5-Trichlorobenzene-d3	185.00	11.756(0.938)	1074494	65.62	65.62
$	13	1,4-Dibromobenzene-d4	240.00	12.635(1.264)	872665	78.08	78.08
$	14	2-Fluorobiphenyl	172.00	14.764(0.913)	2416514	86.48	86.48
$	15	2,4,6-Tribromophenol	329.80	17.808(1.101)	339266	74.49	74.49
$	16	Anthracene-d10	188.00	19.393(1.006)	3097920	91.75	91.75
$	17	Pyrene-d10	212.00	22.245(0.894)	2707808	107.42	107.42
$	18	Terphenyl-d14	244.00	22.609(0.909)	1539408	100.56	100.56
	42	Naphthalene	128.00	12.571(1.003)	1159688	25.90	25.90
	46	2-Methylnaphthalene	142.00	13.998(1.117)	291657	10.61	10.61
	50	2-Chloronaphthalene	Compound Not Detected.				
	56	Acenaphthylene	152.00	~~16.145(0.999)~~	~~9103~~	~~0.22~~	~~0.22(a)~~ NA PAB
	58	Acenaphthene	154.00	16.231(1.004)	95950	4.02	4.02(a)
	65	Fluorene	166.00	17.306(1.070)	161948	4.85	4.85(a)
	73	Phenanthrene	178.00	19.321(1.003)	683654	18.51	18.51
	74	Anthracene	178.00	19.42 ~~19.321(1.003)~~	~~683654~~ 26783	~~19.73~~	~~19.73~~ M PAB
	77	Fluoranthene	202.00	21.796(1.131)	106404	2.93	2.93(a)
	78	Pyrene	Compound Not Detected.				
	83	Benzo(a)anthracene	Compound Not Detected.				
	84	Chrysene	Compound Not Detected.				
	86	Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-09-98.b/VH286.d

Date : 09-JUN-1998 13:07

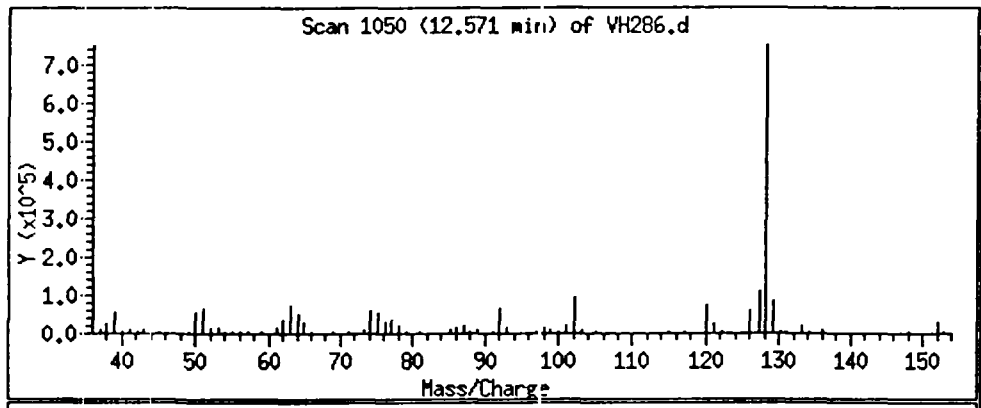
Instrument : V.i

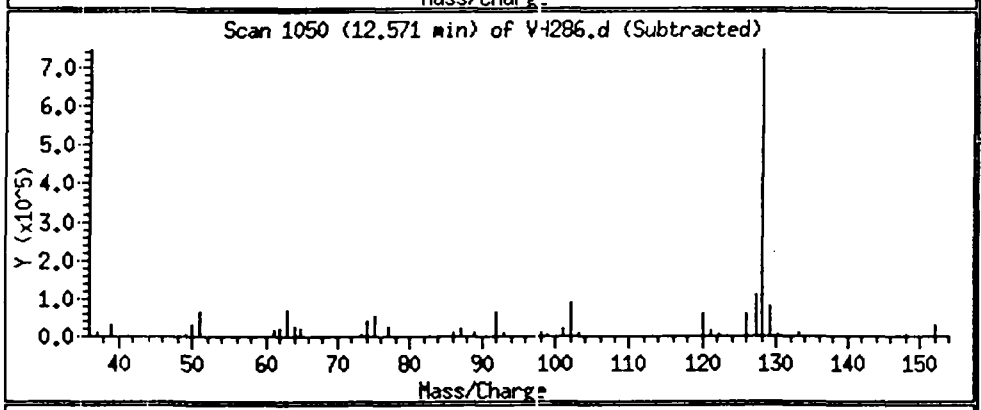
Sample ID : SITE#1 5/29/98 24HR

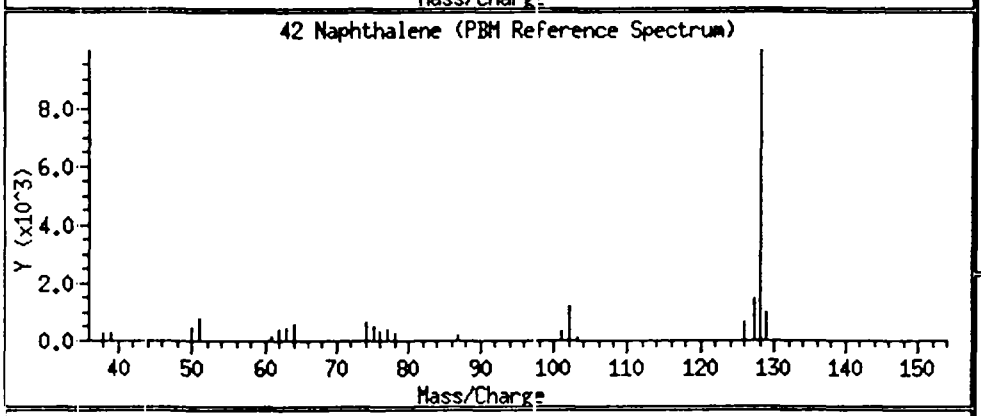
Column phase : DB-5 Column diameter : 0.32

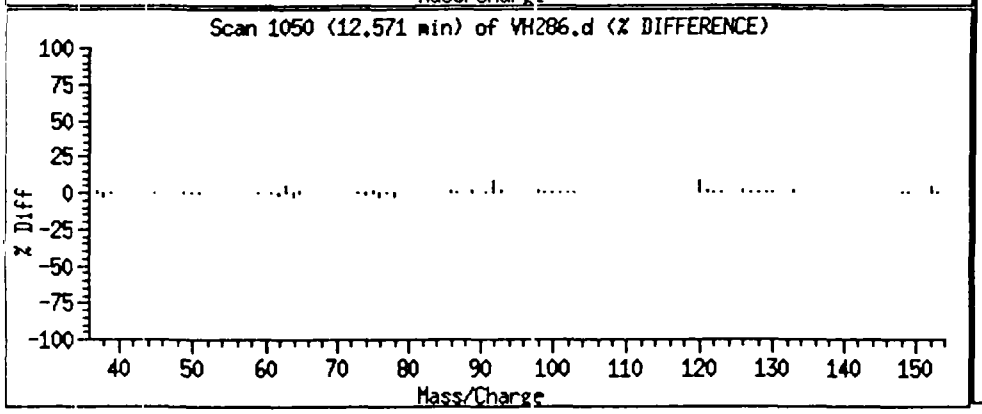
Volume Injected (uL) : 1.0

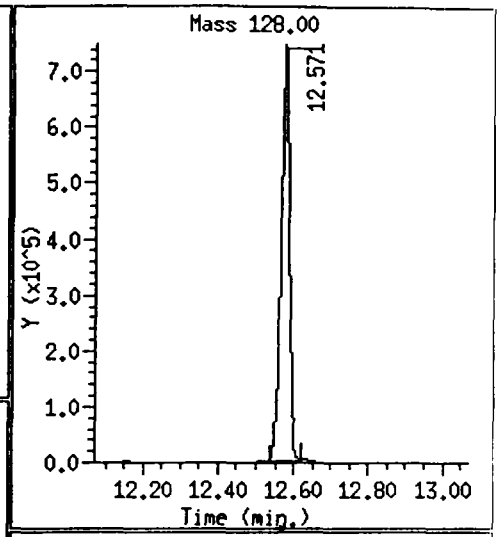
42 Naphthalene

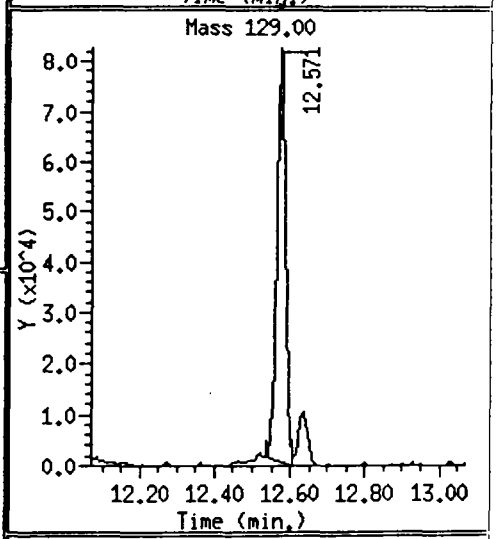


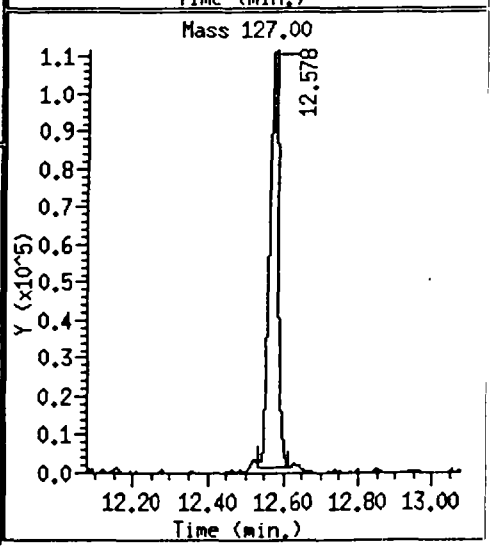












Data File: /chem/V.i/06-09-98.b/VH286.d

Date : 09-JUN-1998 13:07

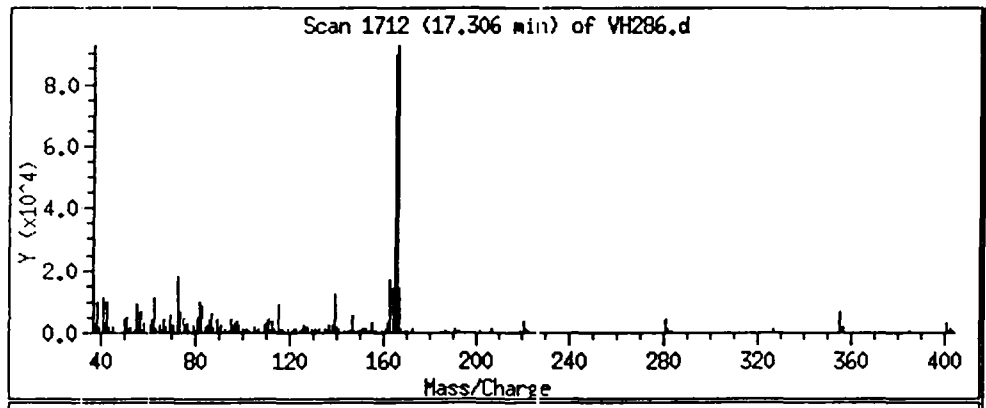
Instrument : V.i

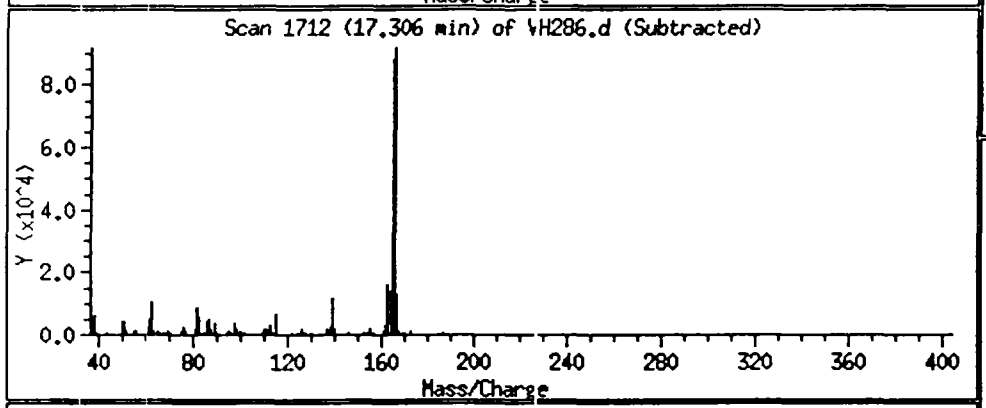
Sample ID : SITE#1 5/29/98 24HR

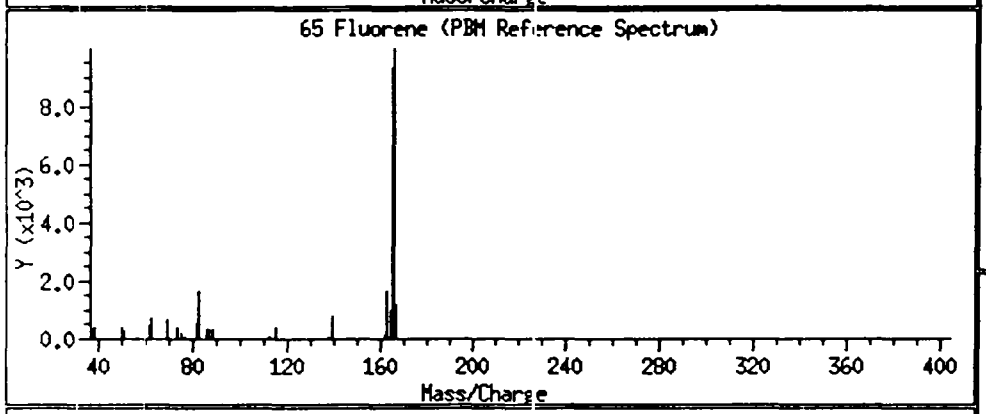
Column phase : DB-5 Column diameter : 0.32

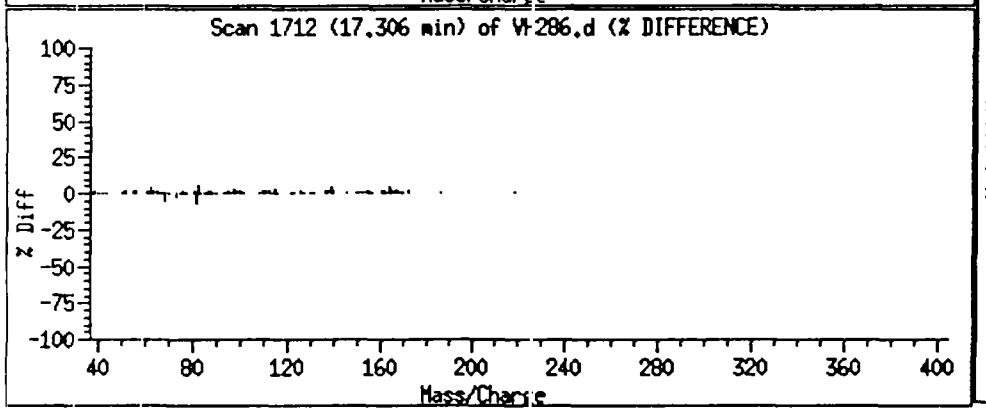
Volume Injected (uL) : 1.0

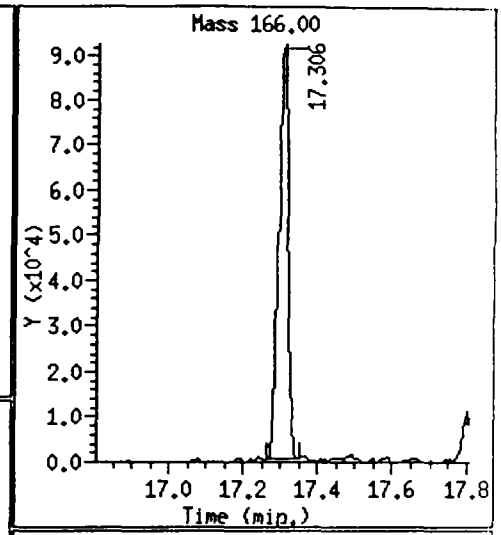
65 Fluorene

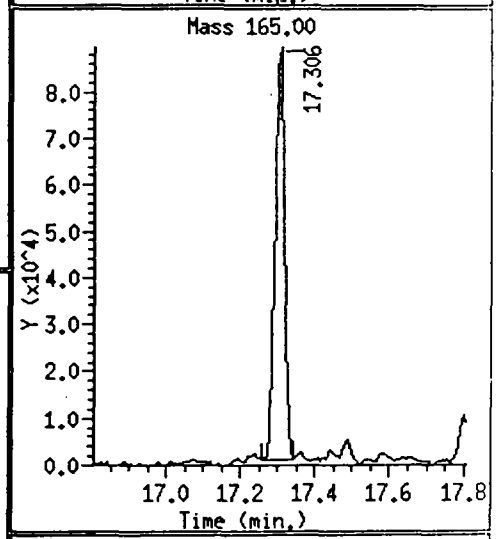


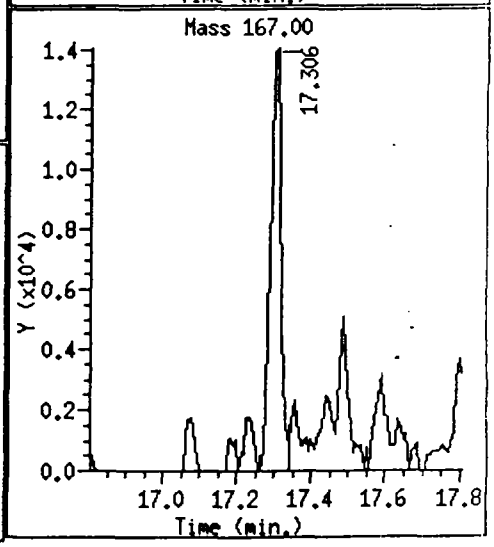












Data File: /chem/V.i/06-09-98.b/VH286.d

Date : 09-JUN-1998 13:07

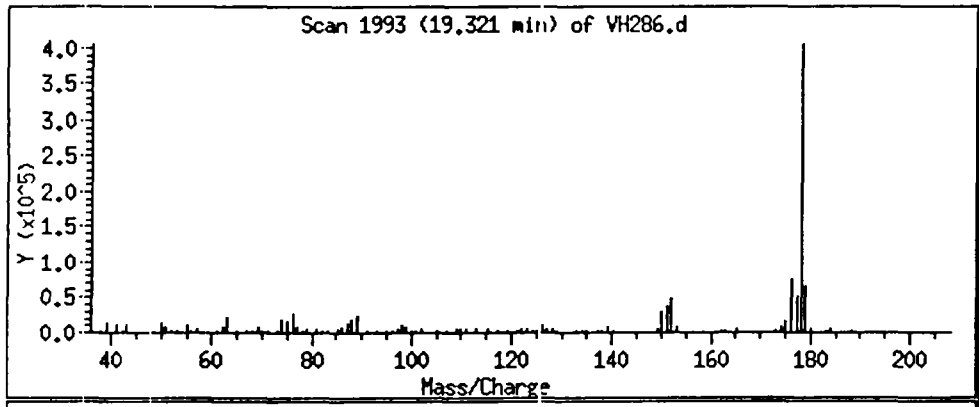
Instrument : V.i

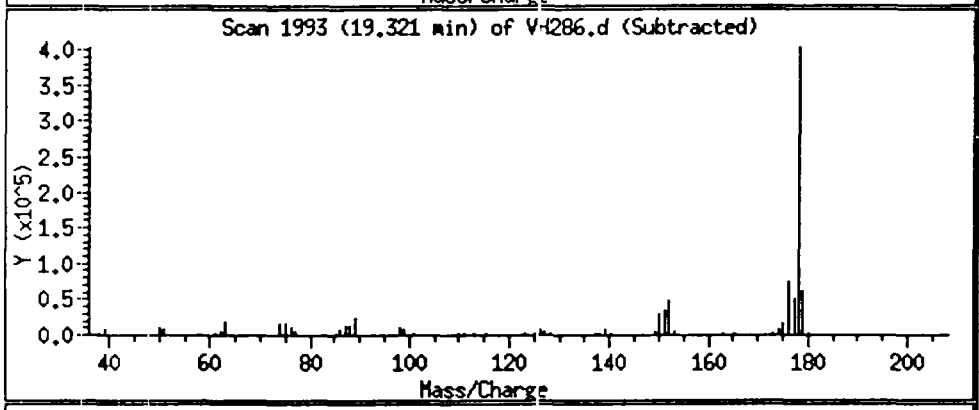
Sample ID : SITE#1 5/29/98 24HR

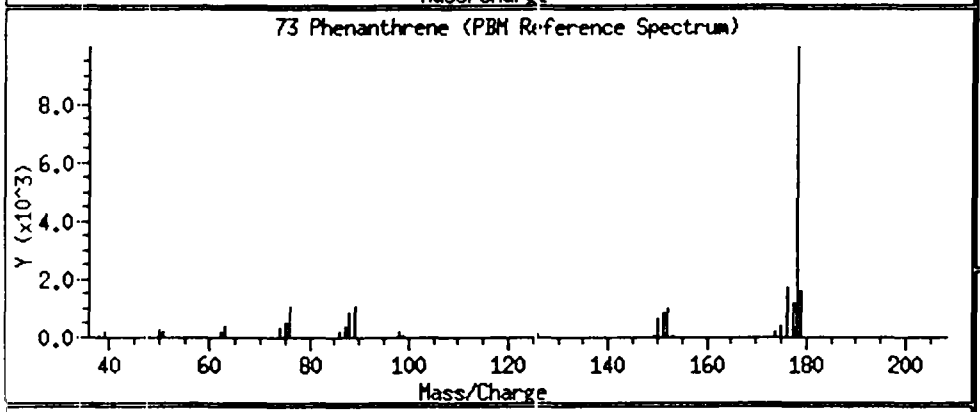
Column phase : DB-5 Column diameter : 0.32

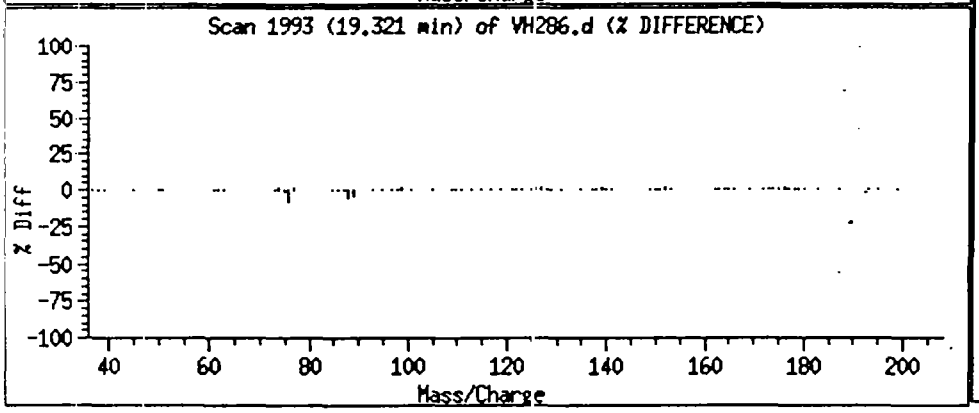
Volume Injected (uL) : 1.0

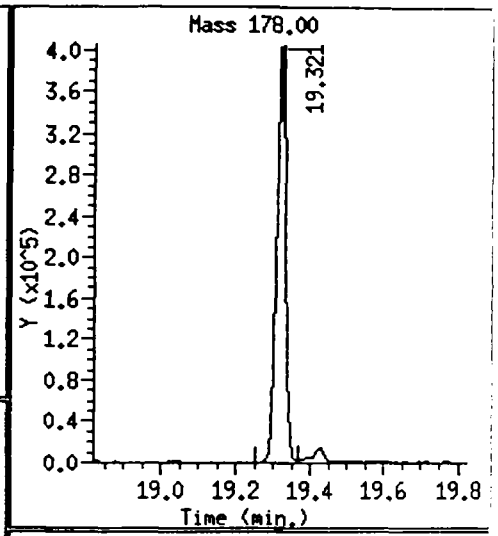
73 Phenanthrene

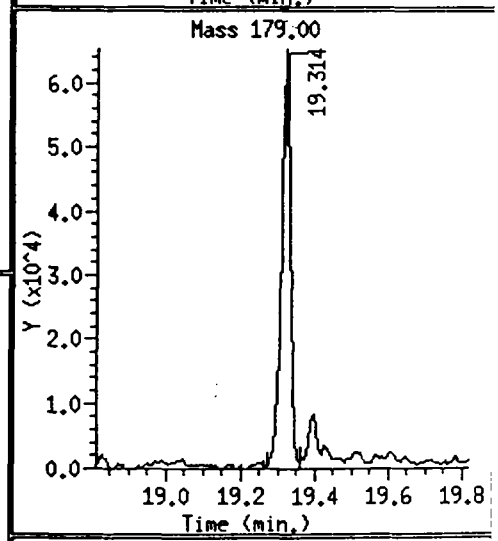


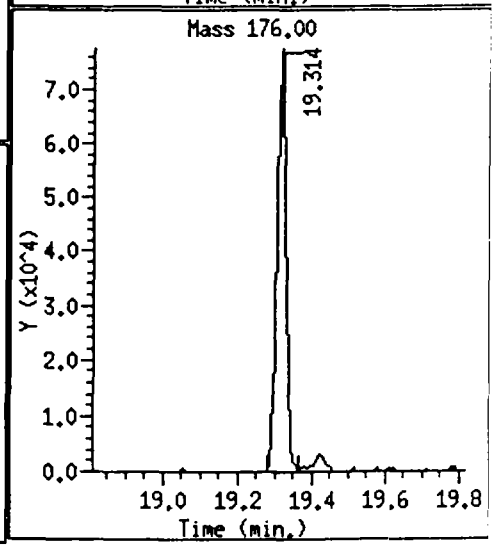












Data File: /chem/V.i/06-09-98.b/VH286.d

Date : 09-JUN-1998 13:07

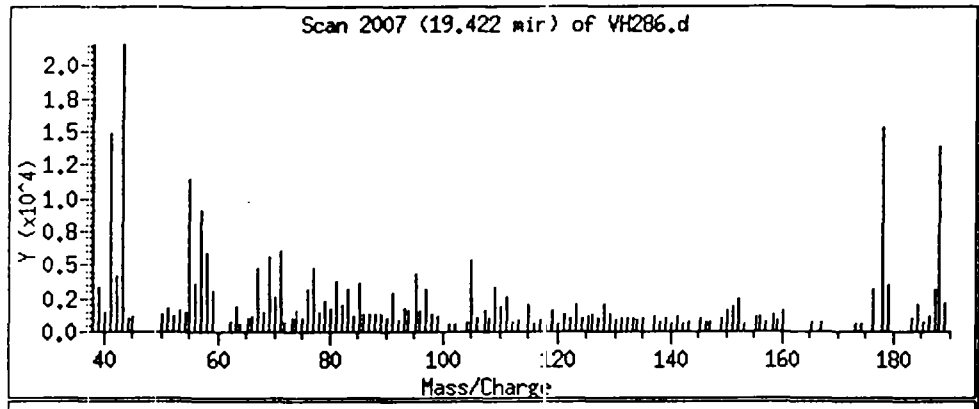
Instrument : V.i

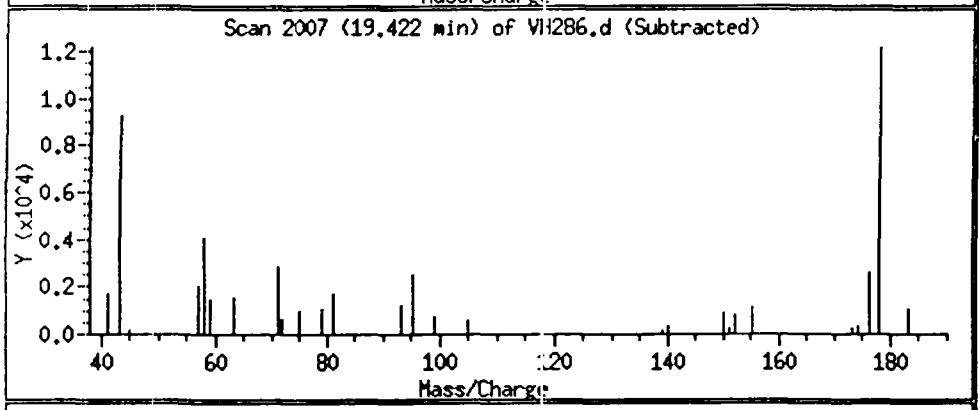
Sample ID : SITE#1 5/29/98 24HR

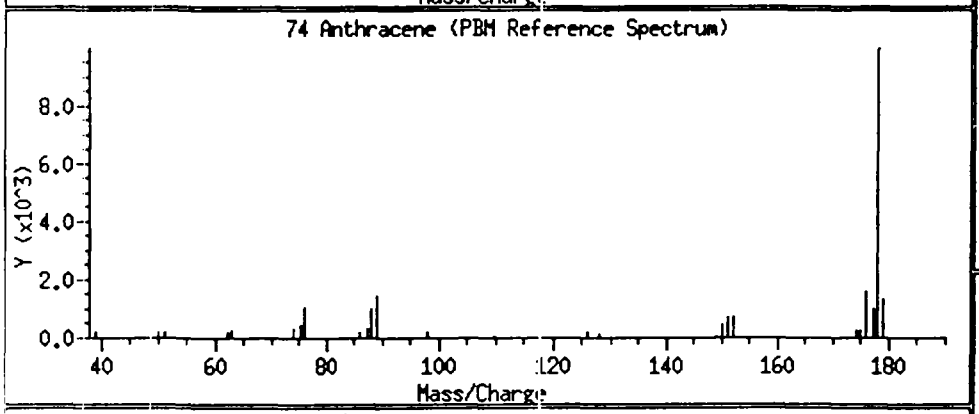
Column phase : DB-5 Column diameter : 0.32

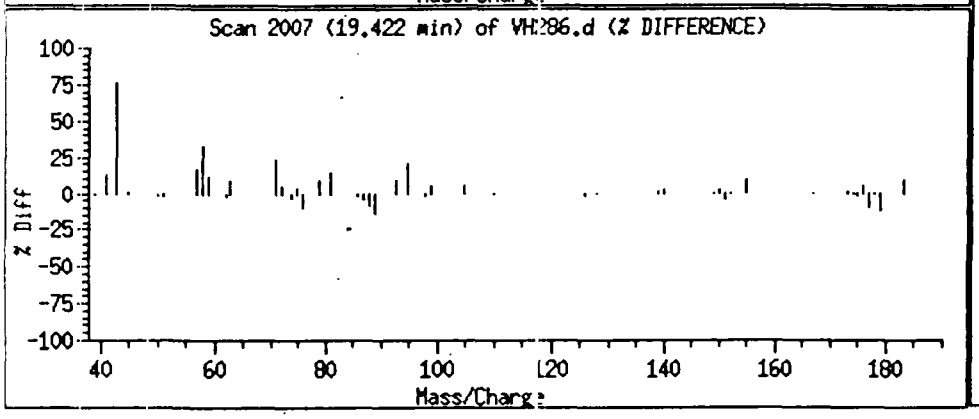
Volume Injected (uL) : 1.0

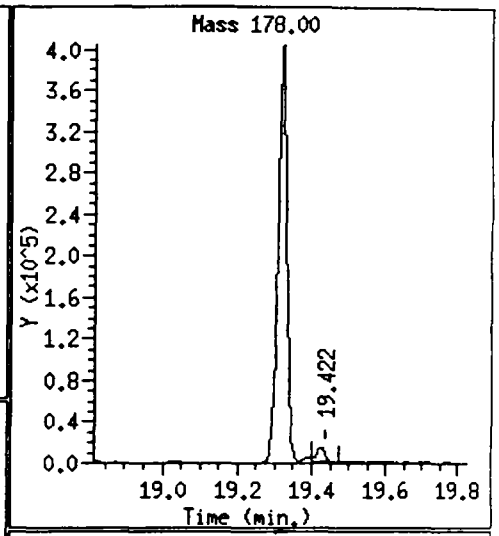
74 Anthracene

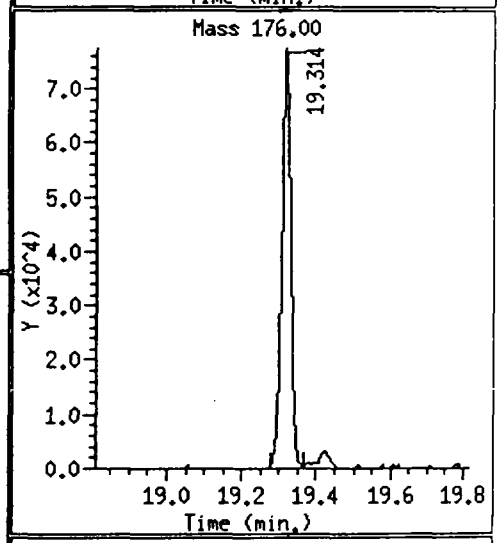


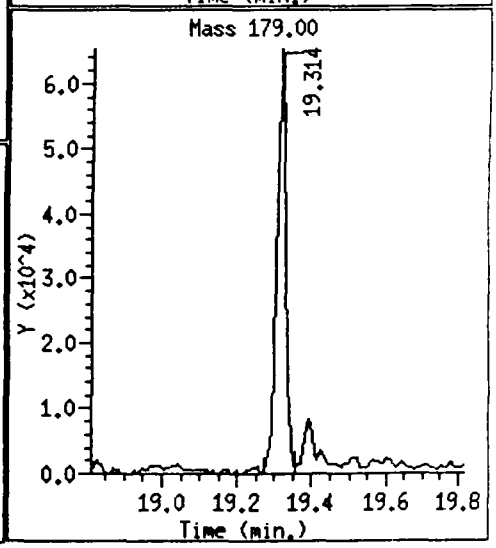












Data File: /chem/V.i/06-09-98.b/VH286.d

Date : 09-JUN-1998 13:07

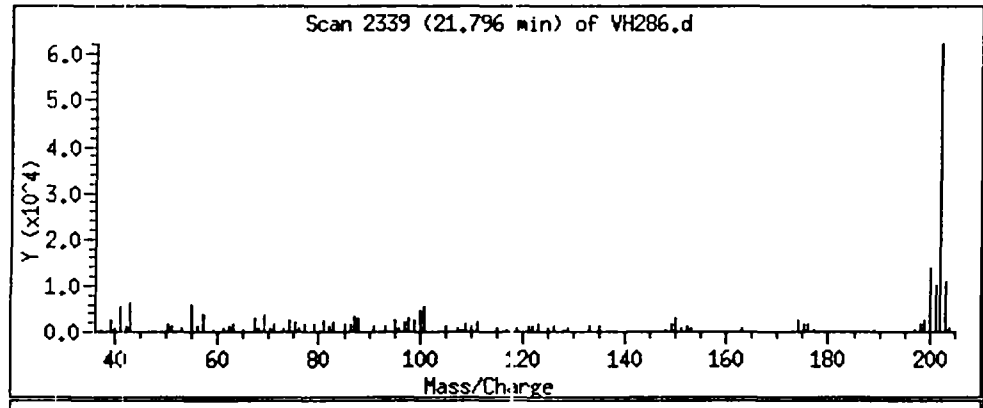
Instrument : V.i

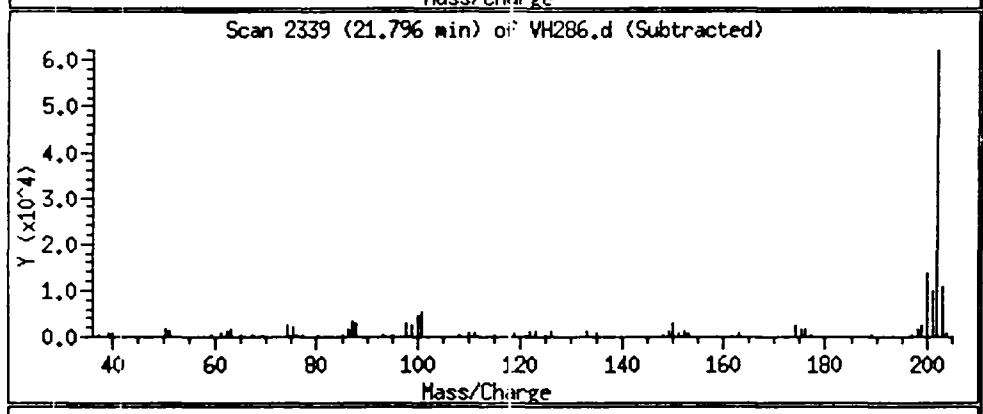
Sample ID : SITE#1 5/29/98 24HR

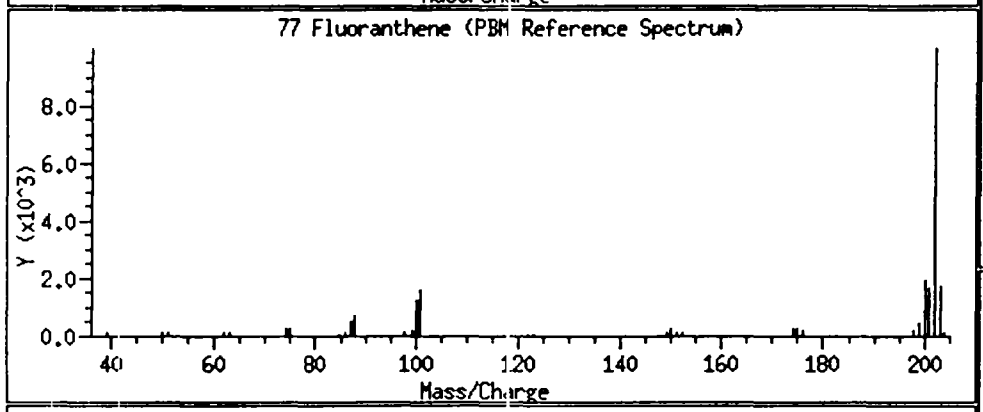
Column phase : DB-5 Column diameter : 0.32

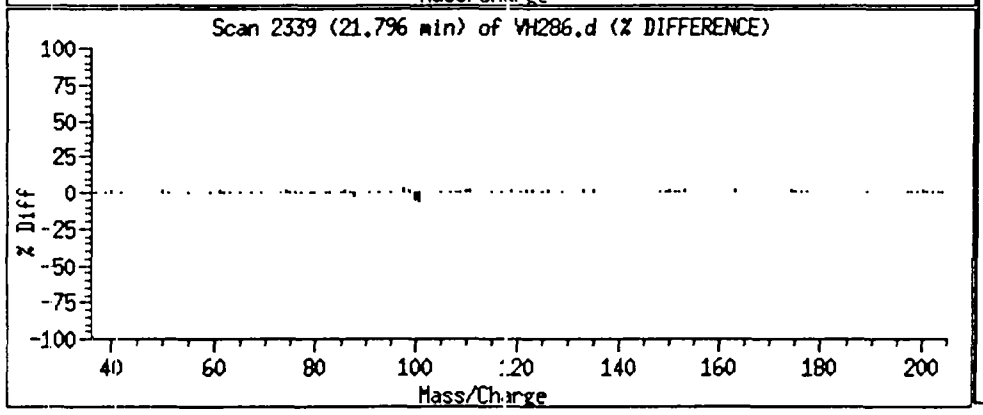
Volume Injected (uL) : 1.0

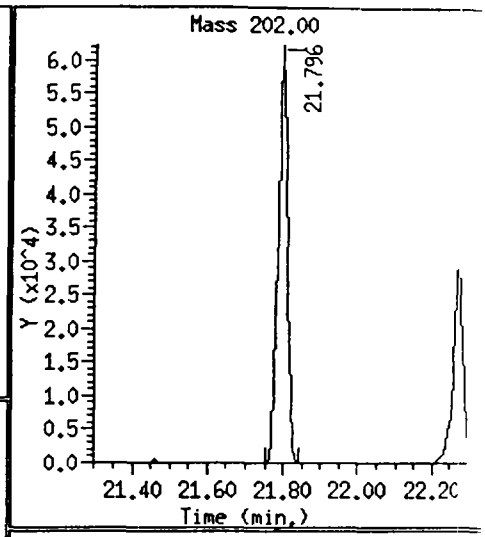
77 Fluoranthene

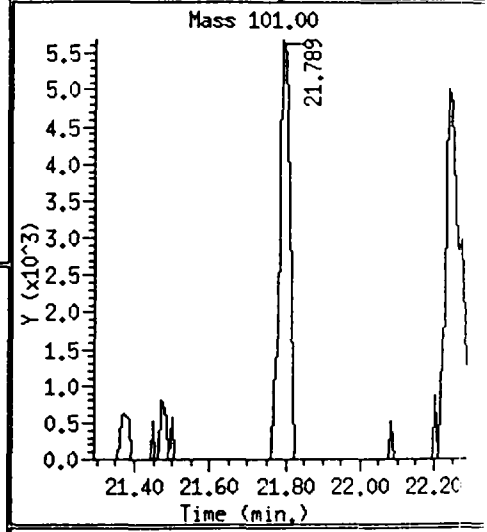


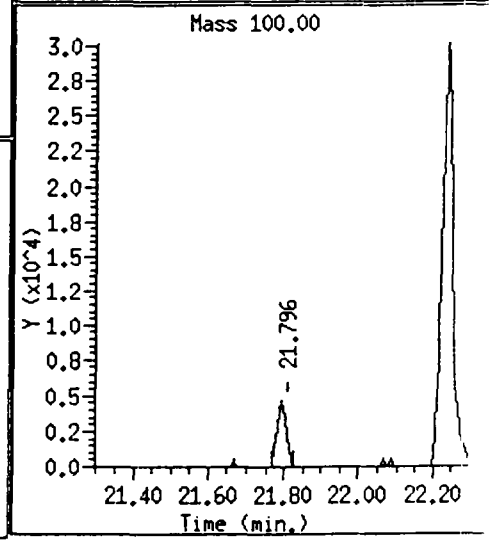












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH278

Method 8270A PUF
Sample ID: SITE#2 5-27-98 24hour

Client Project: Hull & Assoc	Date Received: 06/02/98	Response File: VH267
TLI ID: 209-67-10,15	Date Extracted: 06/04/98	
	Date Analyzed : 06/08/98	
	Dilution Factor: 1.00	

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	19.27	B	12.56		10
Acenaphthene-d_{10}		IS 3	16.16		
Fluorene	8.36	J	17.30		10
Phenanthrene-d_{10}		IS 4	19.27		
Phenanthrene	22.13	B	19.31		10
Anthracene	0.67	J	19.42		10
Fluoranthene	2.83	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.06	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	73.51	19.38	4	74
Pyrene-d_{10}	86.75	22.23	5	87
Terphenyl-d_{14}	75.58	22.60	5	76.

Reviewed by PAB Date 10/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

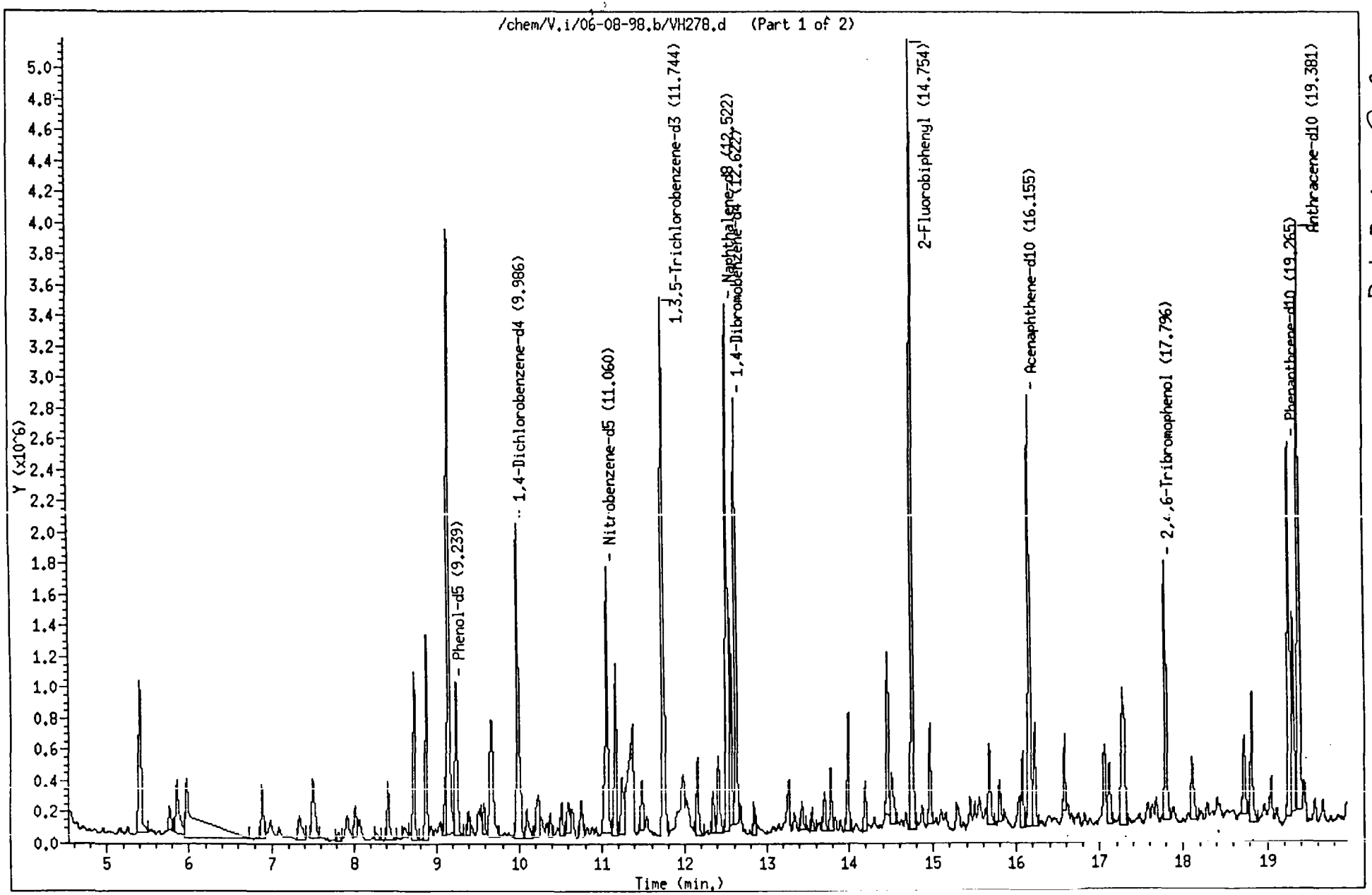
Savar v3.7
Printed: 15:15 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH278.d
Date : 08-JUN-1998 23:10
Instrument : V.i
Sample ID : SITE#2 5-27-98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

78



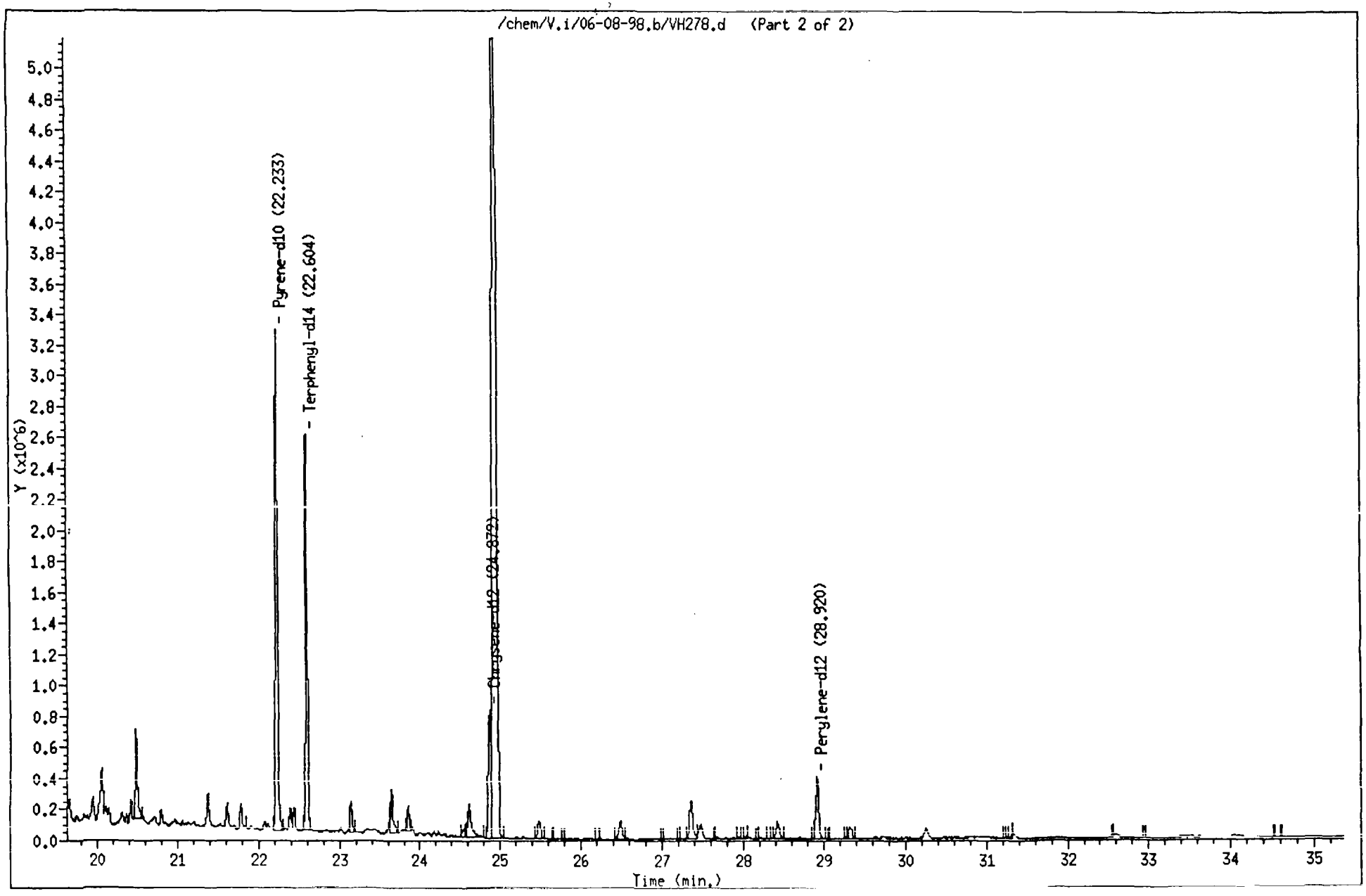
Data Review: [illegible]
Date: 6/12/98

79

Data File: /chem/V.i/06-08-98.b/VH278.d
Date : 08-JUN-1998 23:10
Instrument : V.i
Sample ID : SITE#2 5-27-98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH278.d
Lab. Id. : 45869
Inj Date : 08-JUN-1998 23:10 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#2 5-27-98 24HOUR
Misc Info : 209-67-10,15
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 11
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.986(1.000)	489316	40.00	
*	2 Naphthalene-d8	136.00	12.522(1.000)	1832978	40.00	
*	3 Acenaphthene-d10	164.00	16.155(1.000)	958468	40.00	
*	4 Phenanthrene-d10	188.00	19.265(1.000)	1572828	40.00	
*	5 Chrysene-d12	240.00	24.872(1.000)	503861	40.00	
*	6 Perylene-d12	264.00	28.920(1.000)	383209	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.246(0.926)	398182	26.95	26.95
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.060(0.883)	794731	51.04	51.04
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.744(0.938)	939204	59.35	59.35
$	13 1,4-Dibromobenzene-d4	240.00	12.629(1.265)	742169	67.36	67.36
$	14 2-Fluorobiphenyl	172.00	14.754(0.913)	1967707	71.51	71.51
$	15 2,4,6-Tribromophenol	329.80	17.796(1.102)	248296	45.11	45.11
$	16 Anthracene-d10	188.00	19.381(1.006)	2375810	73.51	73.51
$	17 Pyrene-d10	212.00	22.233(0.894)	1997648	86.74	86.74
$	18 Terphenyl-d14	244.00	22.604(0.909)	1092062	75.60	75.60
	42 Naphthalene	128.00	12.565(1.003)	849677	19.28	19.28
	46 2-Methylnaphthalene	142.00	13.990(1.117)	308758	11.34	11.34
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	154.00	16.220(1.004)	186252	8.11	8.11(a)
	65 Fluorene	166.00	17.301(1.071)	255308	8.36	8.36(a)
	73 Phenanthrene	178.00	19.309(1.002)	798021	22.14	22.14
	74 Anthracene	178.00	A.42 ~~19.309(1.002)~~	~~798021~~ 22673	~~23.73~~	~~23.73~~ (a) PAB
	77 Fluoranthene	202.00	21.791(1.131)	104167	2.83	2.83(a)
	78 Pyrene	Compound Not Detected.				
	83 Benzo(a)anthracene	~~228.00~~	~~24.937(1.003)~~	~~3019~~	~~0.19~~	~~0.19(a)~~ 7P PAB
	84 Chrysene	~~228.00~~	~~24.937(1.003)~~	~~3019~~	~~0.22~~	~~0.22(a)~~ 7P PAB
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIM MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-08-98.b/VH278.d

Date : 08-JUN-1998 23:10

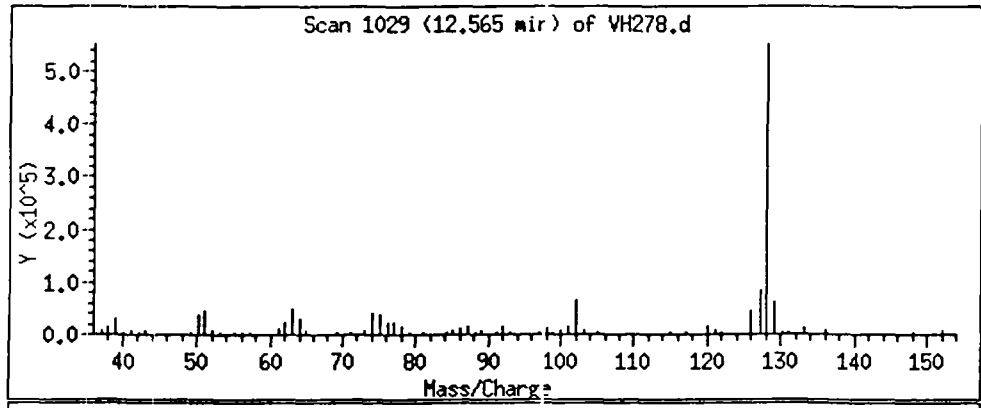
Instrument : V.i

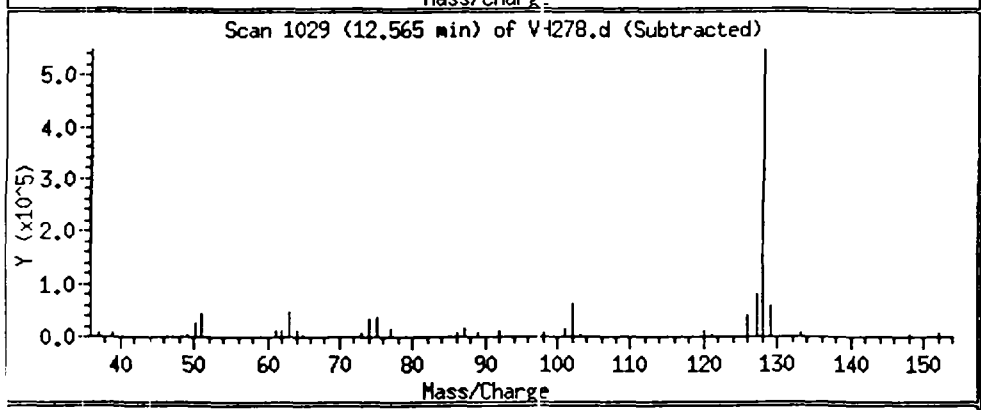
Sample ID : SITE#2 5-27-98 24HR

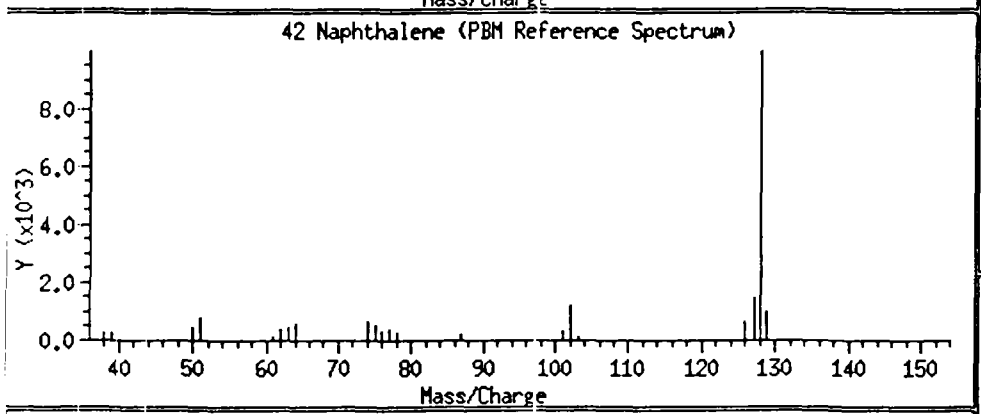
Column phase : DB-5 Column diameter : 0.32

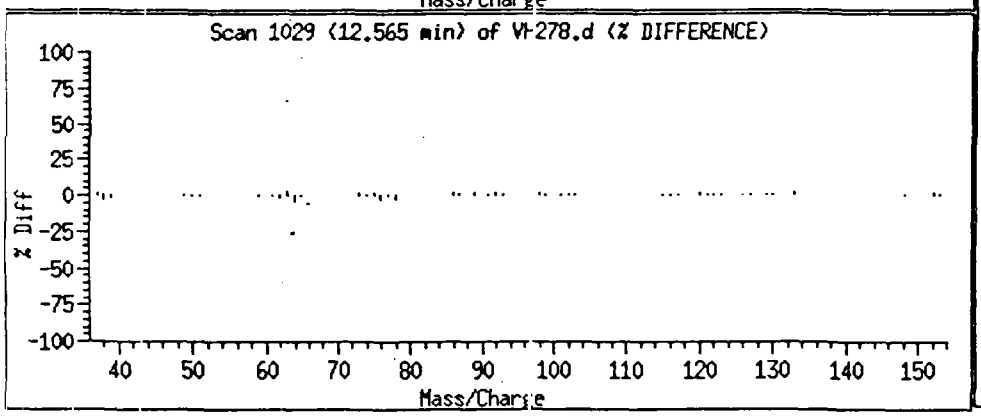
Volume Injected (uL) : 1.0

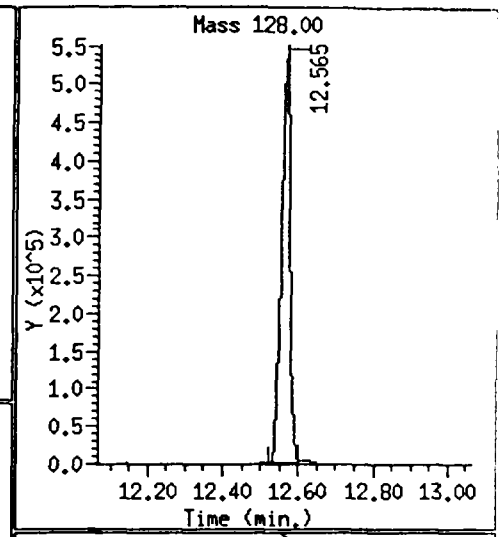
42 Naphthalene

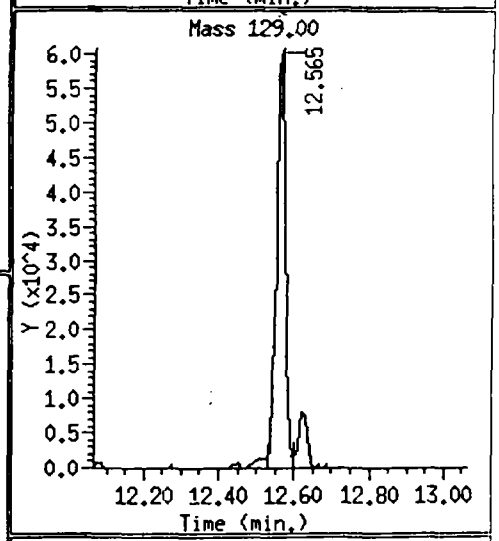


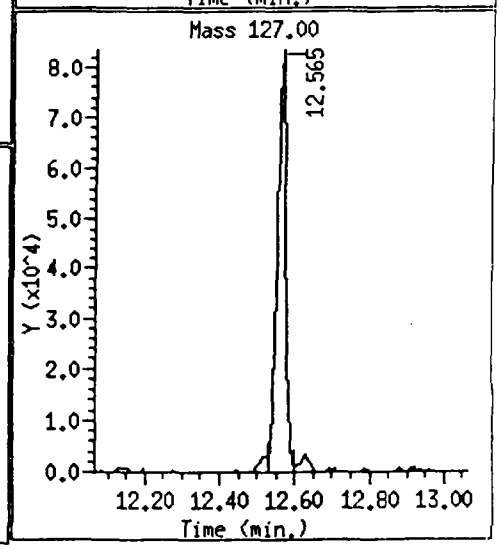












Data File: /chem/V.i/06-08-98.b/VH278.d

Date : 08-JUN-1998 23:10

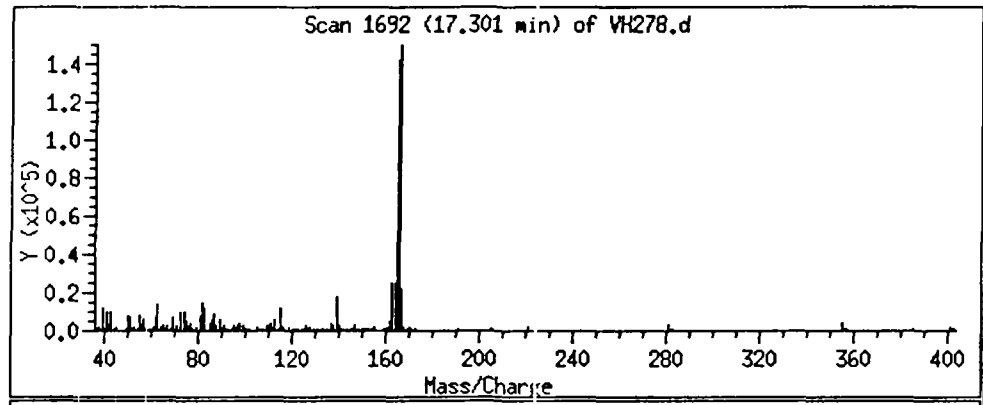
Instrument : V.i

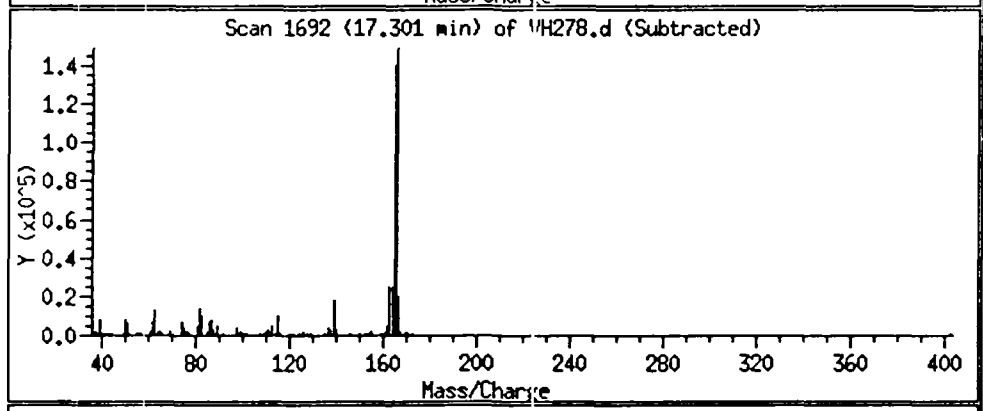
Sample ID : SITE#2 5-27-98 24HR

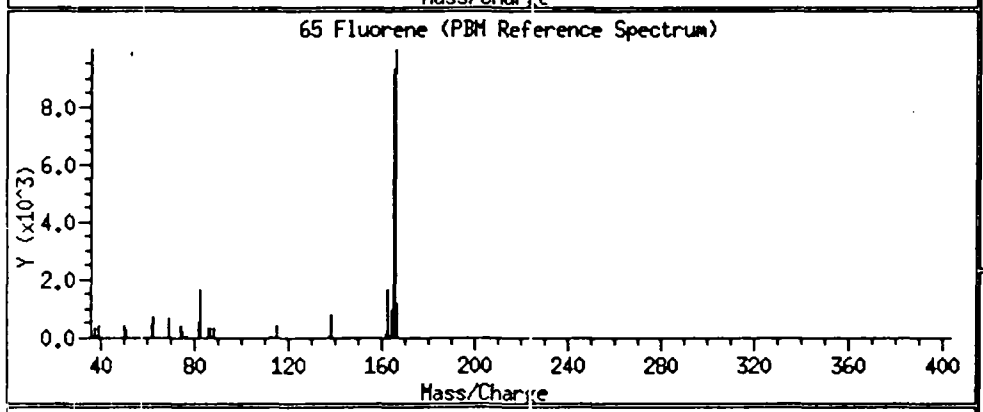
Column phase : DB-5 Column diameter : 0.32

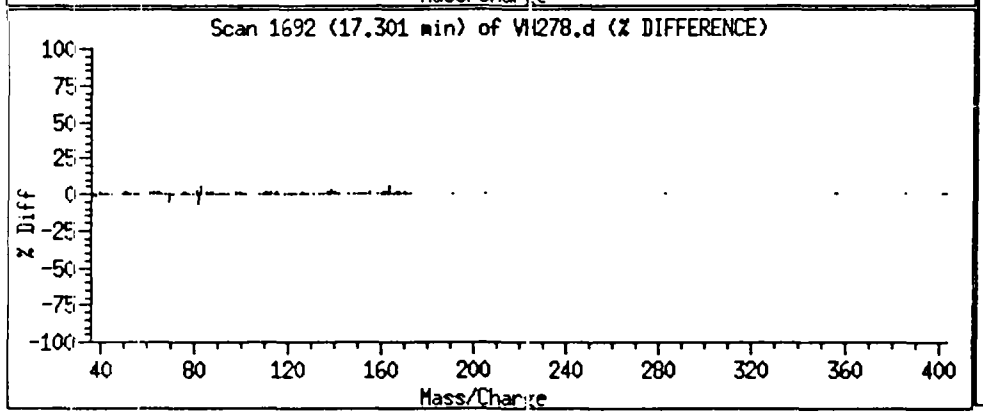
Volume Injected (uL) : 1.0

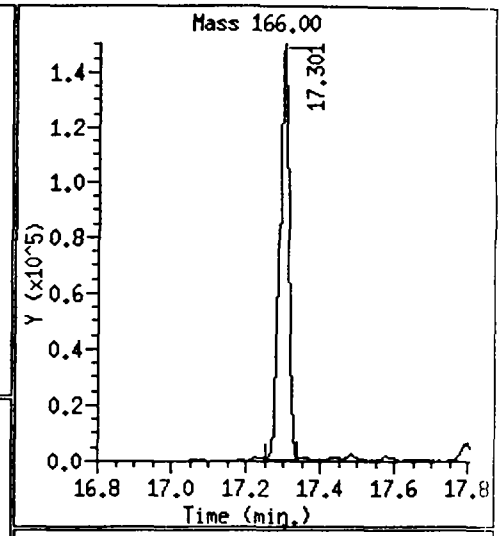
65 Fluorene

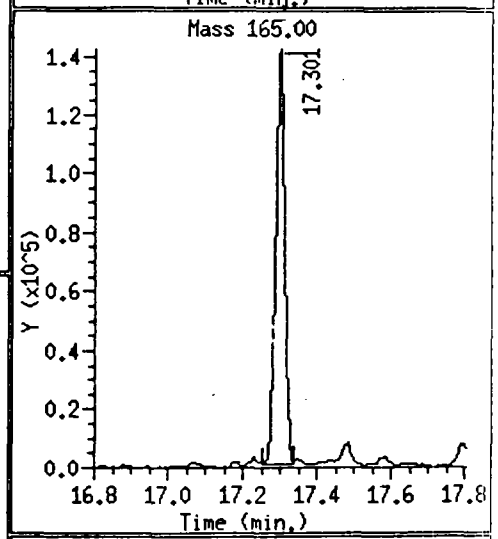


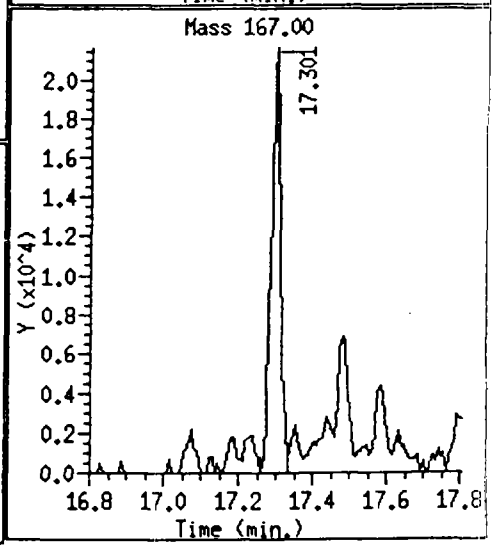












Data File: /chem/V.i/06-08-98.b/VH278.d

Date : 08-JUN-1998 23:10

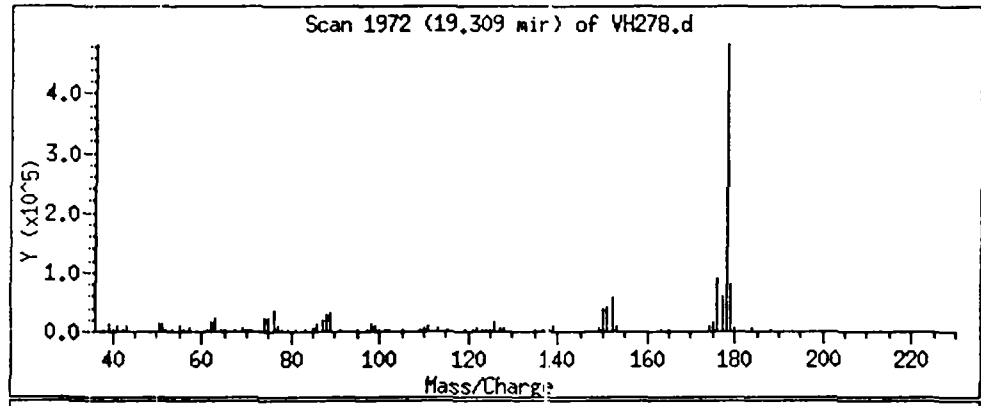
Instrument : V.i

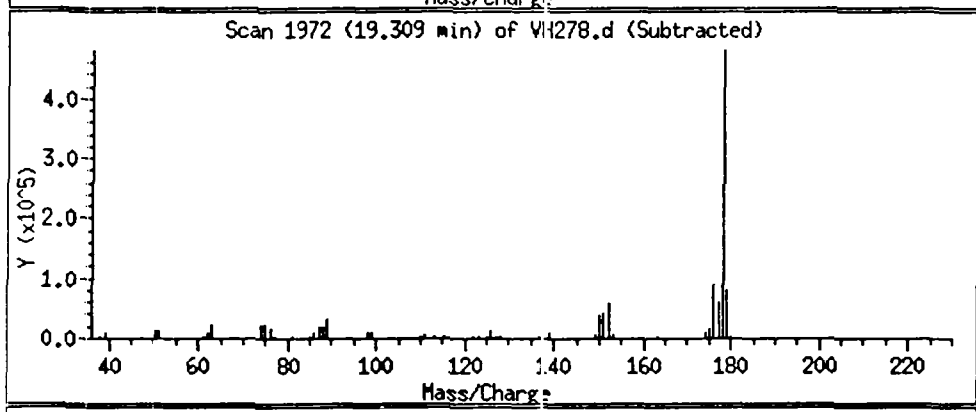
Sample ID : SITE#2 5-27-98 24HR

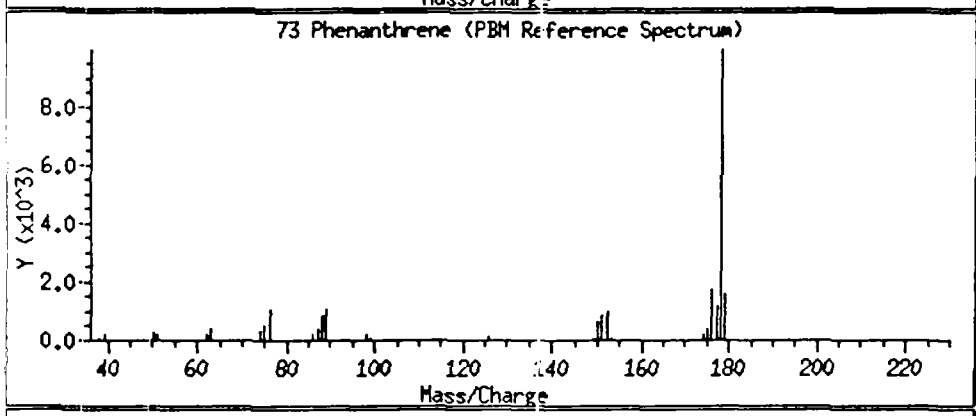
Column phase : DB-5 Column diameter : 0.32

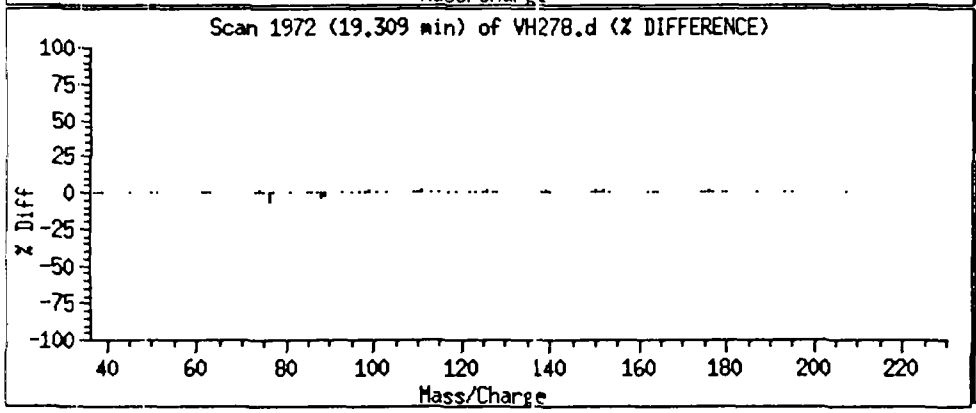
Volume Injected (uL) : 1.0

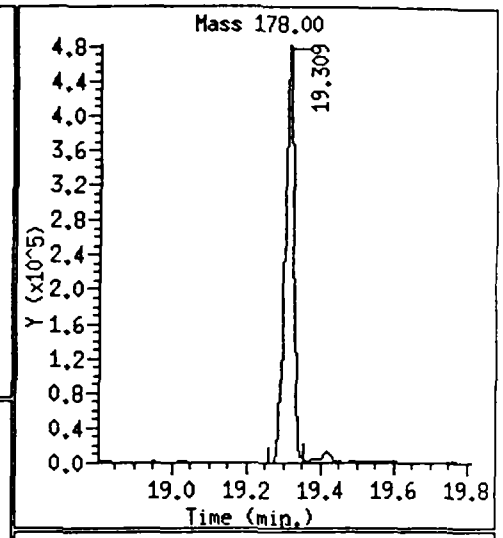
73 Phenanthrene

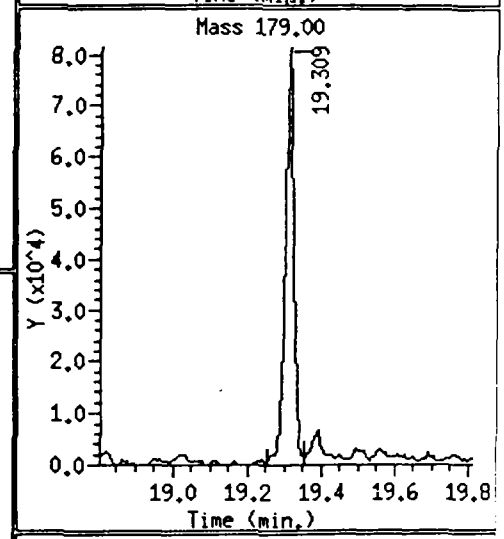


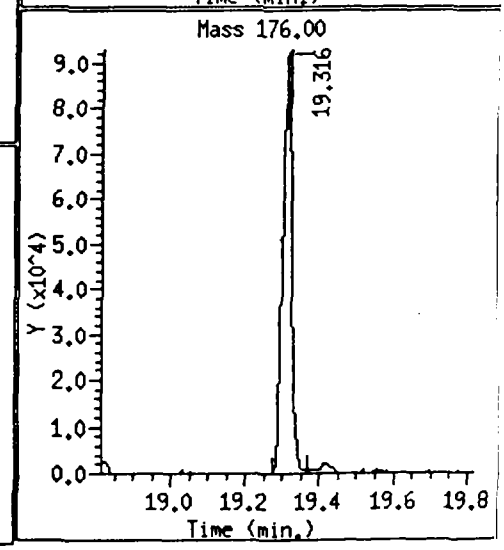












Data File: /chem/V.i/06-08-98.b/VH278.d

Date : 08-JUN-1998 23:10

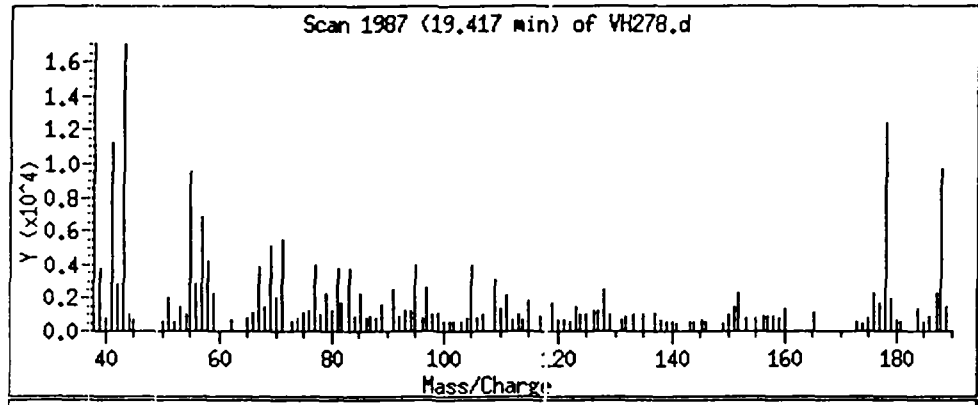
Instrument : V.i

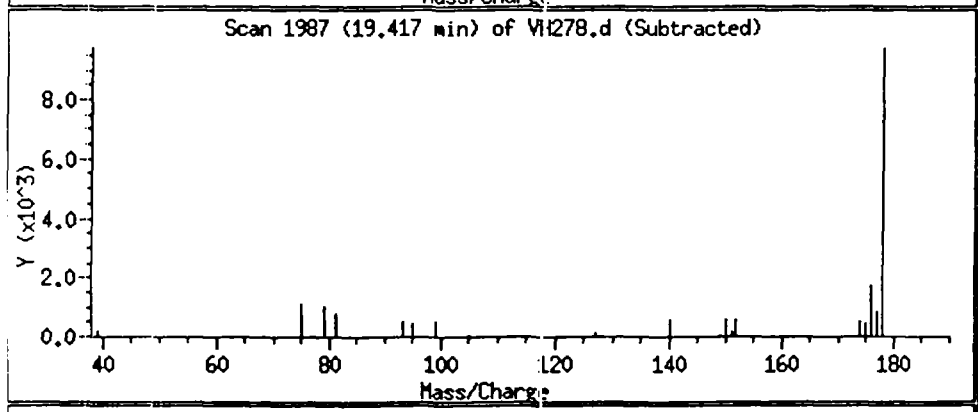
Sample ID : SITE#2 5-27-98 24HR

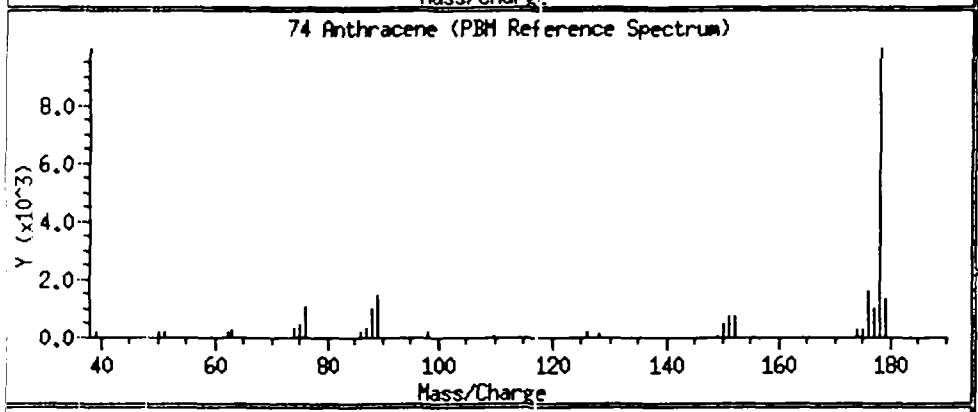
Column phase : DB-5 Column diameter : 0.32

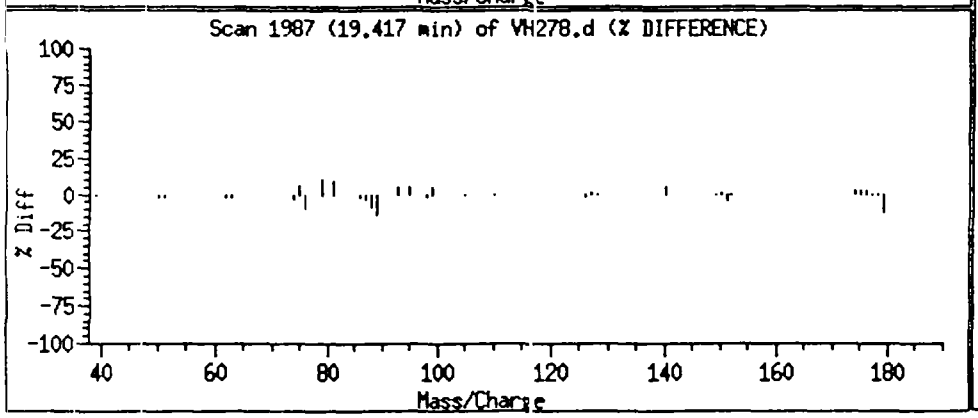
Volume Injected (uL) : 1.0

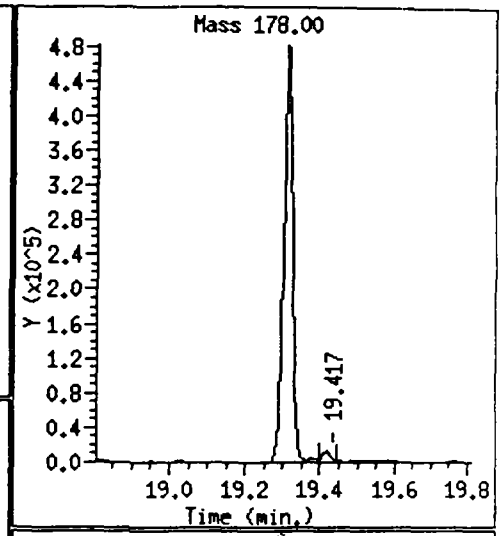
74 Anthracene

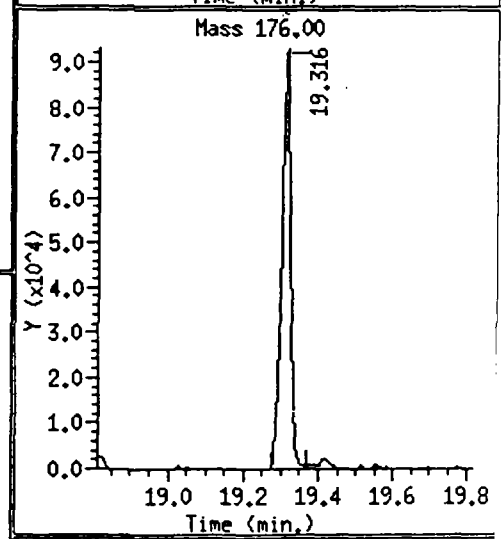


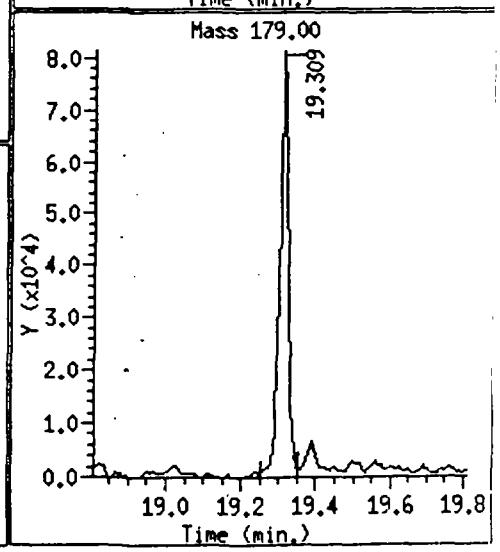












Data File: /chem/V.i/06-08-98.b/VH278.d

Date : 08-JUN-1998 23:10

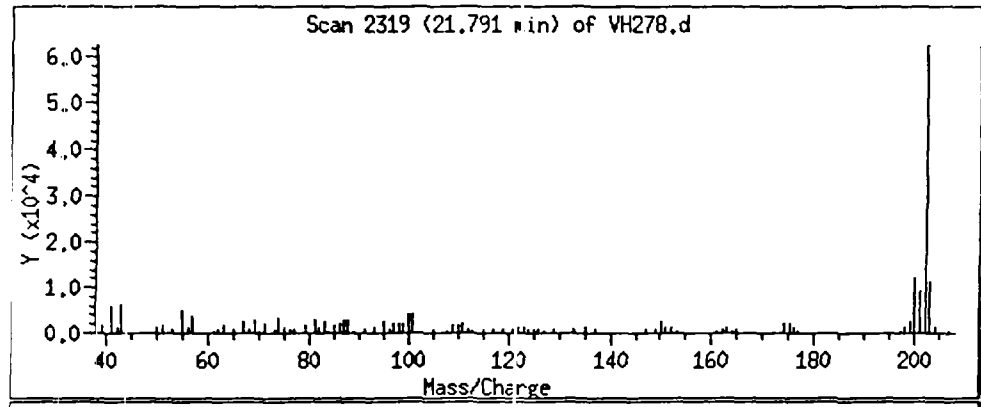
Instrument : V.i

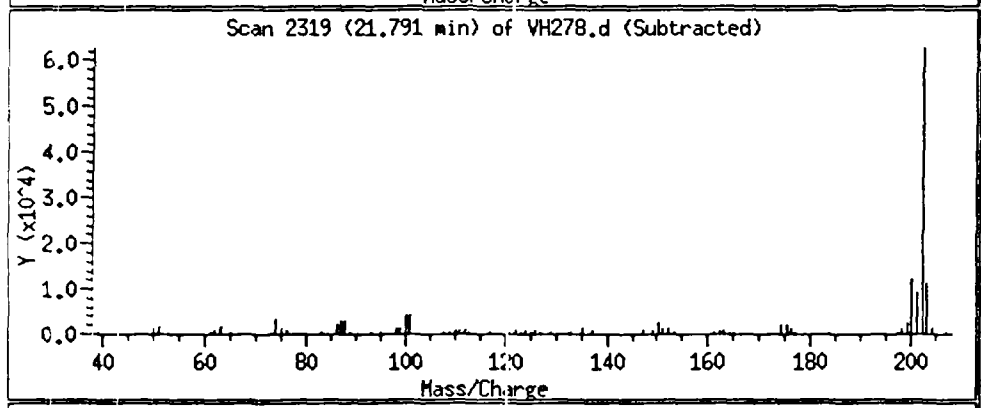
Sample ID : SITE#2 5-27-98 24HR

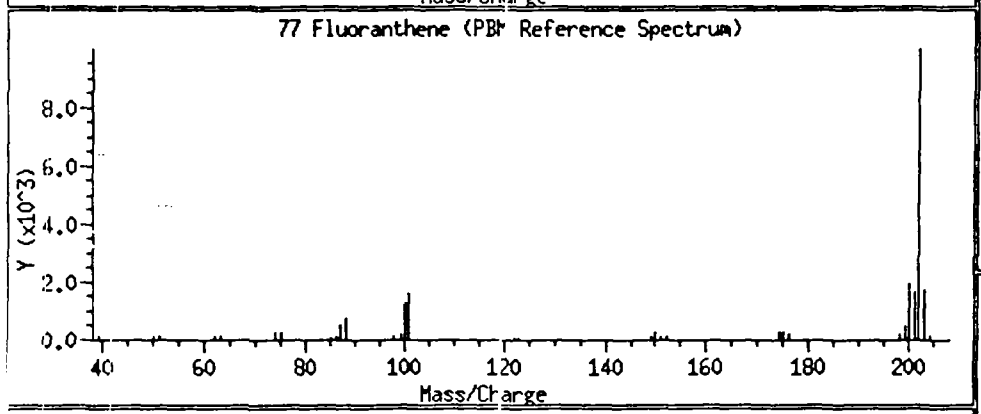
Column phase : DB-5 Column diameter : 0.32

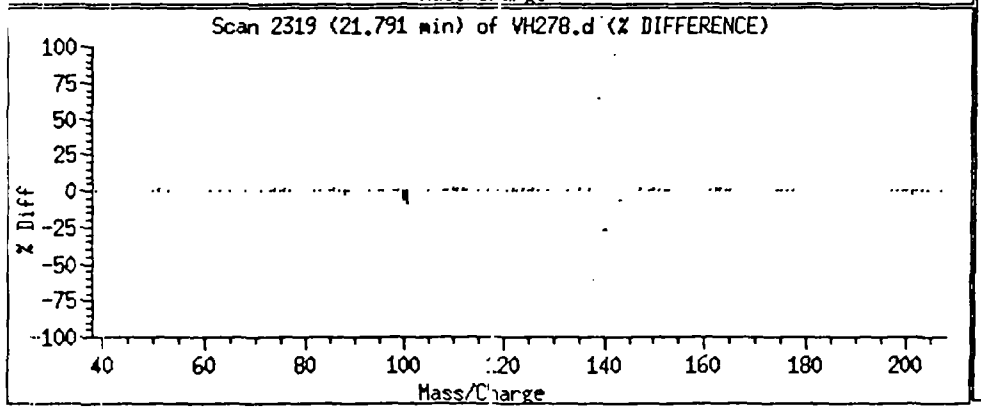
Volume Injected (uL) : 1.0

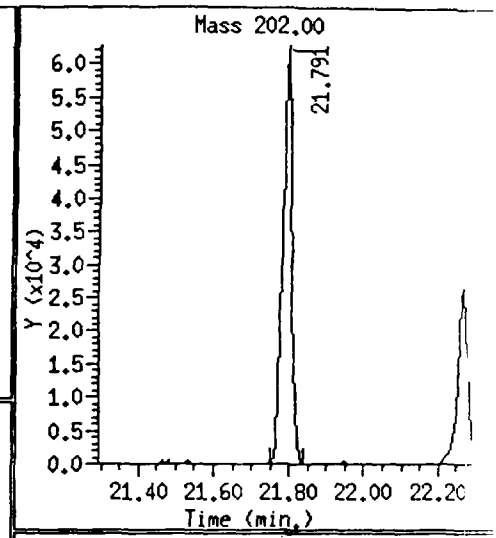
77 Fluoranthene

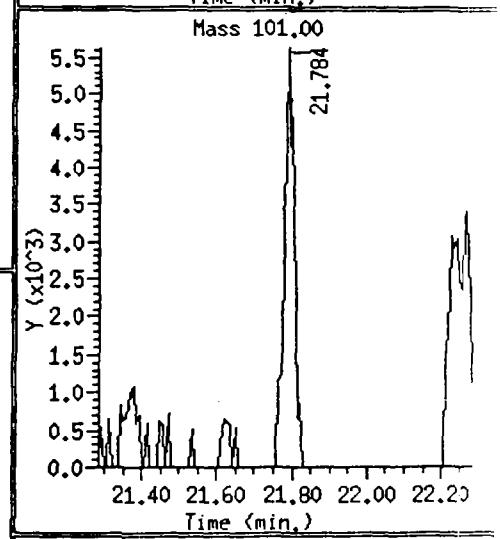


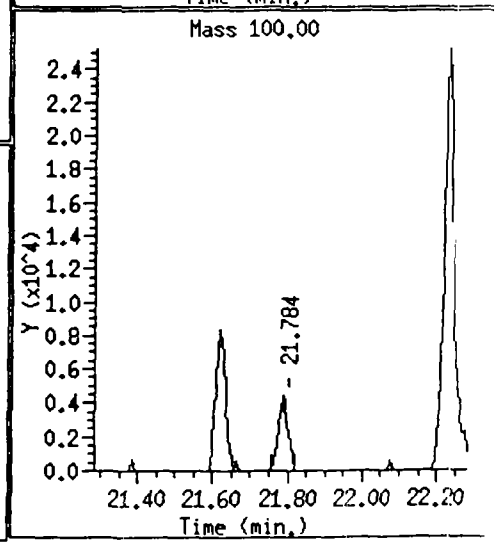












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH274

Method 8270A PUF
Sample ID: SITE#2 5-28-98 8hour

Client Project: Hull & Assoc	Date Received: 06/02/98	Response File: VH267
TLI ID: 209-67-2,6	Date Extracted: 06/04/98	
	Date Analyzed : 06/08/98	
	Dilution Factor: 1.00	

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	9.06	BJ	12.56		10
Acenaphthene-d_{10}		IS 3	16.15		
Fluorene	6.59	J	17.29		10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene	20.58	B	19.31		10
Anthracene	0.93	J	19.42		10
Fluoranthene	2.67	J	21.79		10
Chrysene-d_{12}		IS 5	24.88		
Perylene-d_{12}		IS 6	28.93		
Benzo(a)pyrene		U		0.81	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	93.10	19.38	4	93
Pyrene-d_{10}	108.30	22.23	5	108
Terphenyl-d_{14}	98.33	22.60	5	98

Reviewed by PAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

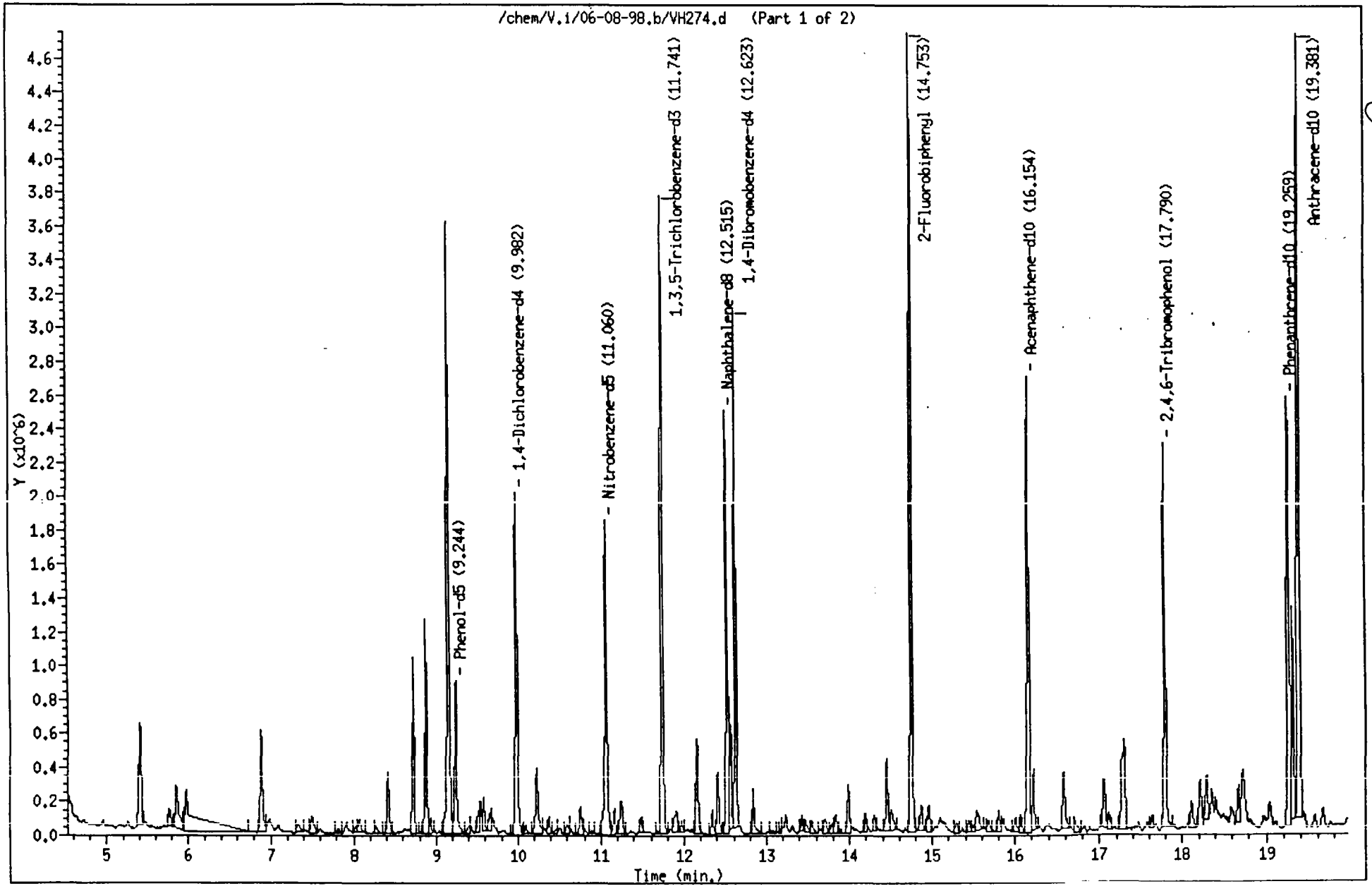
Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

Savar v3.7
Printed: 15:14 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH274.d
Date : 08-JUN-1998 19:54
Instrument : V.i
Sample ID : SITE#2 5-28-98 8HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



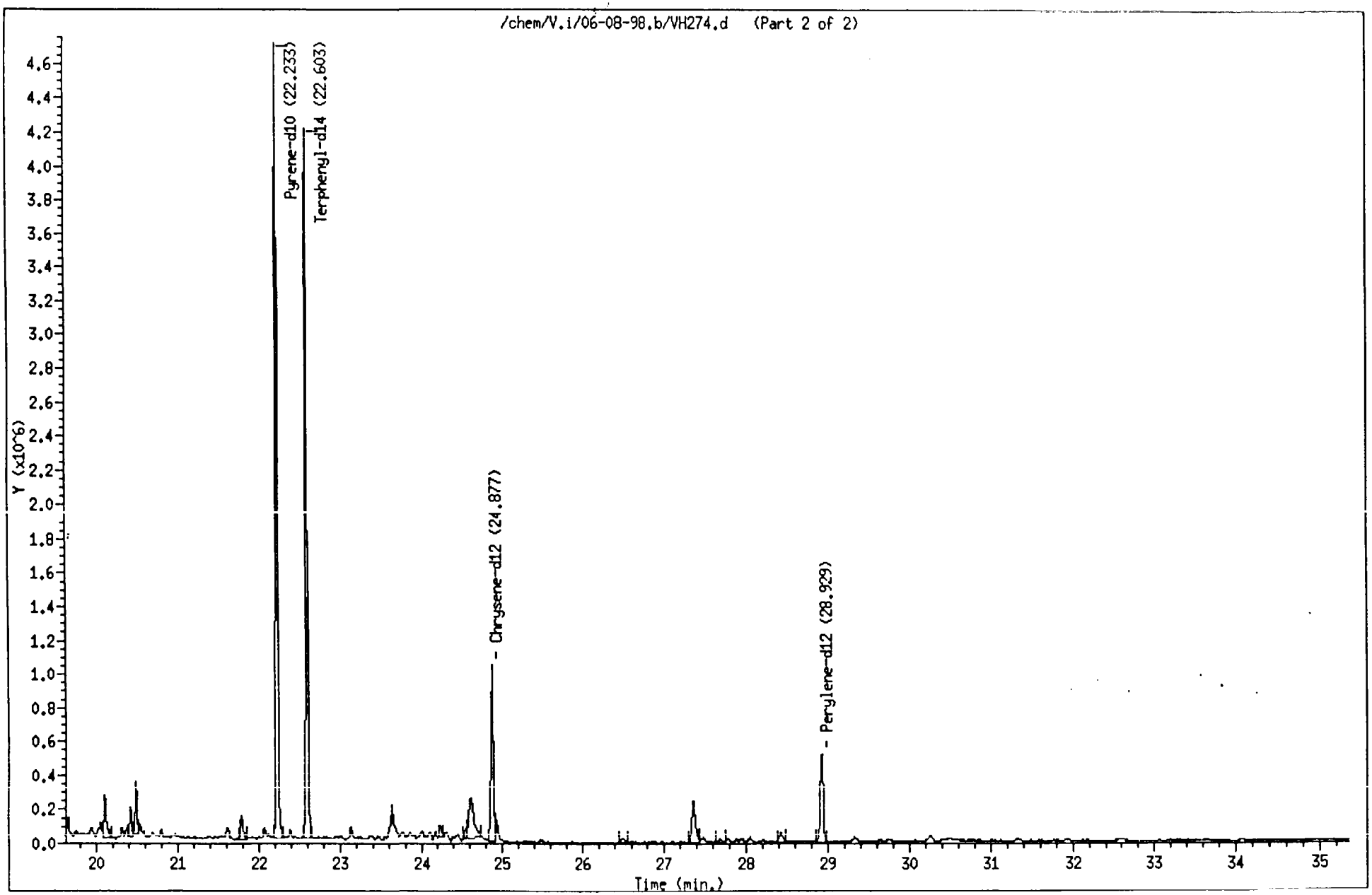
Data Review: PAB
Date: 6/12/98

88

Data File: /chem/V.i/06-08-98.b/VH274.d
Date : 08-JUN-1998 19:54
Instrument : V.i
Sample ID : SITE#2 5-28-98 8HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH274.d
Lab. Id. : 45869
Inj Date : 08-JUN-1998 19:54 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#2 5-28-98 3HOUR
Misc Info : 209-67-2,6
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 7
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

					CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
	Compounds	QUANT S G MASS	RT (REL RT)	RESPONSE		
*	1 1,4-Dichlorobenzene-d4	152.00	9.982(1.000)	489467	40.00	
*	2 Naphthalene-d8	136.00	12.515(1.000)	1761355	40.00	
*	3 Acenaphthene-d10	164.00	16.154(1.000)	967494	40.00	
*	4 Phenanthrene-d10	188.00	19.259(1.000)	1614299	40.00	
*	5 Chrysene-d12	240.00	24.877(1.000)	624560	40.00	
*	6 Perylene-d12	264.00	28.929(1.000)	498551	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.244(0.926)	422278	28.57	28.57
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.060(0.884)	879714	58.80	58.80
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.741(0.938)	1051729	69.17	69.17
$	13 1,4-Dibromobenzene-d4	240.00	12.623(1.265)	844162	76.59	76.59
$	14 2-Fluorobiphenyl	172.00	14.753(0.913)	2175402	78.32	78.32
$	15 2,4,6-Tribromophenol	329.80	17.797(1.102)	363570	65.44	65.44
$	16 Anthracene-d10	188.00	19.381(1.006)	3088388	93.10	93.10
$	17 Pyrene-d10	212.00	22.233(0.894)	3091095	108.28	108.28
$	18 Terphenyl-d14	244.00	22.603(0.909)	1761064	98.36	98.36
	42 Naphthalene	128.00	12.558(1.003)	383913	9.07	9.07(a)
	46 2-Methylnaphthalene	142.00	13.985(1.117)	104547	4.00	4.00(a)
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	154.00	16.218(1.004)	107714	4.64	4.64(a)
	65 Fluorene	166.00	17.292(1.070)	203348	6.59	6.59(a)
	73 Phenanthrene	178.00	19.309(1.003)	761732	20.59	20.59
	74 Anthracene	178.00	19.42 ~~19.309(1.003)~~	~~761732~~ 31958	~~22.07~~	~~22.07~~ (M) PAB
	77 Fluoranthene	202.00	21.792(1.132)	100758	2.67	2.67(a)
	78 Pyrene	Compound Not Detected.				
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-08-98.b/VH274.d

Date : 08-JUN-1998 19:54

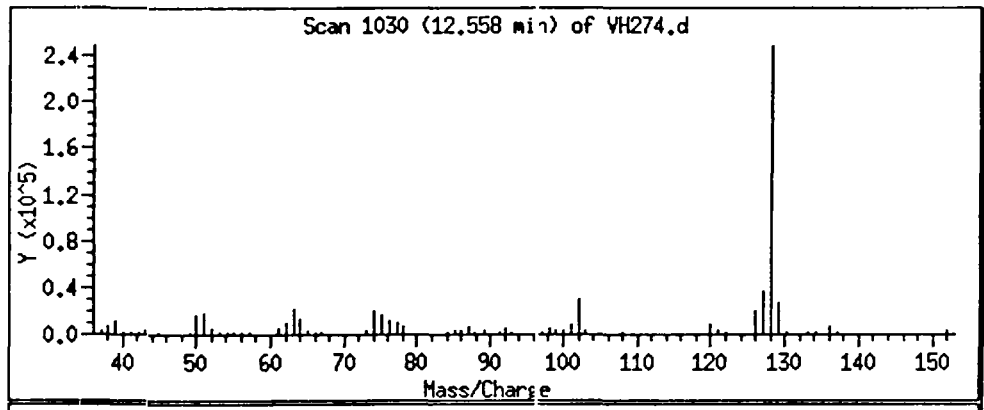
Instrument : V.i

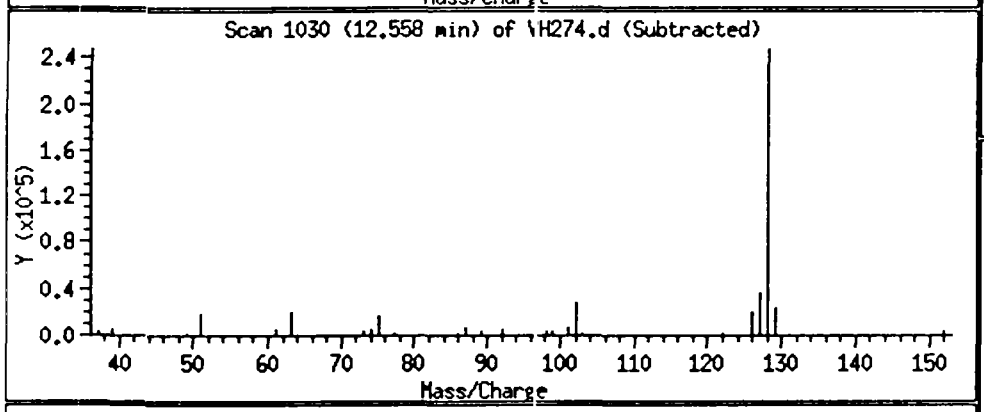
Sample ID : SITE#2 5-28-98 8HR

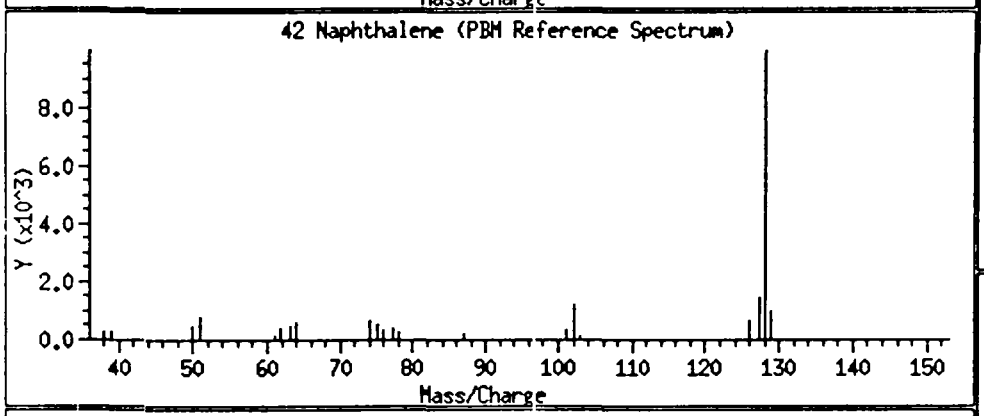
Column phase : DB-5 Column diameter : 0.32

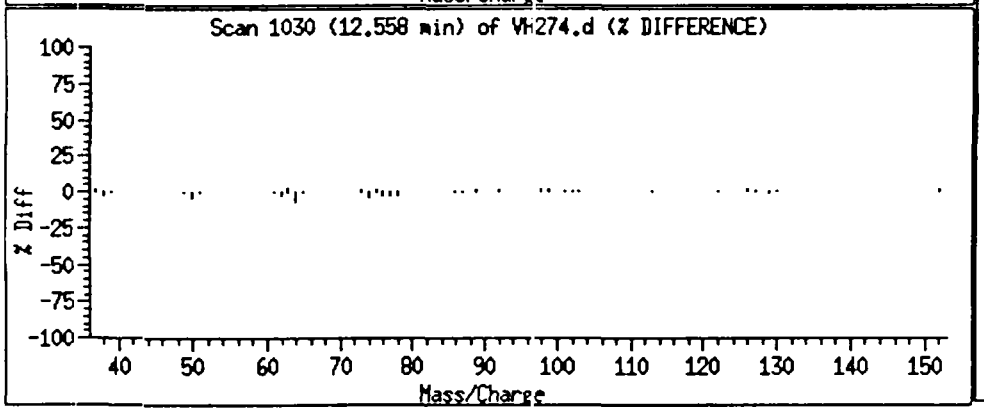
Volume Injected (uL) : 1.0

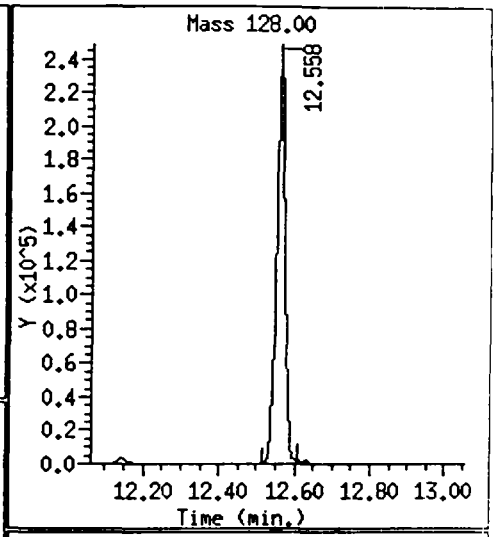
42 Naphthalene

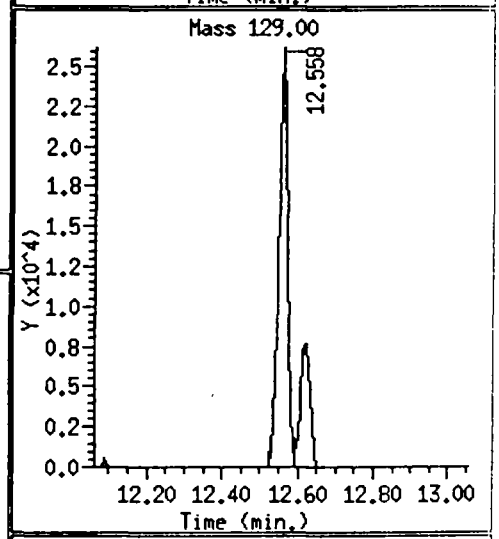


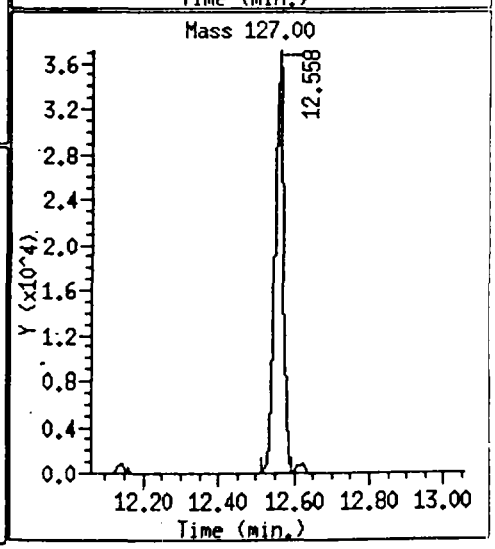












Data File: /chem/V.i/06-08-98.b/VH274.d

Date : 08-JUN-1998 19:54

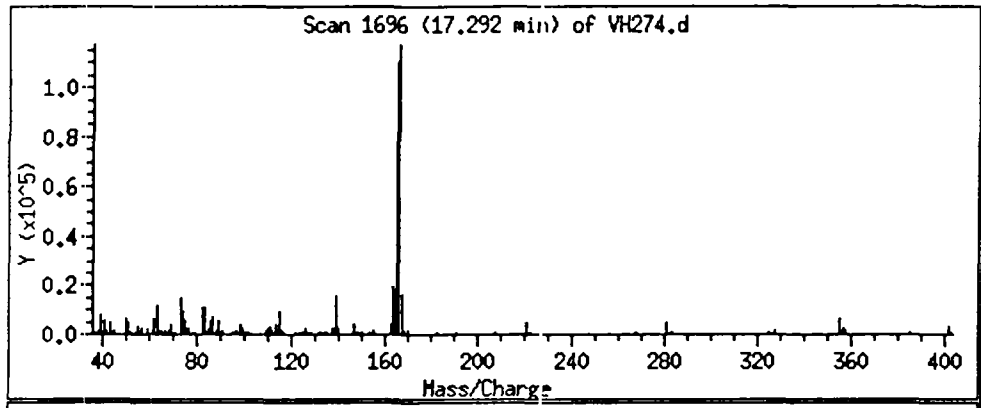
Instrument : V.i

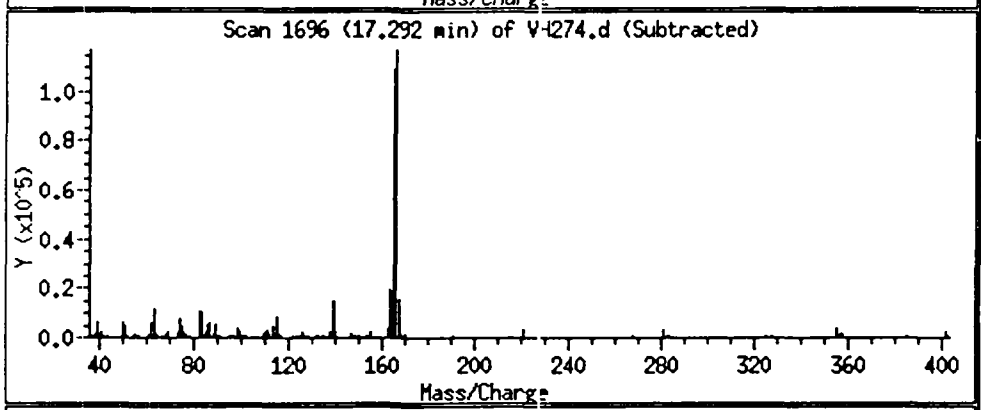
Sample ID : SITE#2 5-28-98 8HR

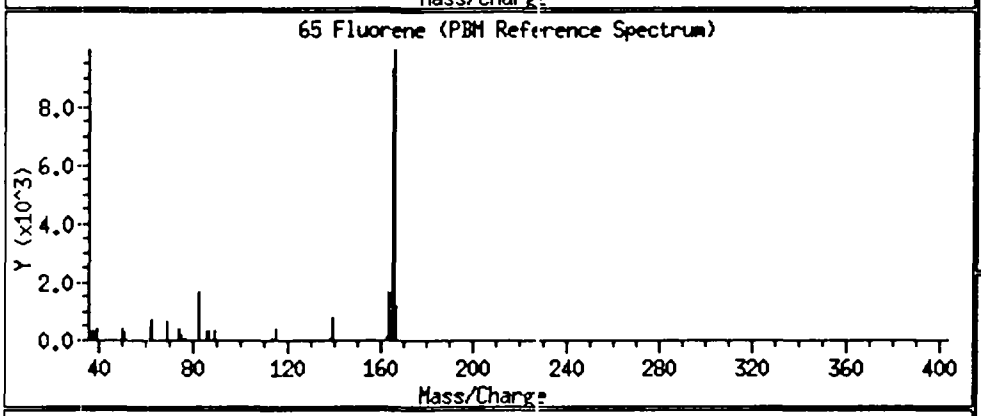
Column phase : DB-5 Column diameter : 0.32

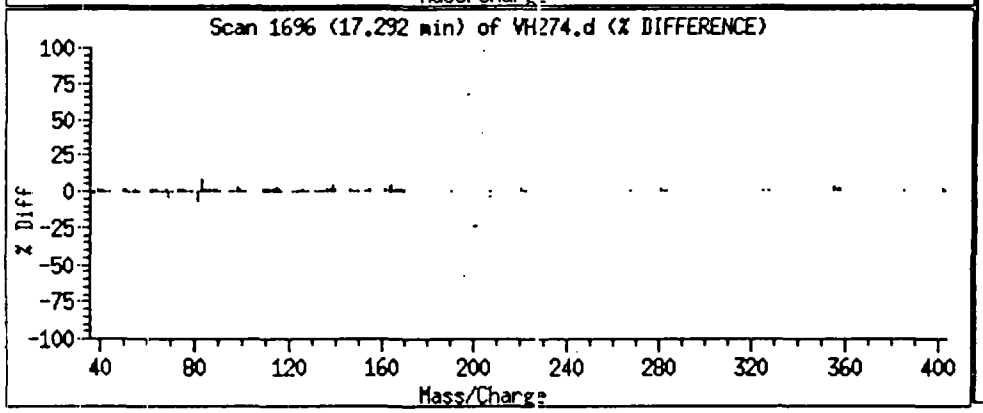
Volume Injected (uL) : 1.0

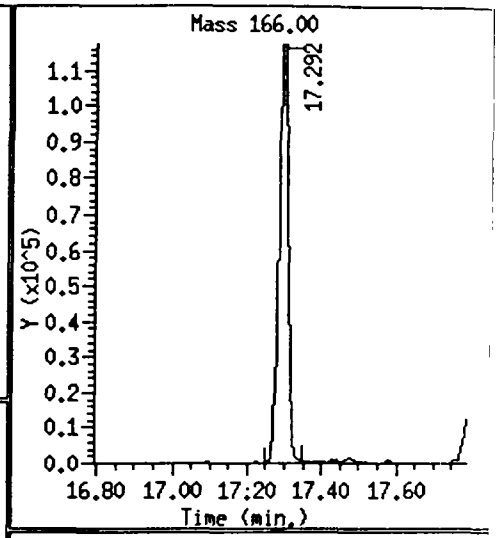
65 Fluorene

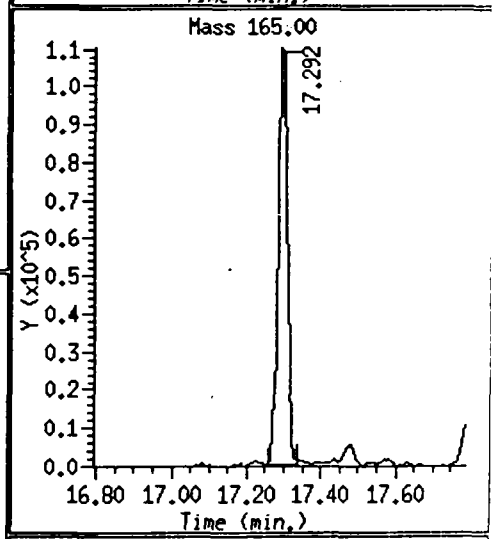


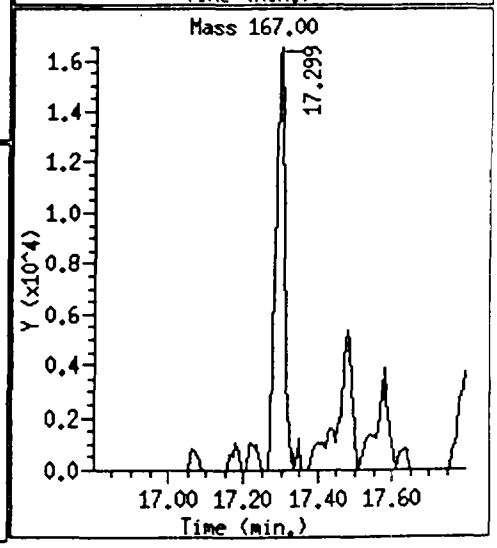












Data File: /chem/V.i/06-08-98.b/VH274.d

Date : 08-JUN-1998 19:54

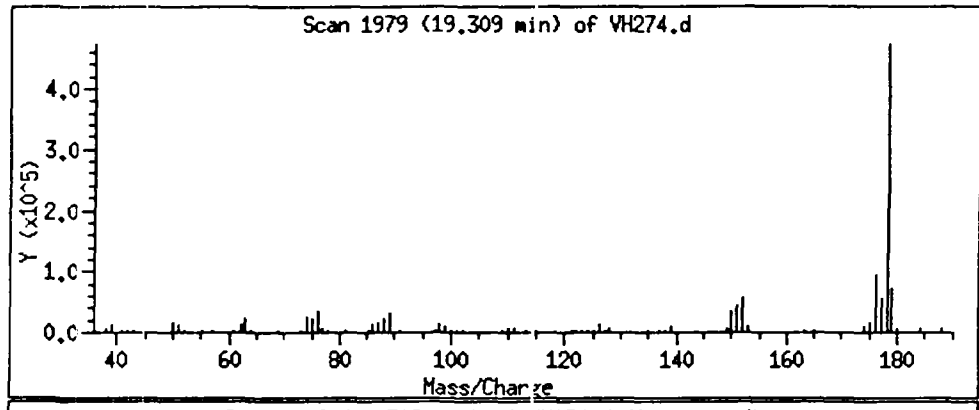
Instrument : V.i

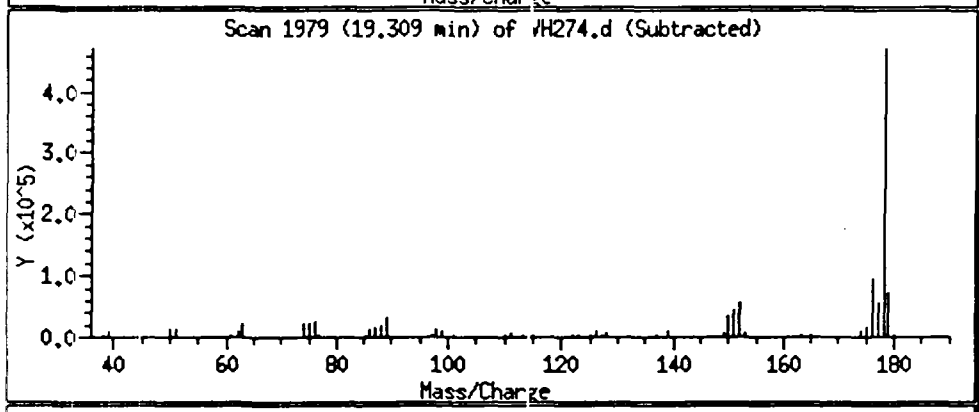
Sample ID : SITE#2 5-28-98 8HR

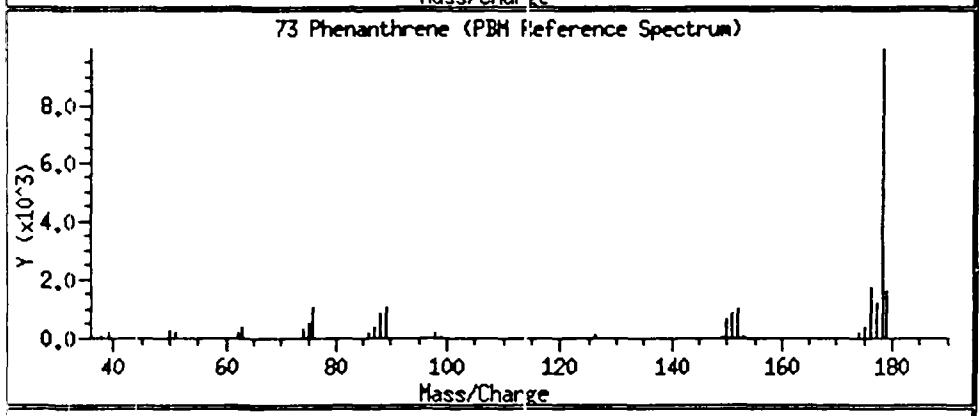
Column phase : DB-5 Column diameter : 0.32

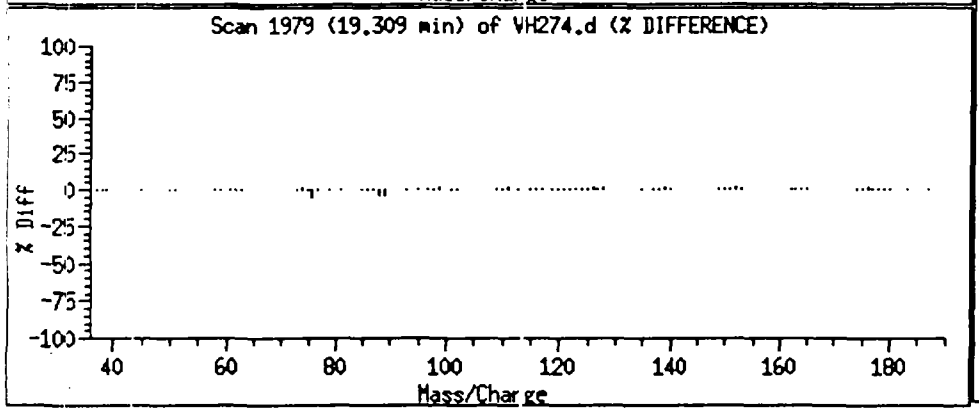
Volume Injected (uL) : 1.0

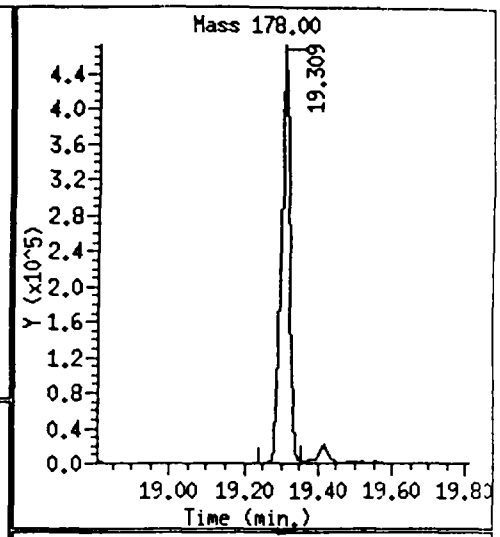
73 Phenanthrene

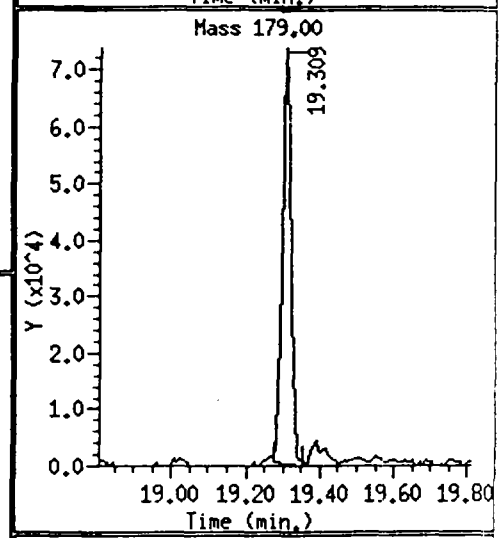


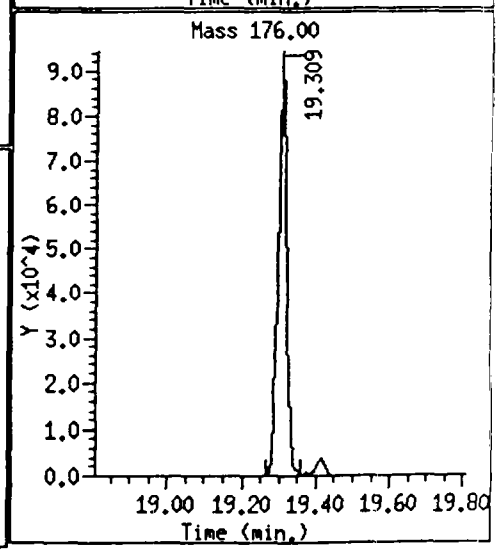












Data File: /chem/V.i/06-08-98.b/VH274.d

Date : 08-JUN-1998 19:54

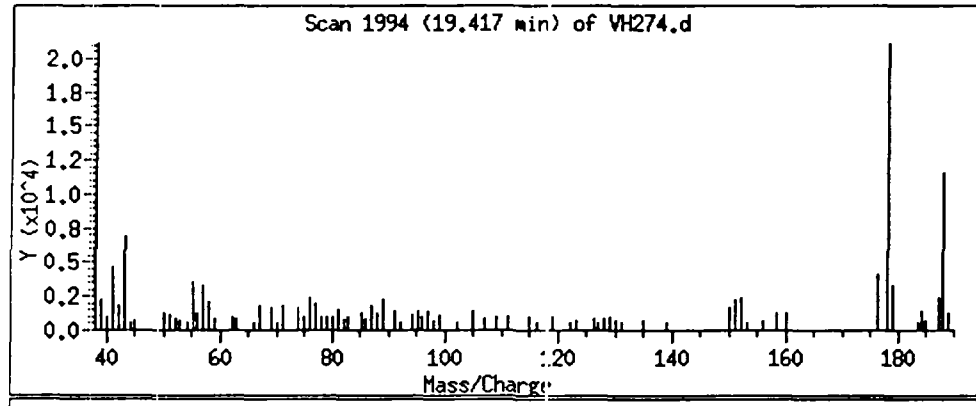
Instrument : V.i

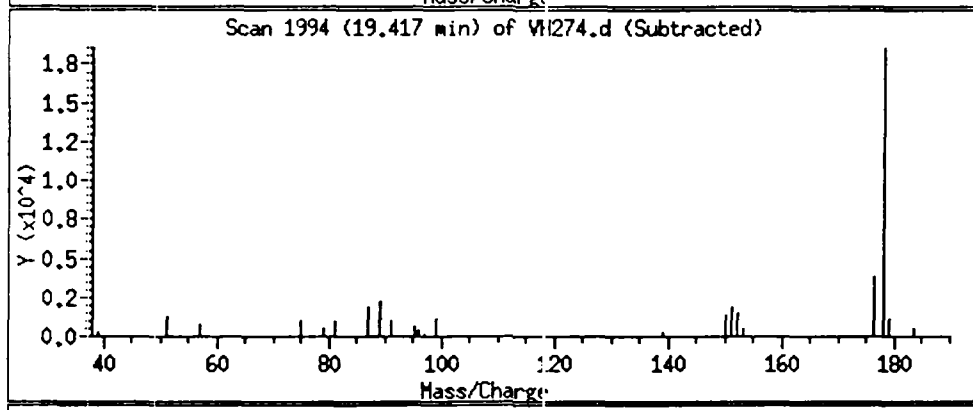
Sample ID : SITE#2 5-28-98 8HR

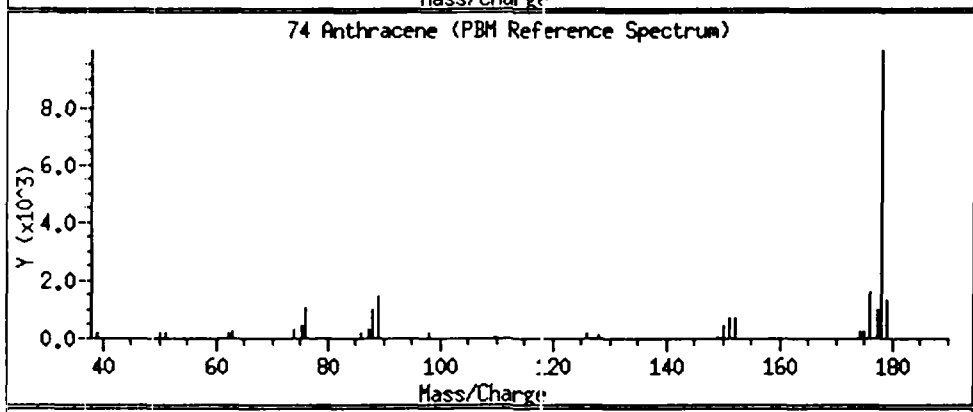
Column phase : DB-5 Column diameter : 0.32

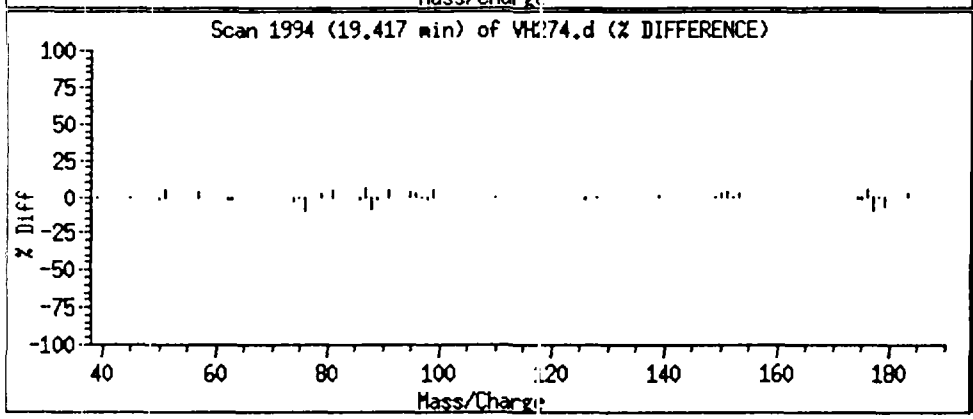
Volume Injected (uL) : 1.0

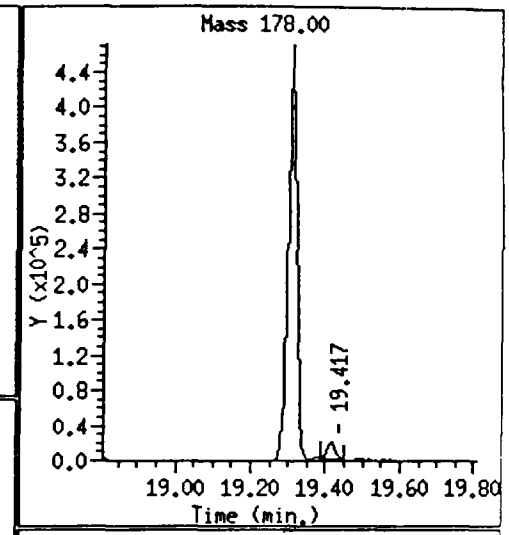
74 Anthracene

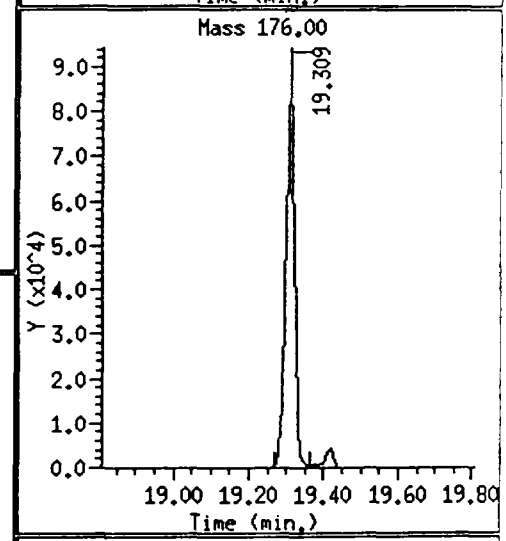


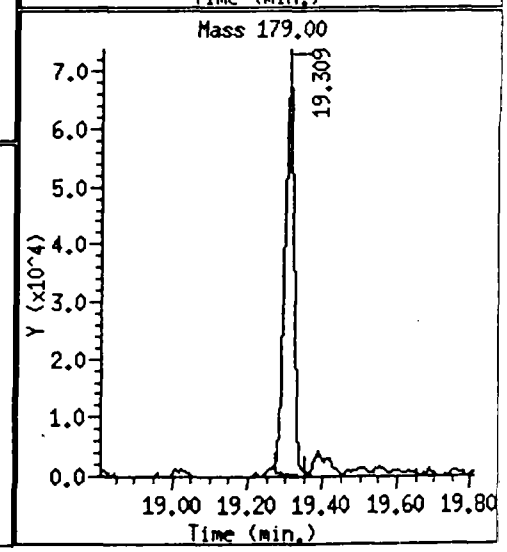












Data File: /chem/V.i/06-08-98.b/VH274.d

Date : 08-JUN-1998 19:54

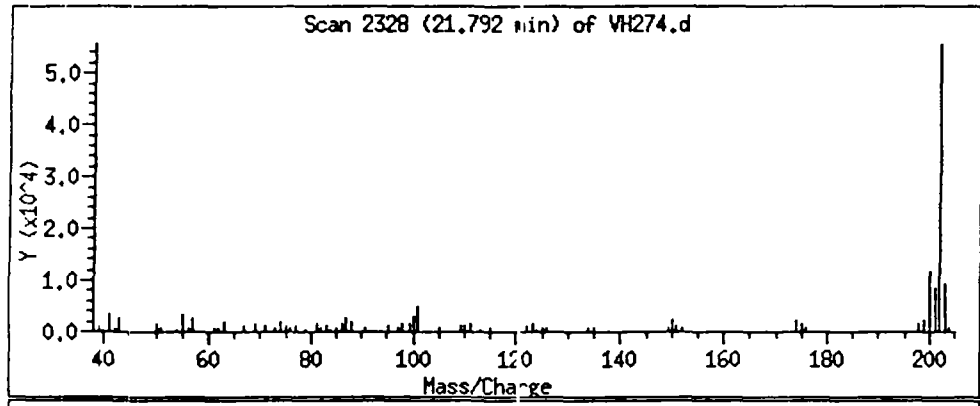
Instrument : V.i

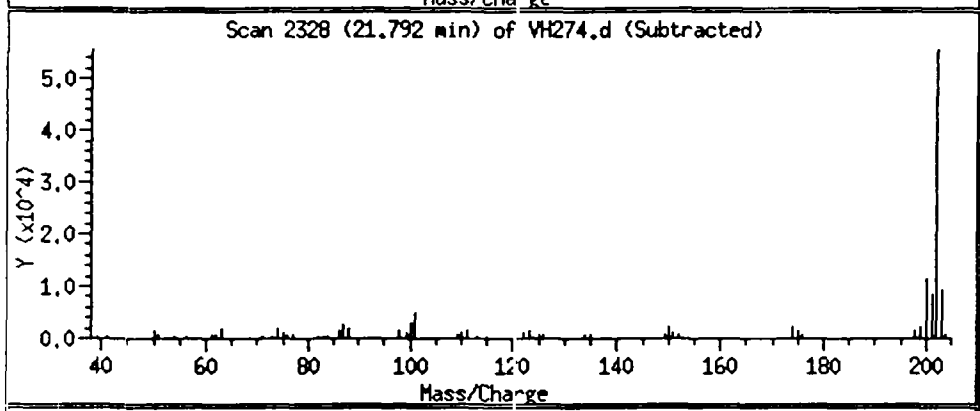
Sample ID : SITE#2 5-28-98 8HR

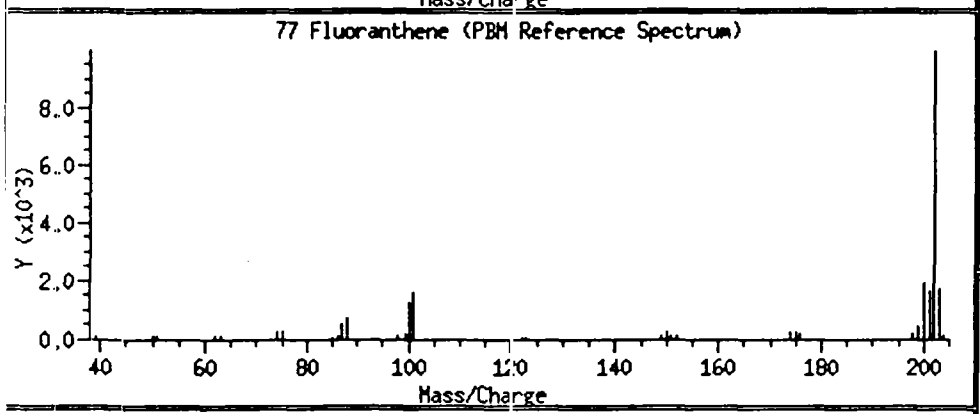
Column phase : DB-5 Column diameter : 0.32

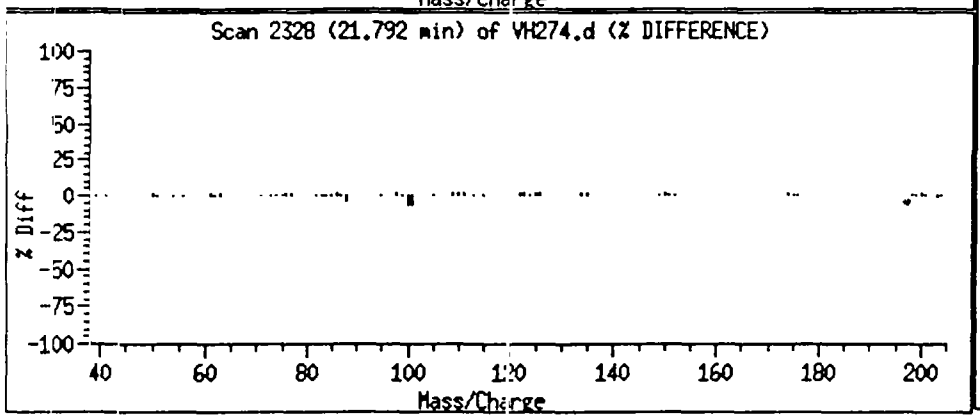
Volume Injected (uL) : 1.0

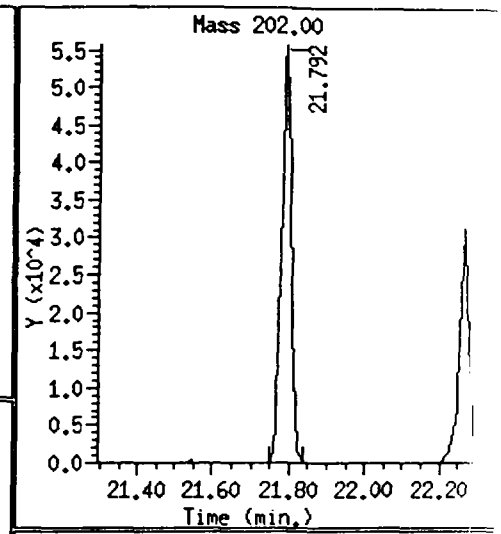
77 Fluoranthene

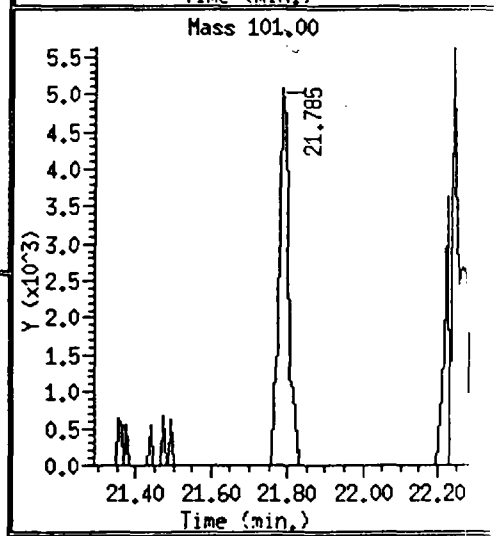


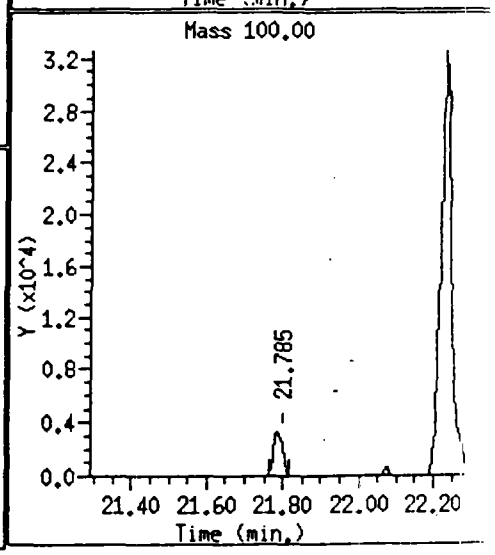












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH287

Method 8270A PUF
Sample ID: SITE#2 5-29-98 24hour

Client Project: Hull & Assoc TLI ID: 209-67-19,22	Date Received: 06/02/98 Date Extracted: 06/04/98 Date Analyzed : 06/09/98	Response File: VH284
	Dilution Factor: 1.00	

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	58.73	B	12.57		10
Acenaphthene-d_{10}		IS 3	16.16		
Fluorene	12.06		17.30		10
Phenanthrene-d_{10}		IS 4	19.27		
Phenanthrene	40.45	B	19.32		10
Anthracene	1.71	J	19.42		10
Fluoranthene	5.30	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.31	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	79.67	19.38	4	80
Pyrene-d_{10}	87.96	22.23	5	88
Terphenyl-d_{14}	77.35	22.60	5	77

Reviewed by BAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

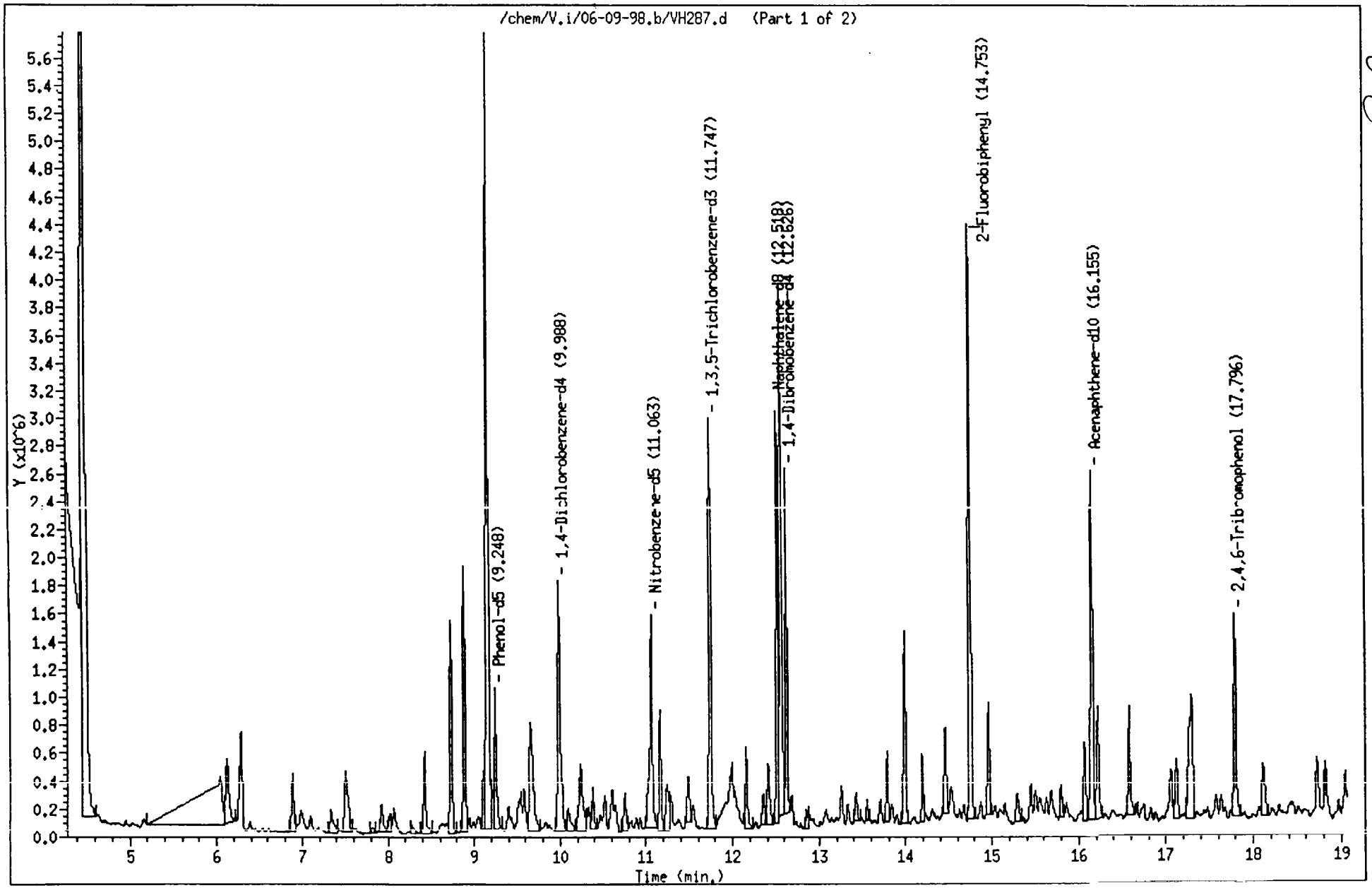
Savar v3.7
Printed: 15:16 06/12/1998

Data File: /chem/V.i/06-09-98.b/VH287.d
Date : 09-JUN-1998 13:55
Instrument : V.i
Sample ID : SITE#2 5/29/98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

98

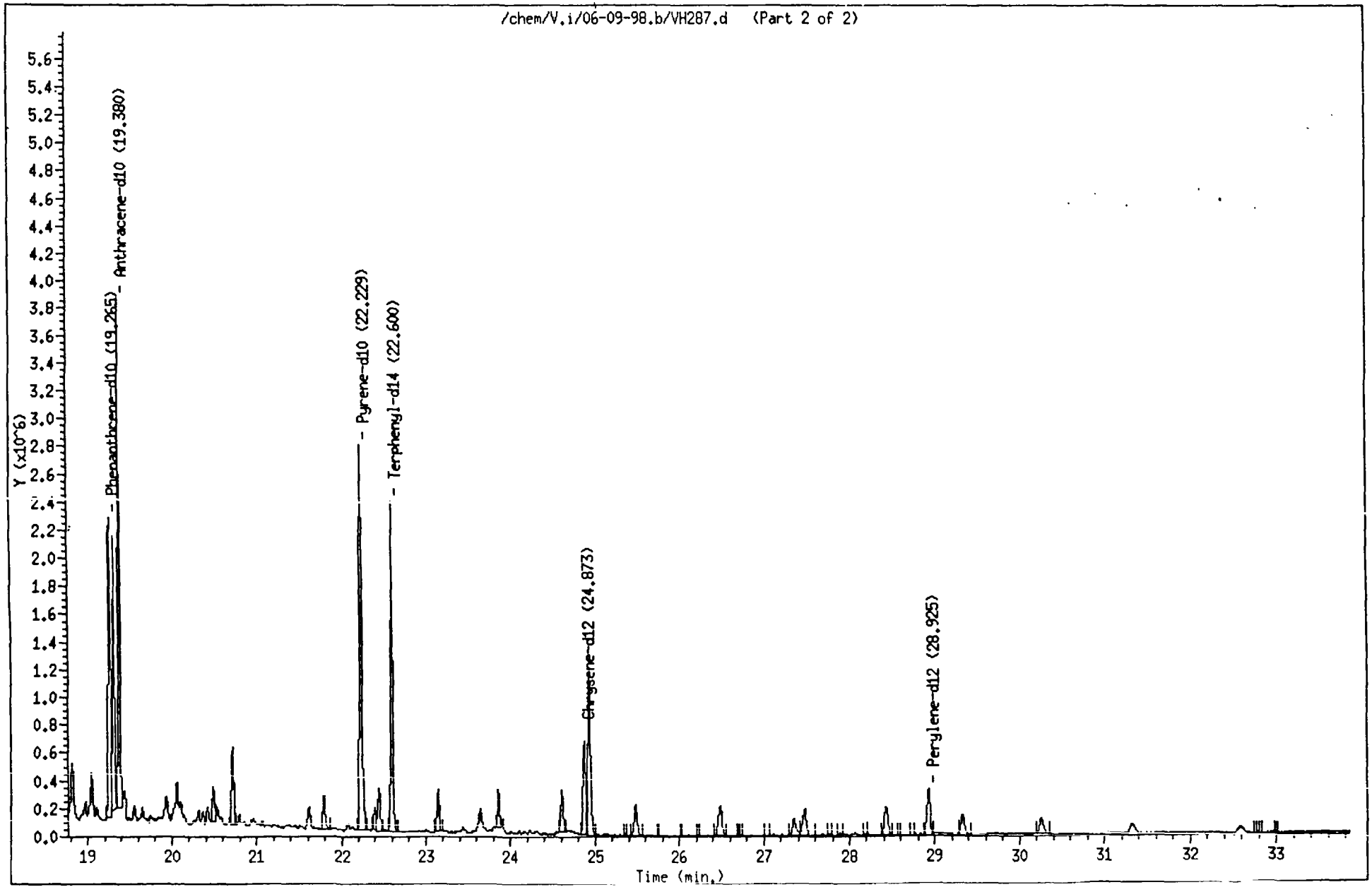


Data Review: PaB
Date: 6/12/98

Data File: /chem/V.i/06-09-98.b/VH287.d
Date : 09-JUN-1998 13:55
Instrument : V.i
Sample ID : SITE#2 5/29/98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-09-98.b/VH287.d
Lab. Id. : 45869
Inj Date : 09-JUN-1998 13:55 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#2 5/29/98 24HOUR
Misc Info : 209-67-19,22
Comment :
Method : /chem/V.i/06-09-98.b/cclist.m
Meth Date : 09-Jun-1998 12:27 target
Cal Date : 09-JUN-1998 10:31 Cal File: VH284.d
Als bottle: 3
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.988(1.000)	418942	40.00	
*	2 Naphthalene-d8	136.00	12.518(1.000)	1593867	40.00	
*	3 Acenaphthene-d10	164.00	16.155(1.000)	847076	40.00	
*	4 Phenanthrene-d10	188.00	19.265(1.000)	1383191	40.00	
*	5 Chrysene-d12	240.00	24.873(1.000)	390260	40.00	
*	6 Perylene-d12	264.00	28.925(1.000)	309067	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.248(0.926)	381137	29.50	29.50
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.063(0.884)	711200	53.03	53.03
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.747(0.938)	829117	57.41	57.41
$	13 1,4-Dibromobenzene-d4	240.00	12.626(1.264)	657806	62.53	62.53
$	14 2-Fluorobiphenyl	172.00	14.753(0.913)	1758876	72.97	72.97
$	15 2,4,6-Tribromophenol	329.80	17.796(1.102)	197214	50.20	50.20
$	16 Anthracene-d10	188.00	19.380(1.006)	2264531	79.64	79.64
$	17 Pyrene-d10	212.00	22.229(0.894)	1765301	87.96	87.96
$	18 Terphenyl-d14	244.00	22.600(0.909)	942576	77.34	77.34
	42 Naphthalene	128.00	12.568(1.004)	2319117	58.73	58.73
	46 2-Methylnaphthalene	142.00	13.988(1.117)	523955	21.62	21.62
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	154.00	16.220(1.004)	247827	12.05	12.05
	65 Fluorene	166.00	17.301(1.071)	347639	12.06	12.06
	73 Phenanthrene	178.00	19.316(1.003)	1257514	40.44	40.44
	74 Anthracene	178.00	19.42 ~~19.316(1.003)~~	~~1257514~~ 49782	~~43.10~~	~~43.10~~ (M) PAB
	77 Fluoranthene	202.00	21.788(1.131)	162337	5.30	5.30(a)
	78 Pyrene	202.00	22.265(0.895)	79272	3.02	3.02(a)
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-09-98.b/VH287.d

Date : 09-JUN-1998 13:55

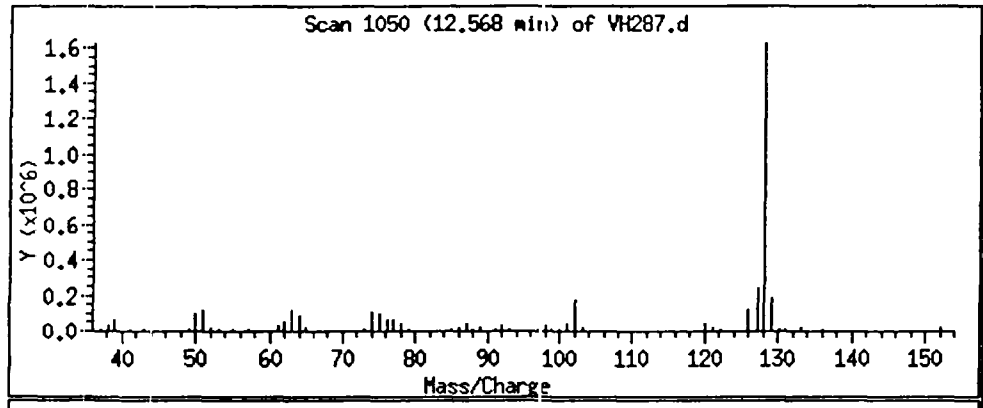
Instrument : V.i

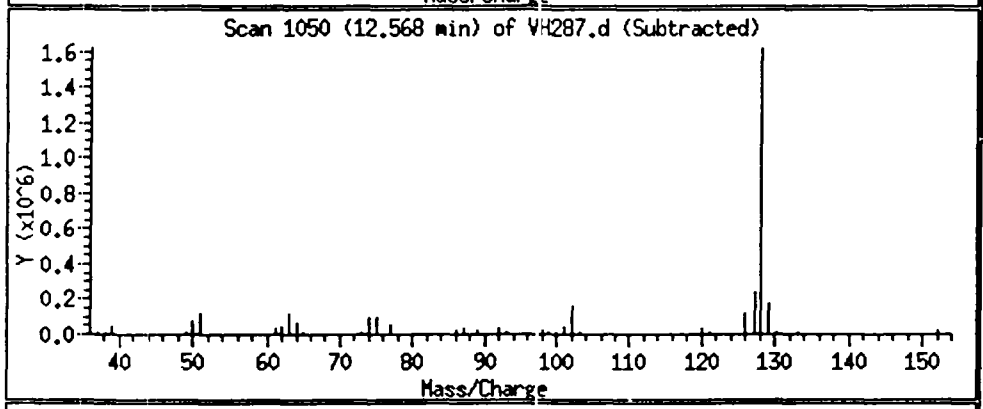
Sample ID : SITE#2 5/29/98 24HR

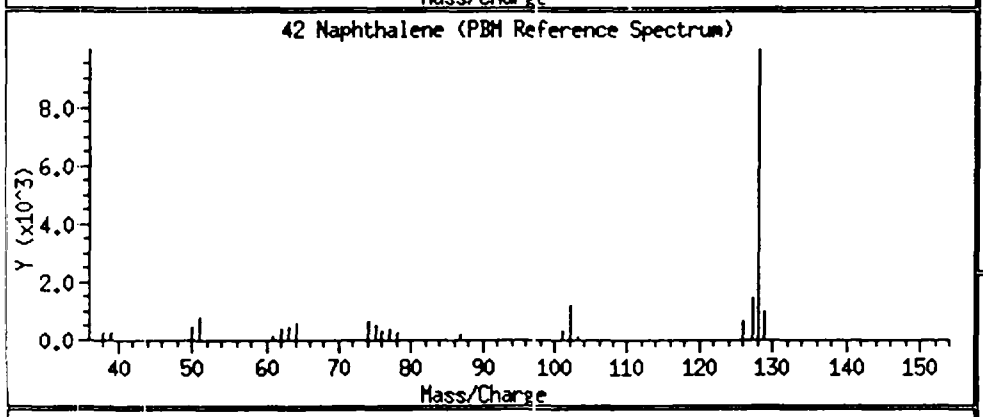
Column phase : DB-5 Column diameter : 0.32

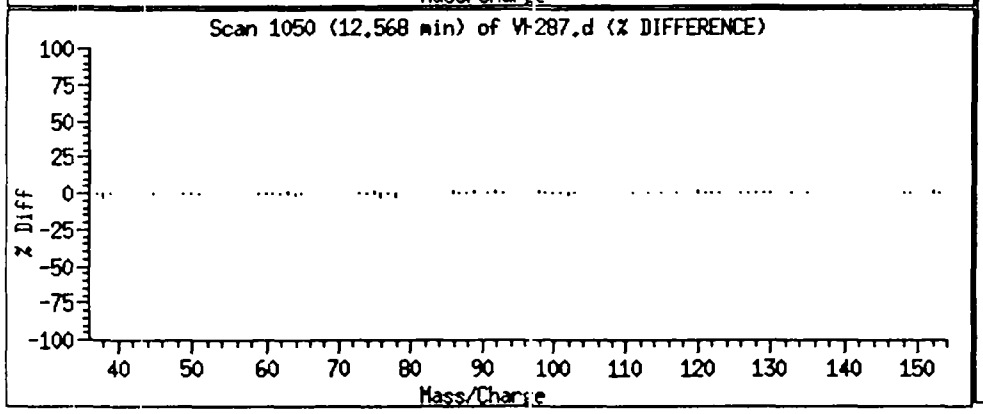
Volume Injected (uL) : 1.0

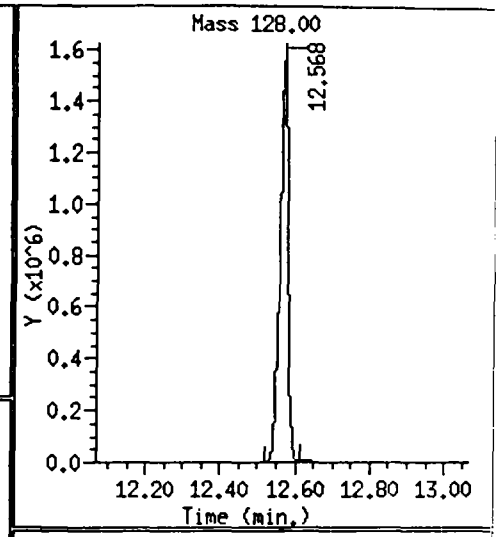
42 Naphthalene

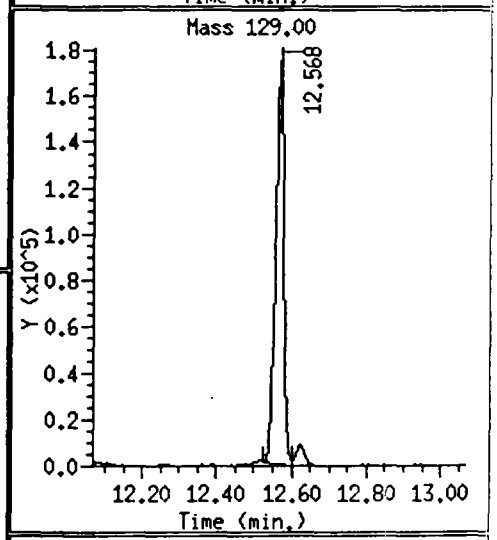


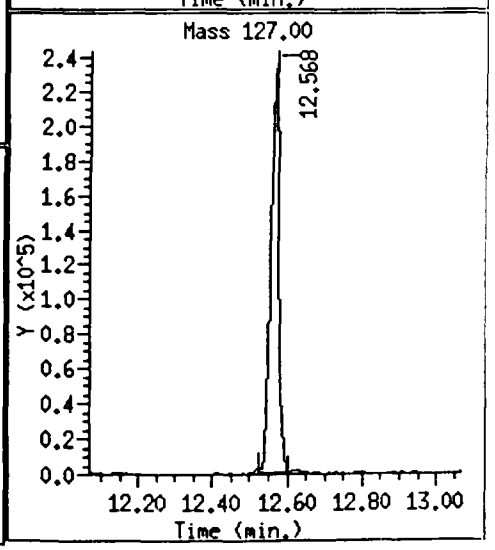












Data File: /chem/V.i/06-09-98.b/VH287.d

Date : 09-JUN-1998 13:55

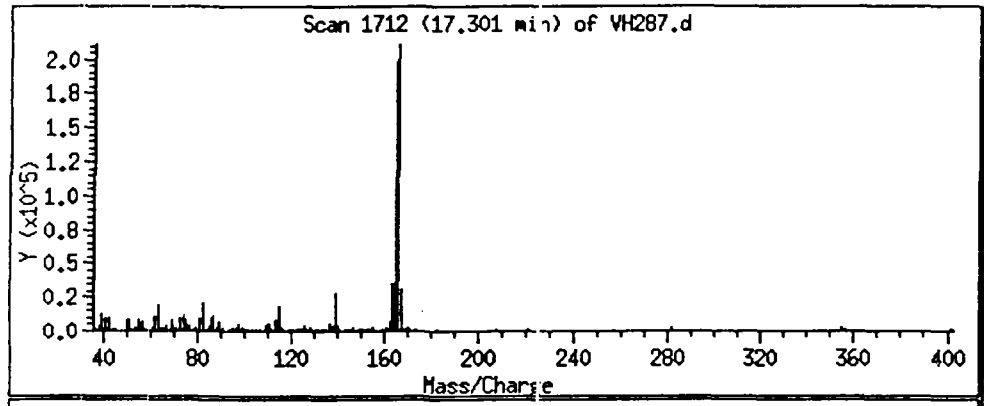
Instrument : V.i

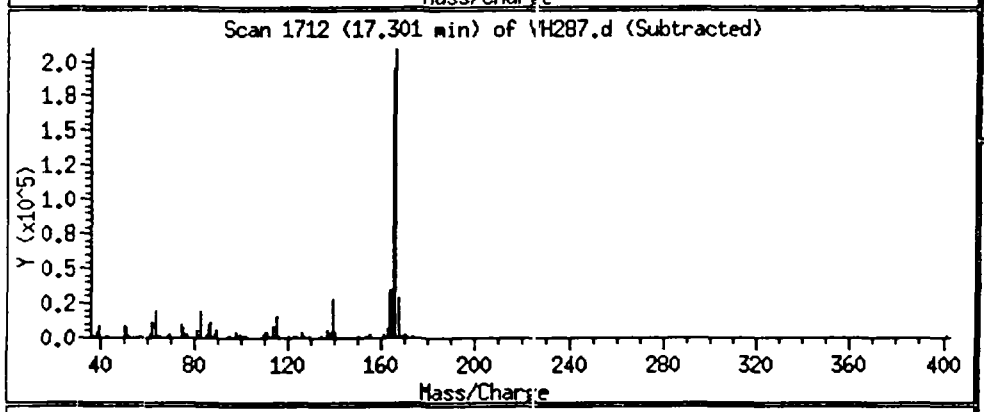
Sample ID : SITE#2 5/29/98 24HR

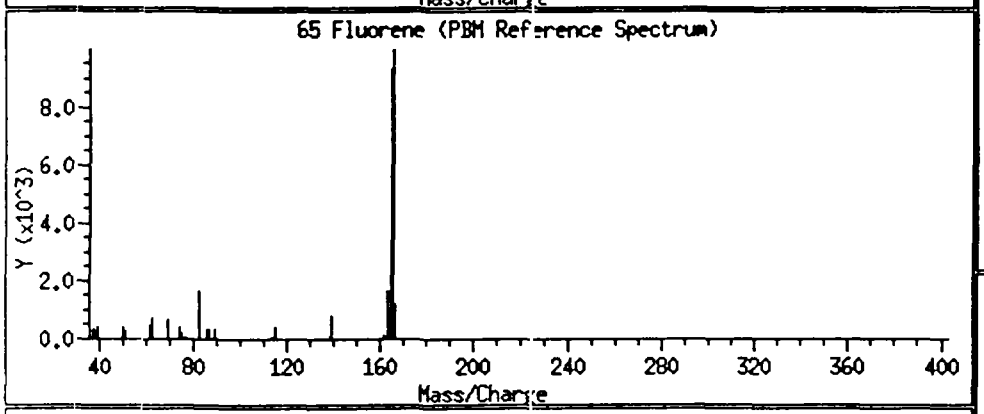
Column phase : DB-5 Column diameter : 0.32

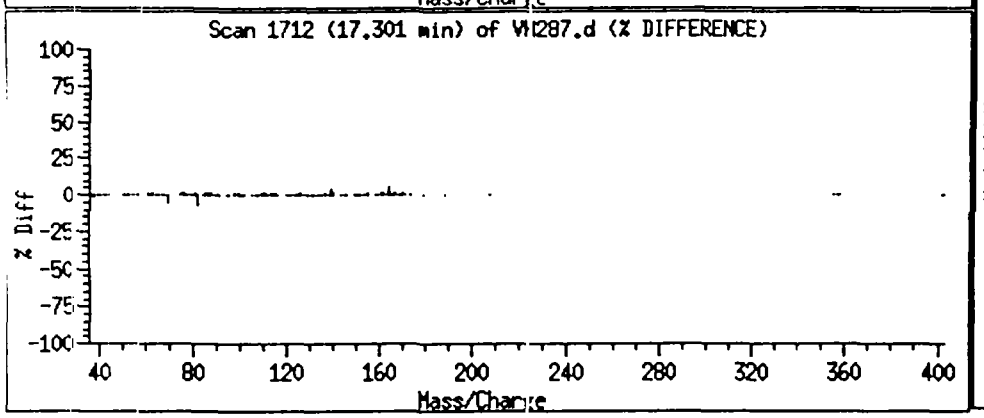
Volume Injected (uL) : 1.0

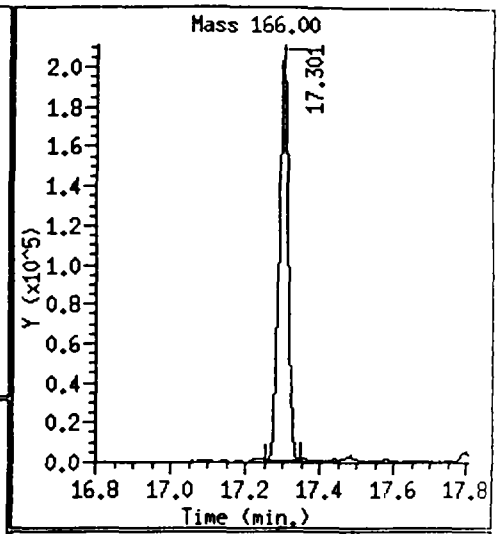
65 Fluorene

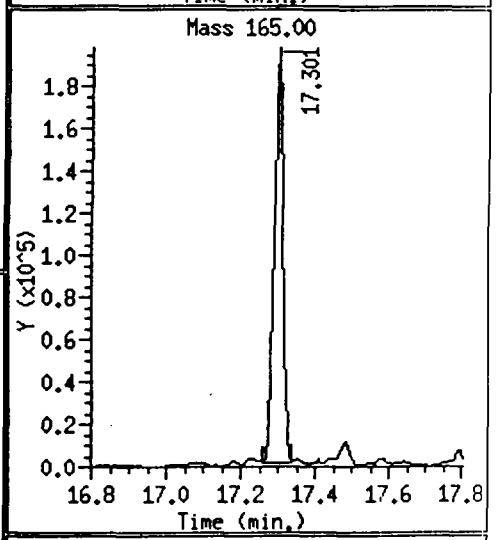


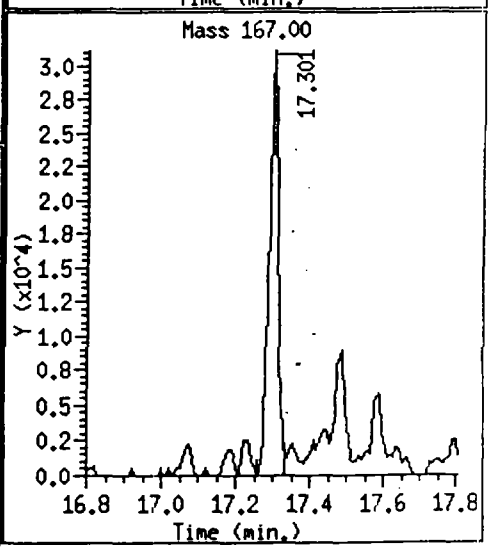












Data File: /chem/V.i/06-09-98.b/VH287.d

Date : 09-JUN-1998 13:55

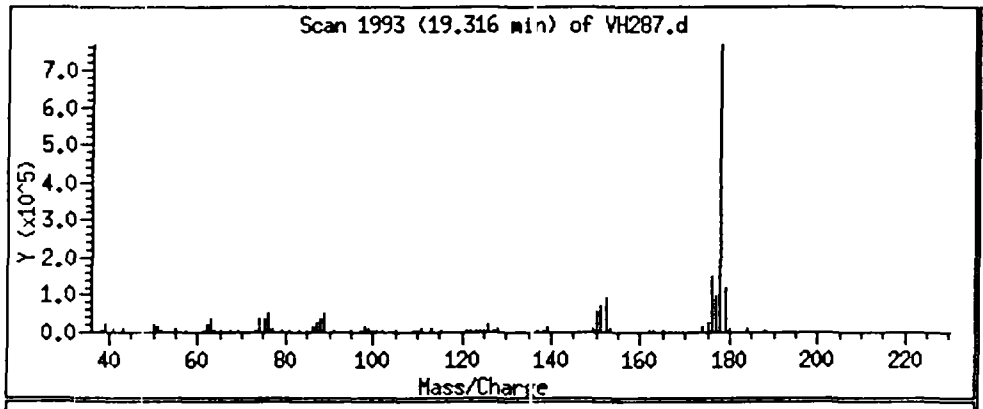
Instrument : V.i

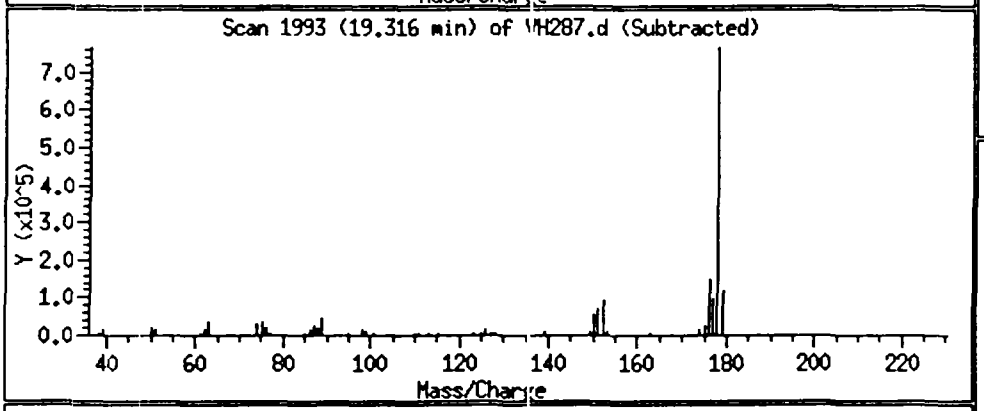
Sample ID : SITE#2 5/29/98 24HR

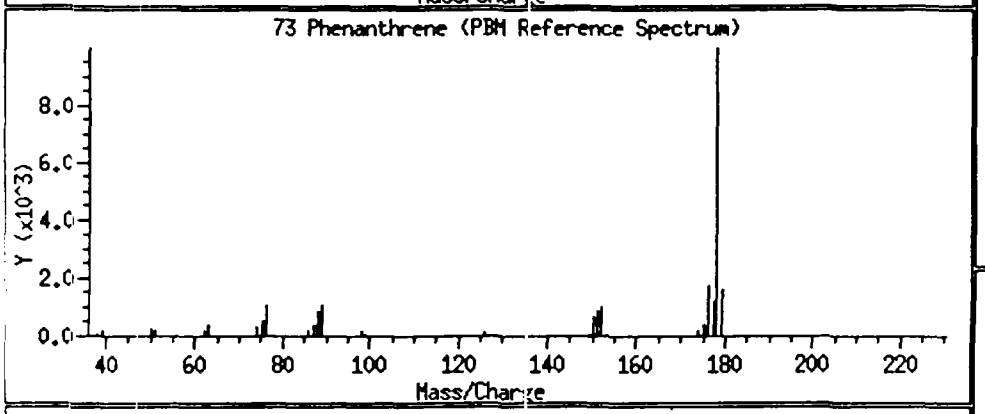
Column phase : DB-5 Column diameter : 0.32

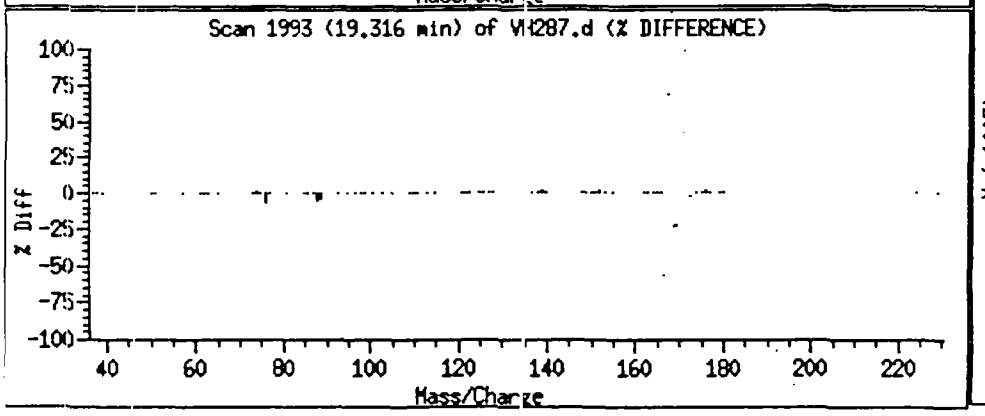
Volume Injected (uL) : 1.0

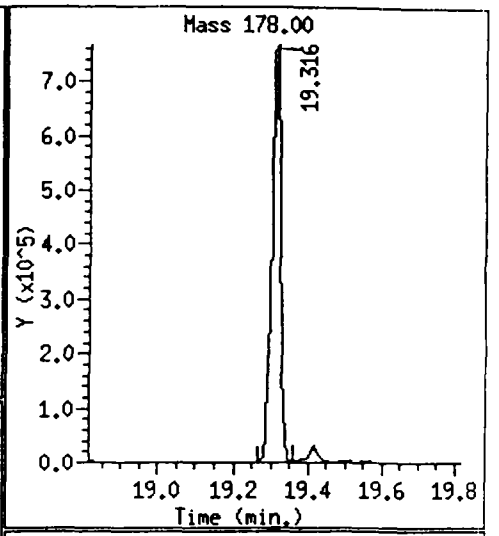
73 Phenanthrene

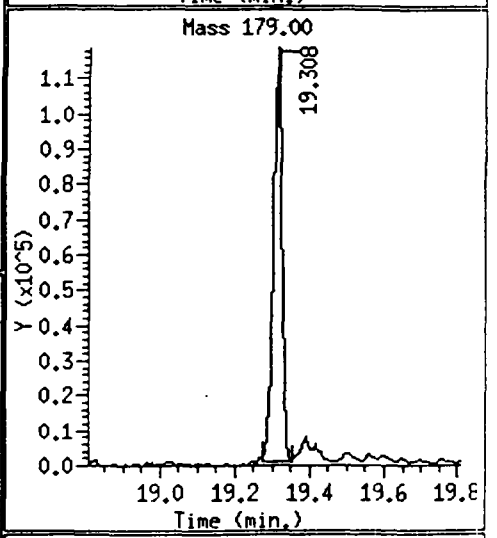


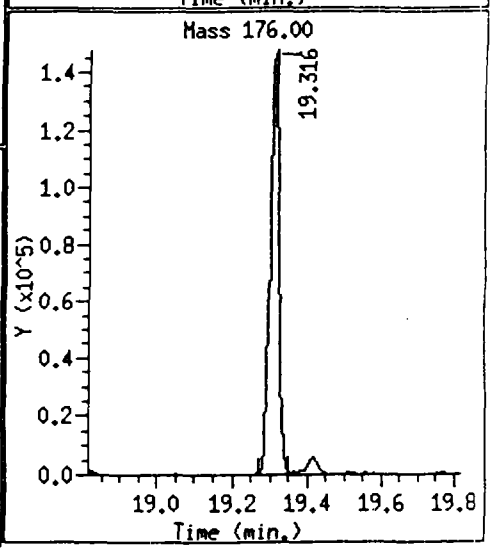












Data File: /chem/V.i/06-09-98.b/VH287.d

Date : 09-JUN-1998 13:55

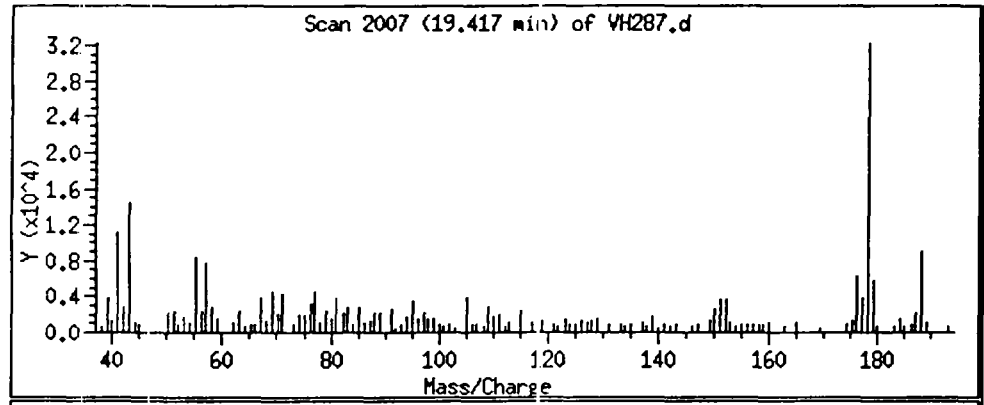
Instrument : V.i

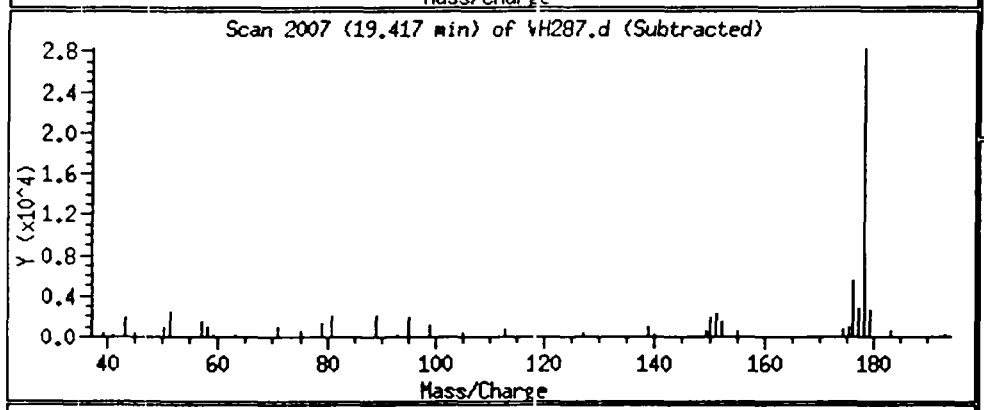
Sample ID : SITE#2 5/29/98 24HR

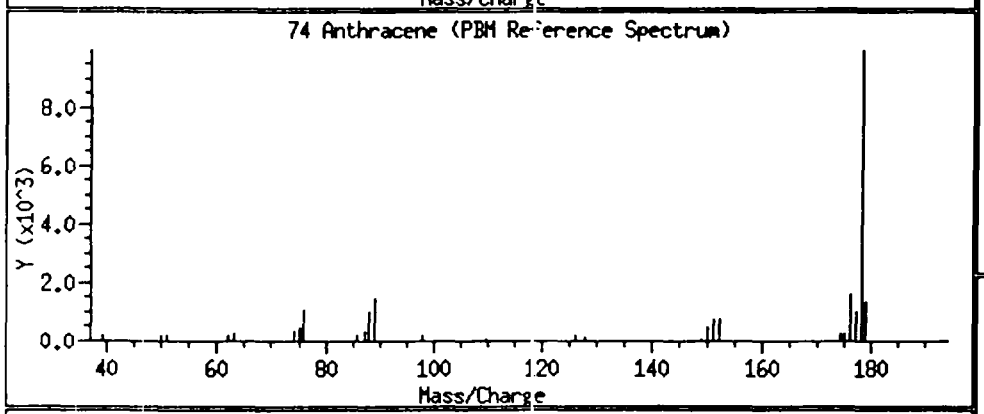
Column phase : DB-5 Column diameter : 0.32

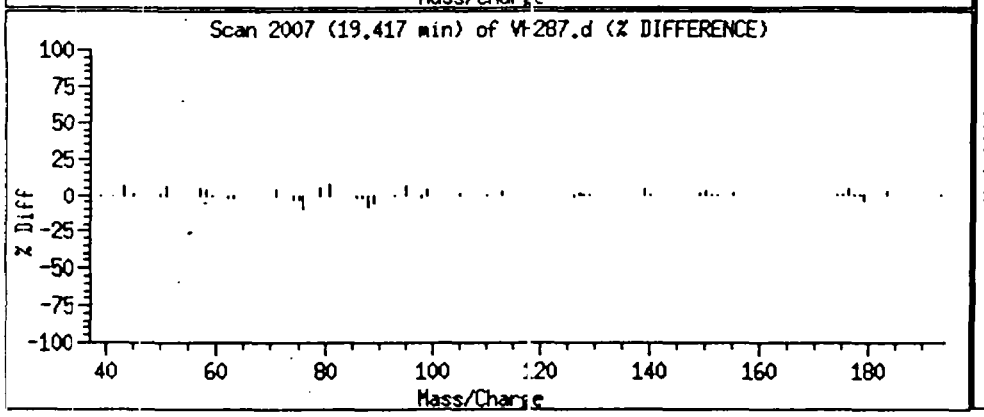
Volume Injected (uL) : 1.0

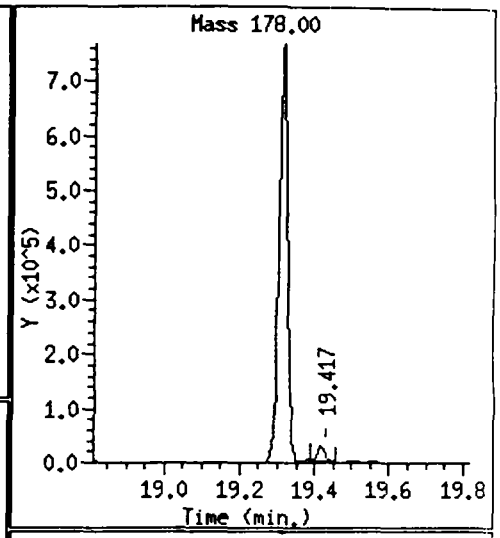
74 Anthracene

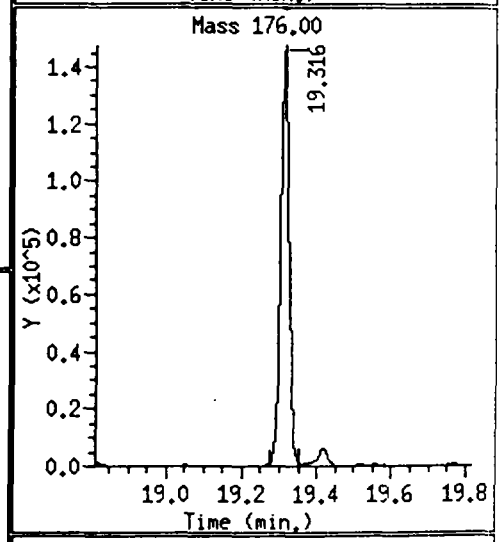


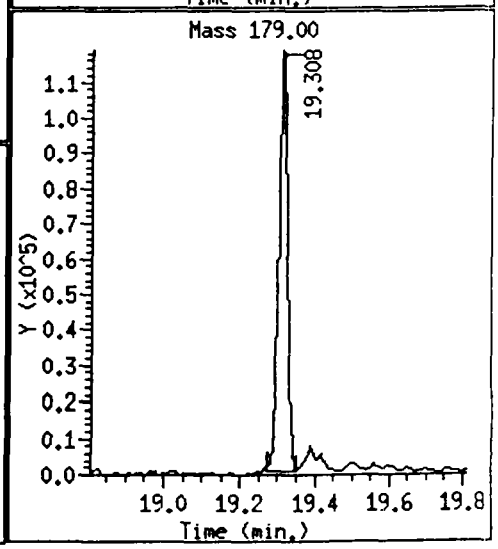












Data File: /chem/V.i/06-09-98.b/VH287.d

Date : 09-JUN-1998 13:55

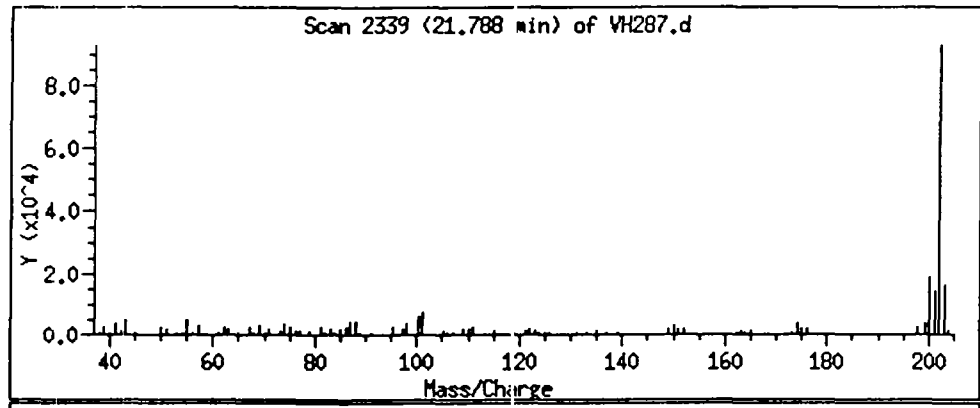
Instrument : V.i

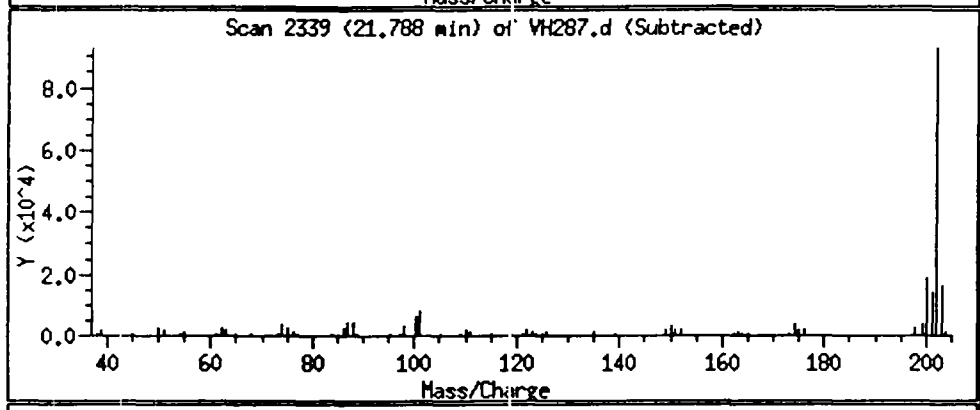
Sample ID : SITE#2 5/29/98 24HR

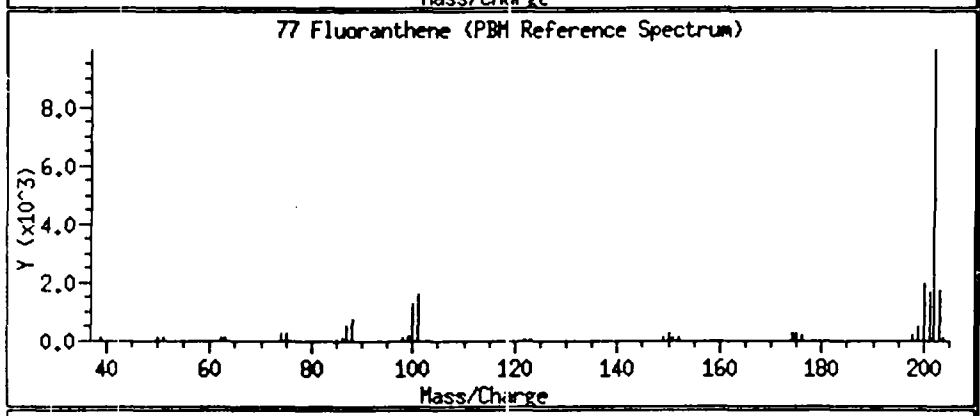
Column phase : DB-5 Column diameter : 0.32

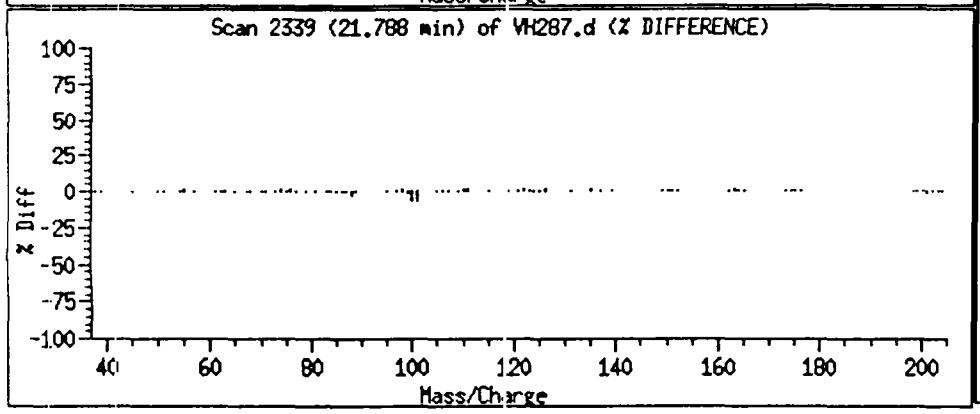
Volume Injected (uL) : 1.0

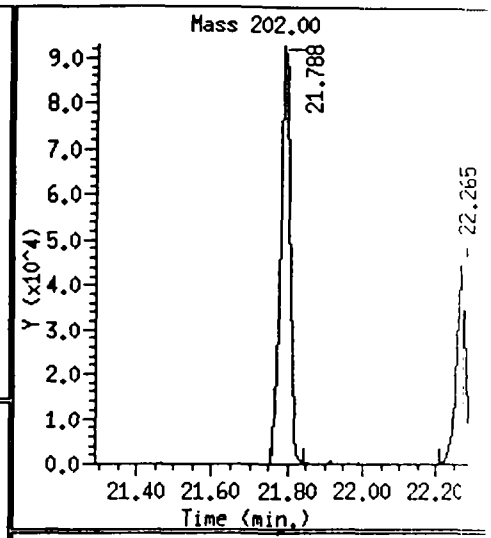
77 Fluoranthene

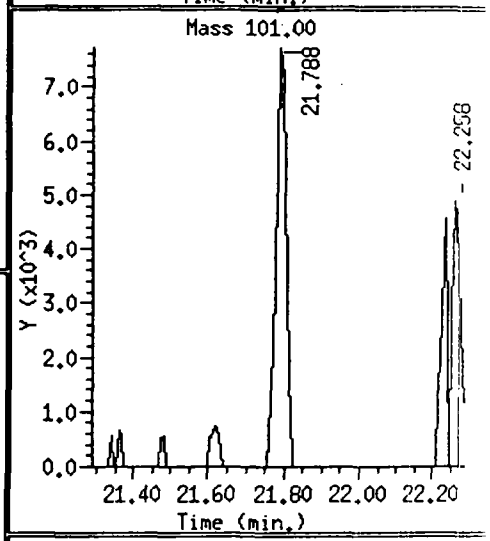


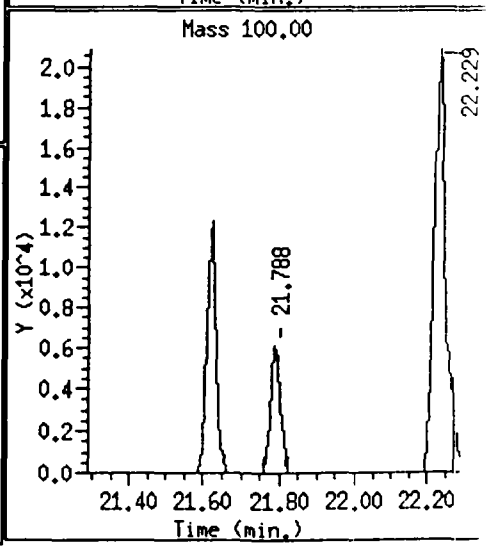












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH279

Method 8270A PUF
Sample ID: SITE#3 5-27-98 24hour

Client Project: Hull & Assoc
TLI ID: 209-67-11,16

Date Received: 06/02/98
Date Extracted: 06/04/98
Date Analyzed : 06/08/98

Response File: VH267

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	14.36	B	12.56		10
Acenaphthene-d_{10}		IS 3	16.16		
Fluorene	7.16	J	17.30		10
Phenanthrene-d_{10}		IS 4	19.27		
Phenanthrene	24.79	B	19.32		10
Anthracene	0.68	J	19.42		10
Fluoranthene	3.26	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.02	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	97.08	19.39	4	97
Pyrene-d_{10}	122.04	22.24	5	122
Terphenyl-d_{14}	110.08	22.61	5	110

Reviewed by GAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone (919) 544-5729 • Fax: (919) 544-5491

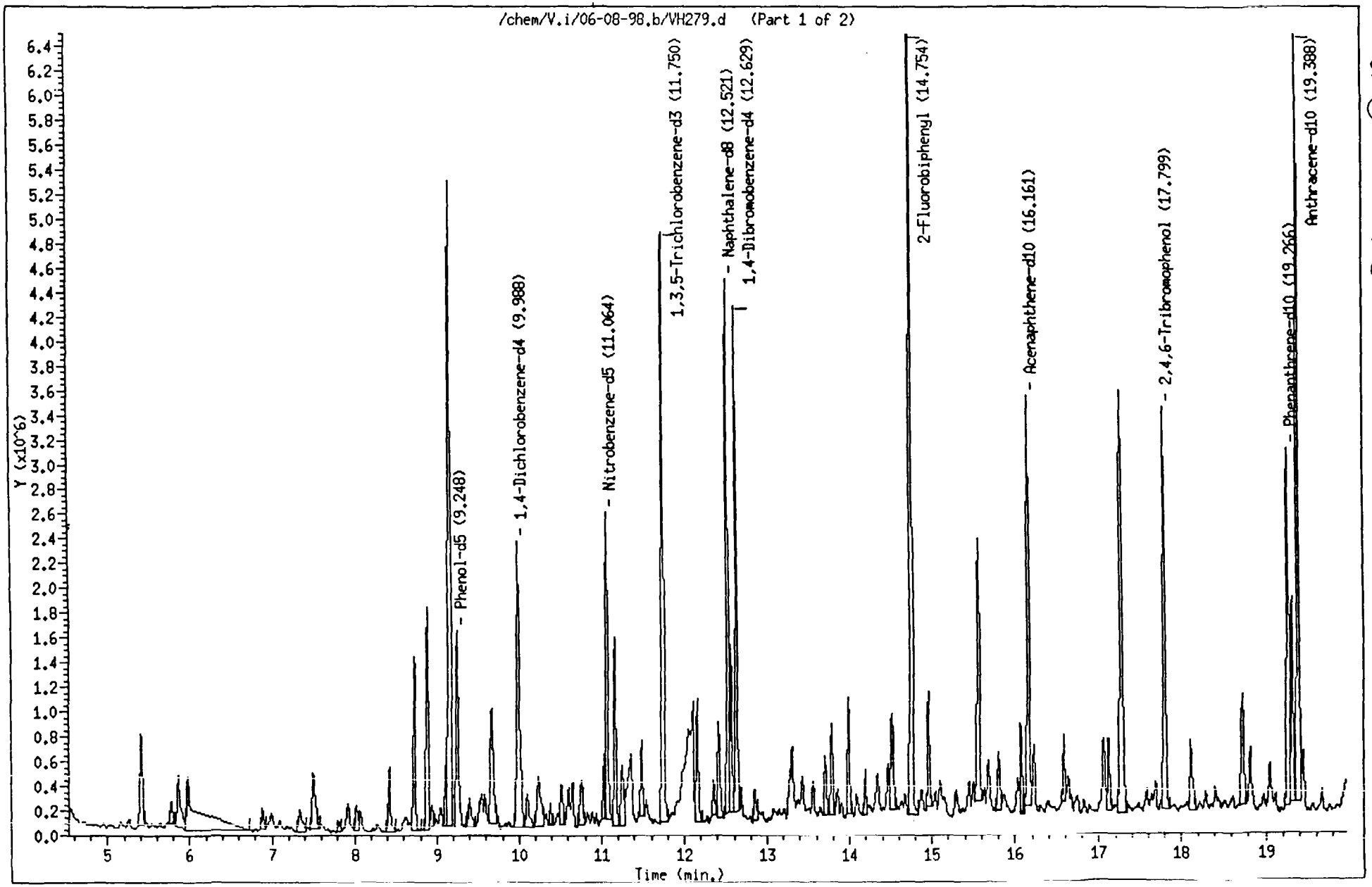
Savar v3.7
Printed: 15:15 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH279.d
Date : 08-JUN-1998 23:58
Instrument : V.i
Sample ID : SITE#3 5-27-98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

108



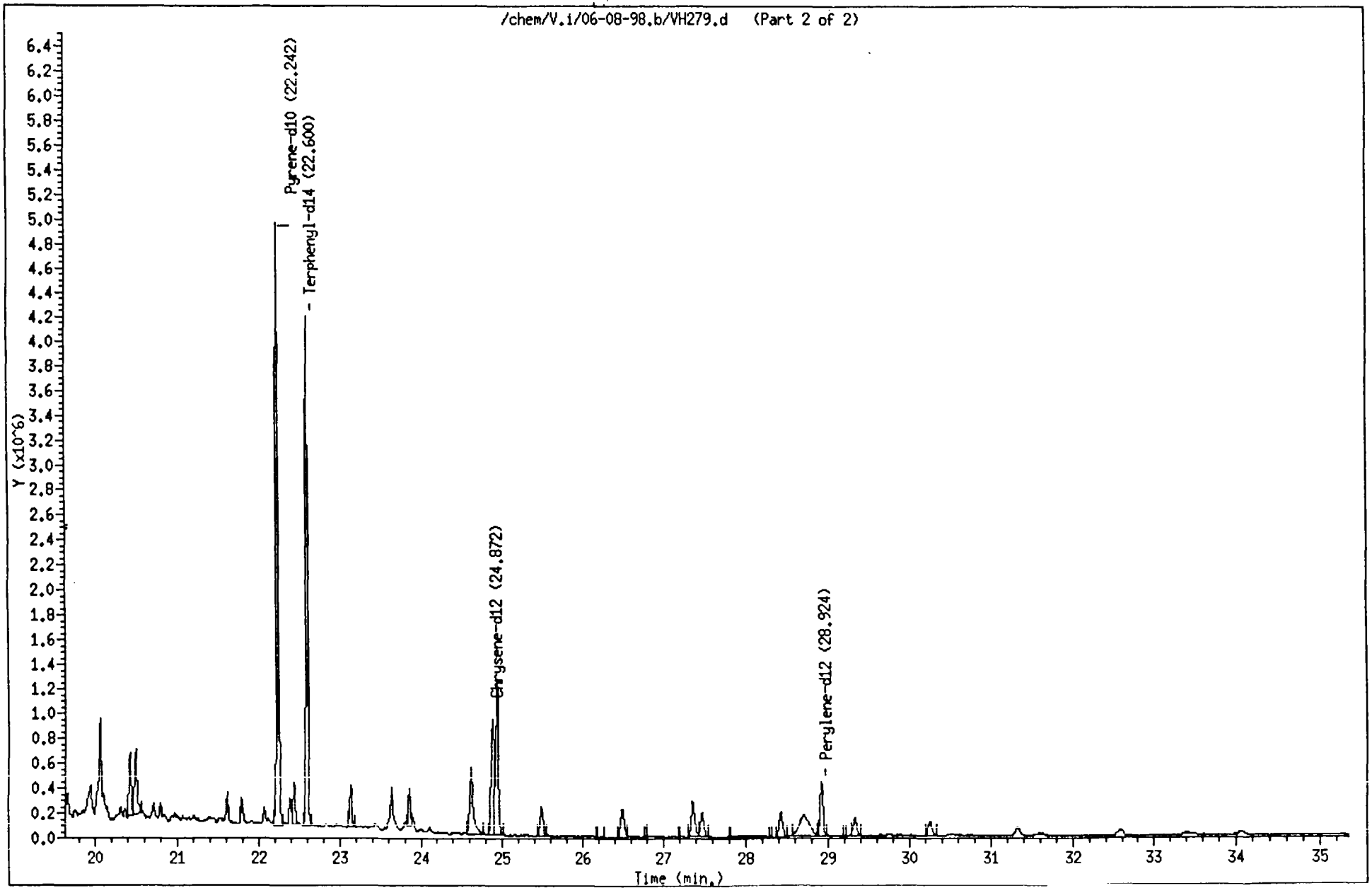
Data Review: [illegible]
Date: 6/12/98

Data File: /chem/V.i/06-08-98.b/VH279.d
Date : 08-JUN-1998 23:58
Instrument : V.i
Sample ID : SITE#3 5-27-98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

109



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH279.d
Lab. Id. : 45869
Inj Date : 08-JUN-1998 23:58 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#3 5-27-98 24HOUR
Misc Info : 209-67-11,16
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 12
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.988(1.000)	582342	40.00	
*	2 Naphthalene-d8	136.00	12.521(1.000)	2214122	40.00	
*	3 Acenaphthene-d10	164.00	16.161(1.000)	1153137	40.00	
*	4 Phenanthrene-d10	188.00	19.266(1.000)	1850491	40.00	
*	5 Chrysene-d12	240.00	24.872(1.000)	563241	40.00	
*	6 Perylene-d12	264.00	28.924(1.000)	397313	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.248(0.926)	613306	34.88	34.88
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.064(0.884)	1199209	63.76	63.76
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.750(0.938)	1354563	70.87	70.87
$	13 1,4-Dibromobenzene-d4	240.00	12.629(1.264)	1098327	83.76	83.76
$	14 2-Fluorobiphenyl	172.00	14.761(0.913)	2970334	89.73	89.73
$	15 2,4,6-Tribromophenol	329.80	17.799(1.101)	506077	76.43	76.43
$	16 Anthracene-d10	188.00	19.388(1.006)	3691890	97.09	97.09
$	17 Pyrene-d10	212.00	22.242(0.894)	3141212	122.02	122.02(R)
$	18 Terpheryl-d14	244.00	22.607(0.909)	1777922	110.11	110.11
	42 Naphthalene	128.00	12.565(1.003)	764692	14.37	14.37
	46 2-Methylnaphthalene	142.00	13.988(1.117)	358499	10.90	10.90
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	152.00	~~16.132(0.998)~~	~~12735~~	~~0.26~~	~~0.26(a)~~ NA PAB
	58 Acenaphthene	154.00	16.218(1.004)	136019	4.92	4.92(a)
	65 Fluorene	166.00	17.296(1.070)	262992	7.16	7.16(a)
	73 Phenanthrene	178.00	19.316(1.003)	1051712	24.80	24.80
	74 Anthracene	178.00	19.42 ~~19.316(1.003)~~	~~1051712~~ 26917	~~26.58~~	~~26.58~~ M PAB
	77 Fluoranthene	202.00	21.792(1.131)	141008	3.26	3.26(a)
	78 Pyrene	202.00	~~21.792(0.876)~~	~~118642~~	~~3.63~~	~~3.63(a)~~ NA PAB
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).
R - Spike/Surrogate failed recovery limits.

Data File: /chem/V.i/06-08-98.b/VH279.d

Date : 08-JUN-1998 23:58

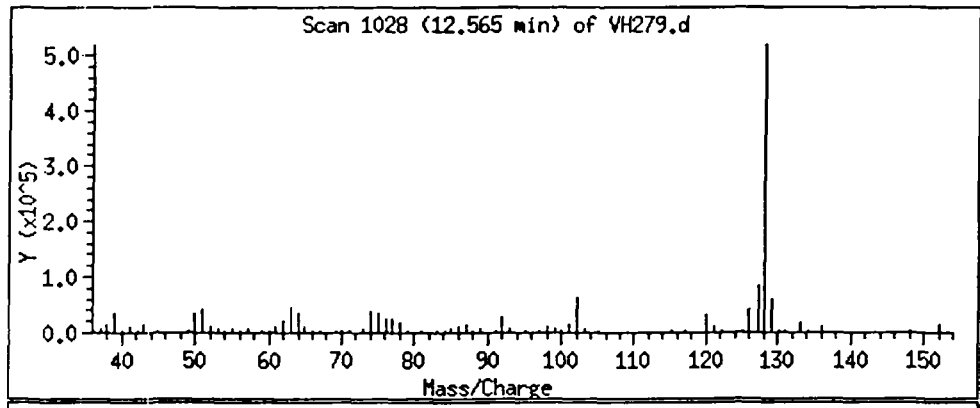
Instrument : V.i

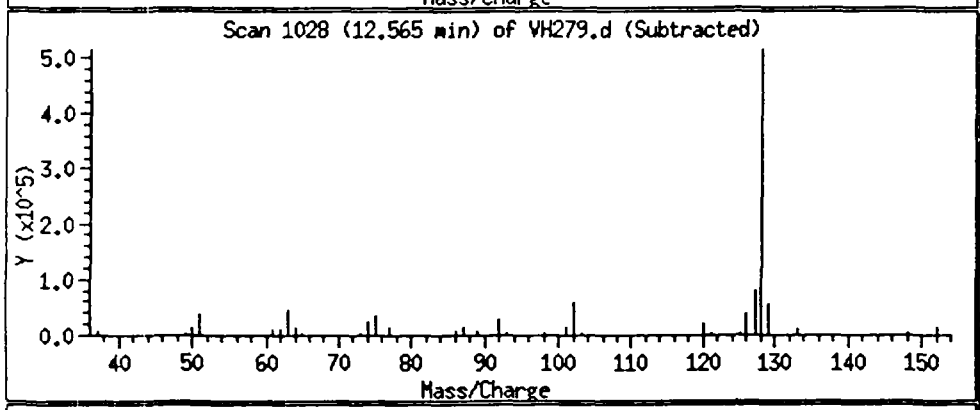
Sample ID : SITE#3 5-27-98 24HR

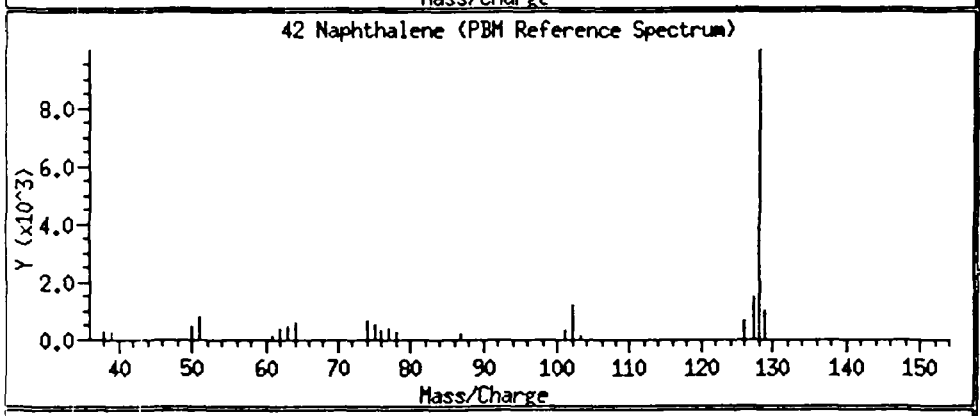
Column phase : DB-5 Column diameter : 0.32

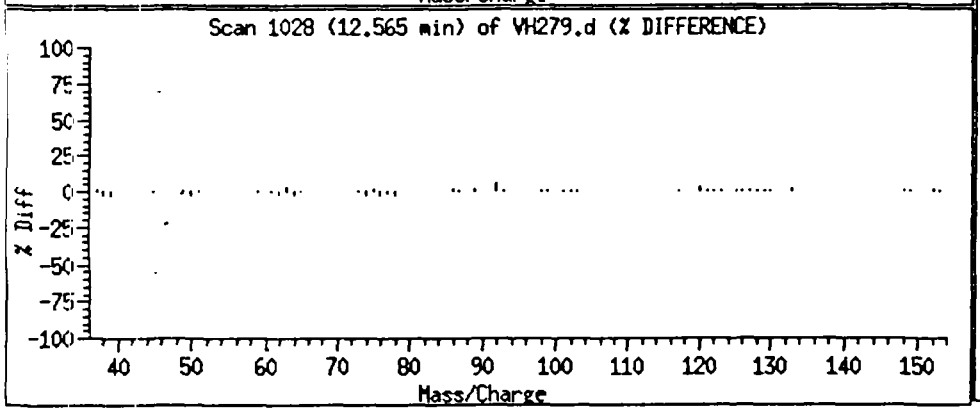
Volume Injected (uL) : 1.0

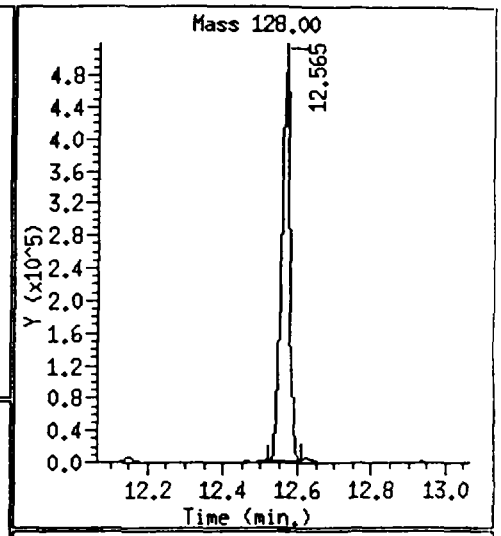
42 Naphthalene

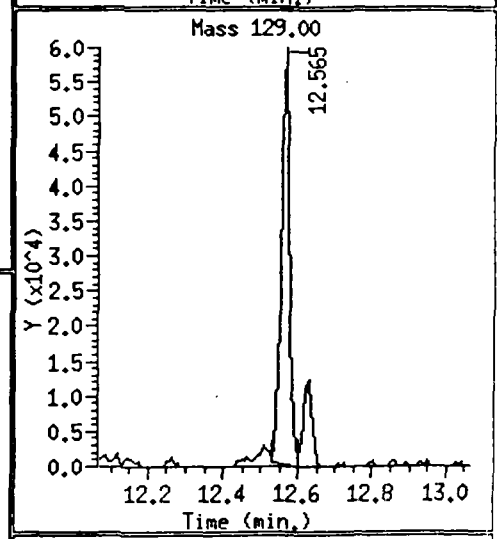


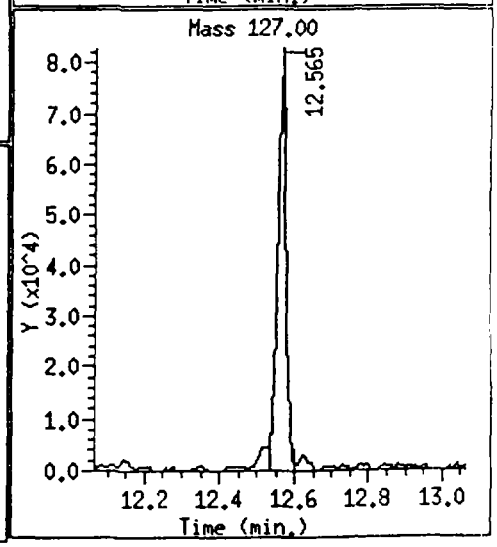












Data File: /chem/V.i/06-08-98.b/VH279.d

Date : 08-JUN-1998 23:58

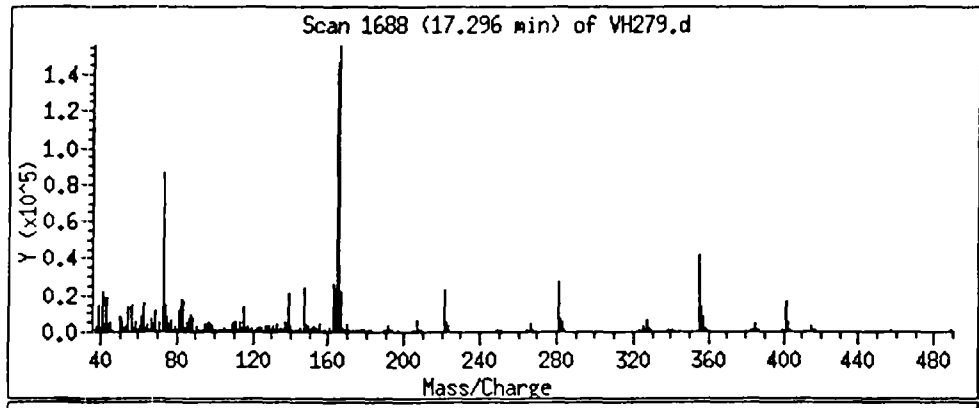
Instrument : V.i

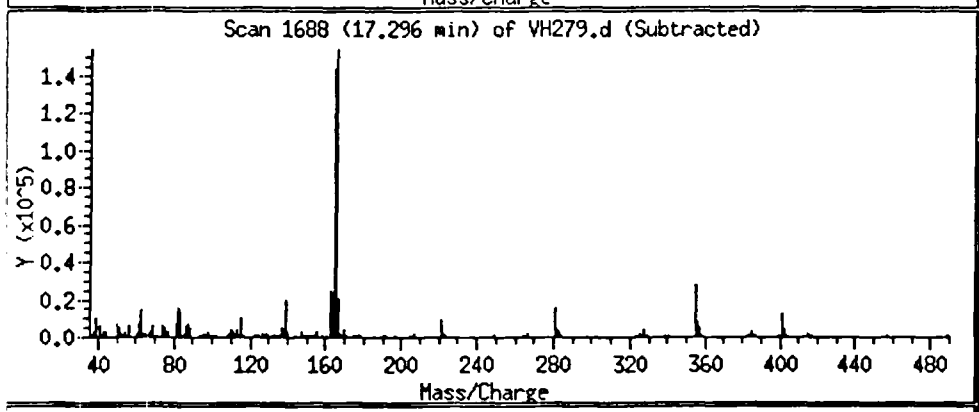
Sample ID : SITE#3 5-27-98 24HR

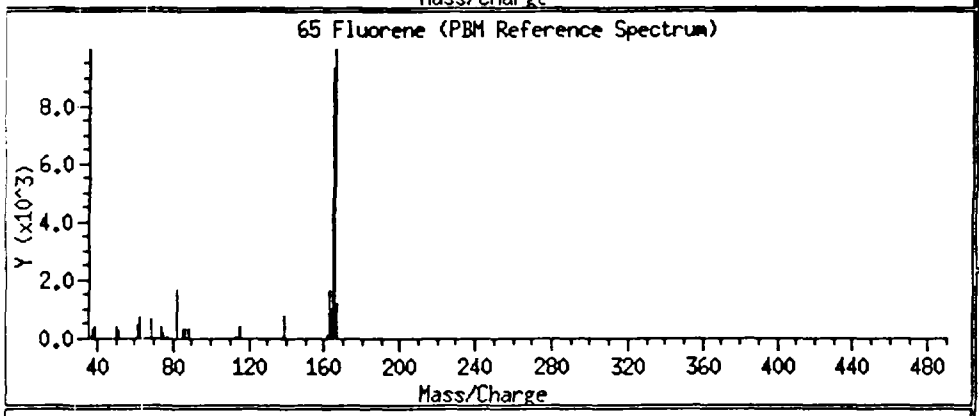
Column phase : DB-5 Column diameter : 0.32

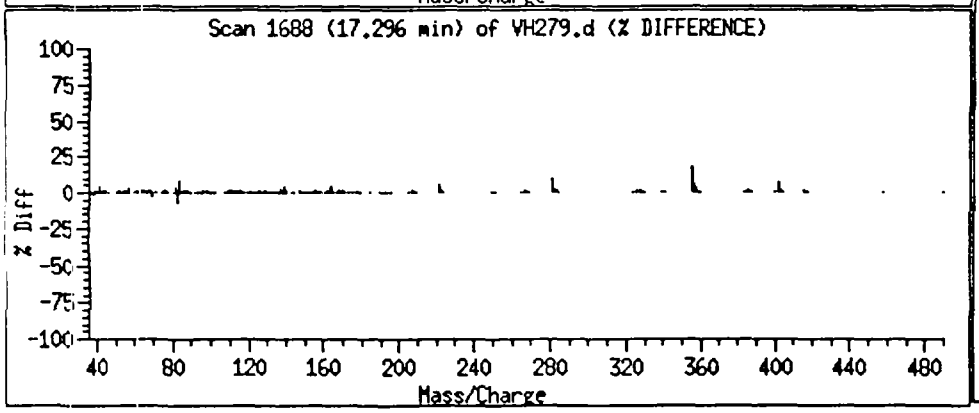
Volume Injected (uL) : 1.0

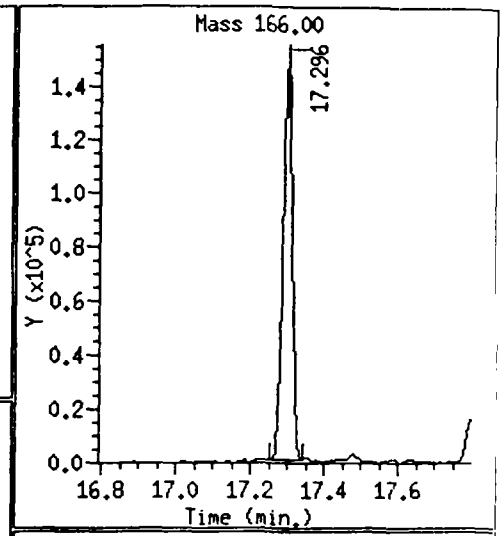
65 Fluorene

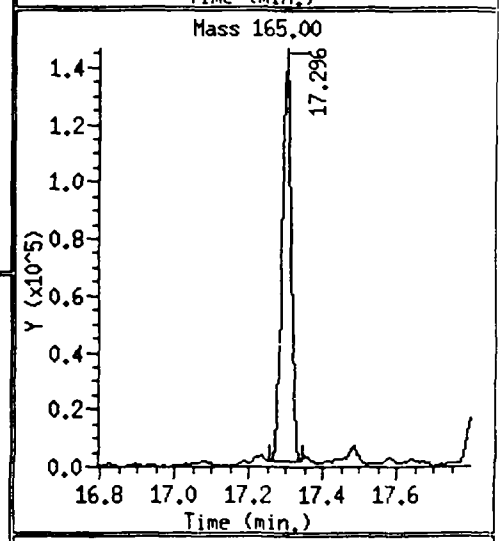


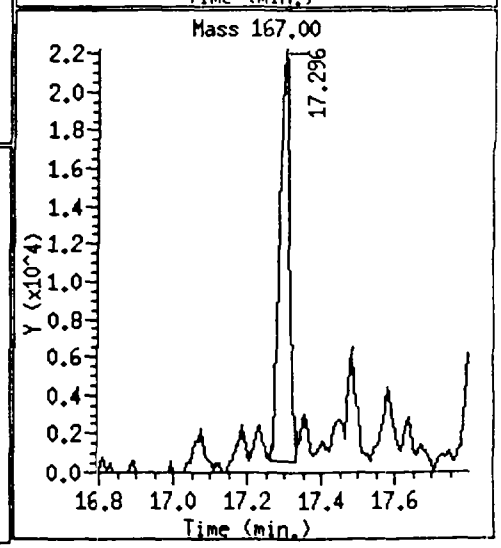












Data File: /chem/V.i/06-08-98.b/VH279.d

Date : 03-JUN-1998 23:58

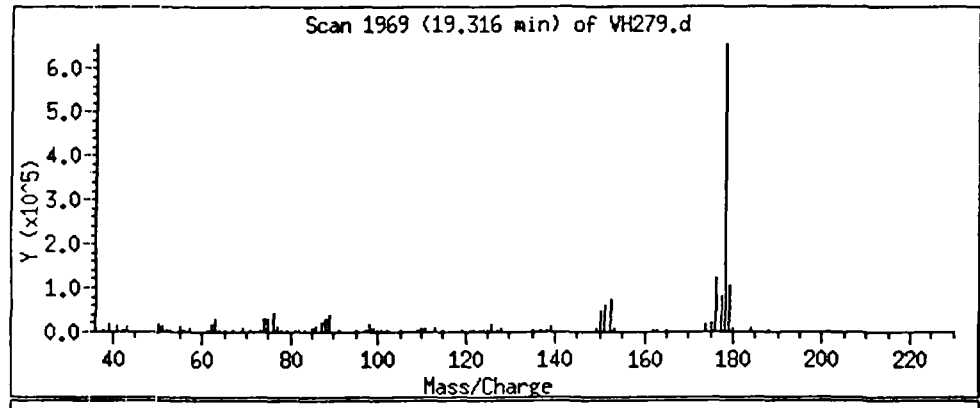
Instrument : V.i

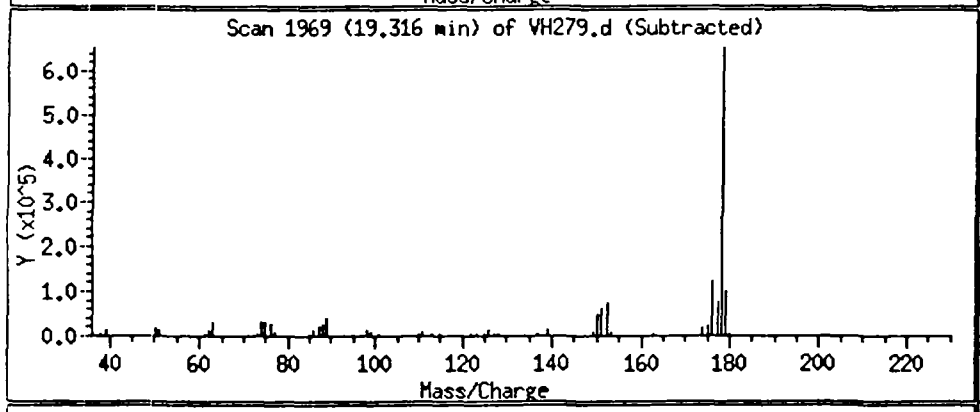
Sample ID : SITE#3 5-27-98 24HR

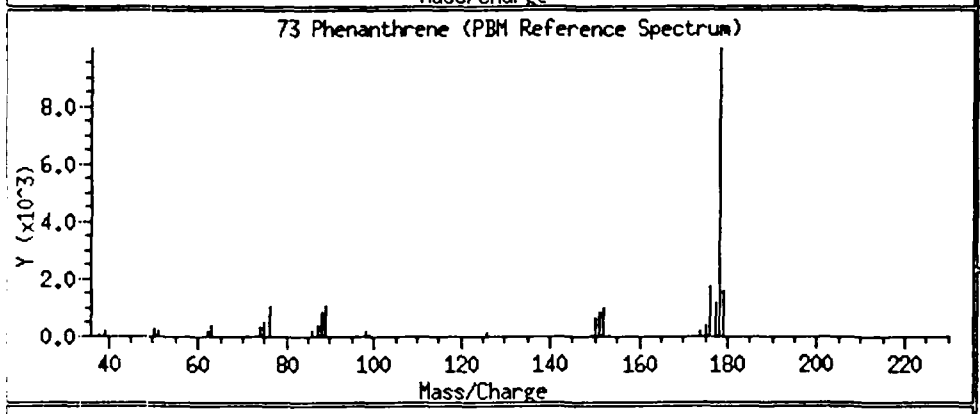
Column phase : DB-5 Column diameter : 0.32

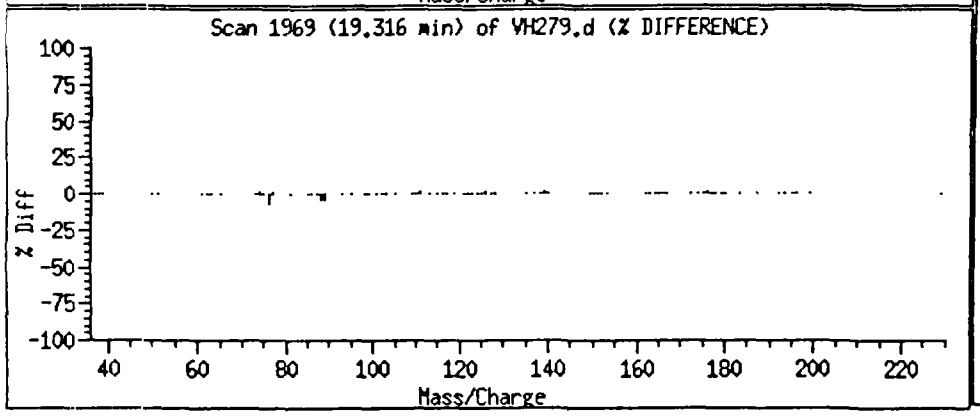
Volume Injected (uL) : 1.0

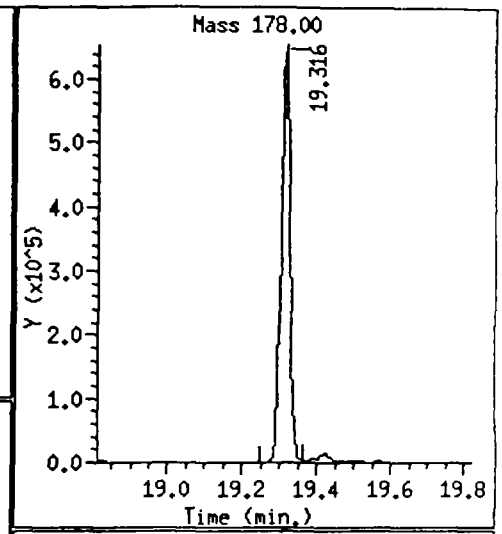
73 Phenanthrene

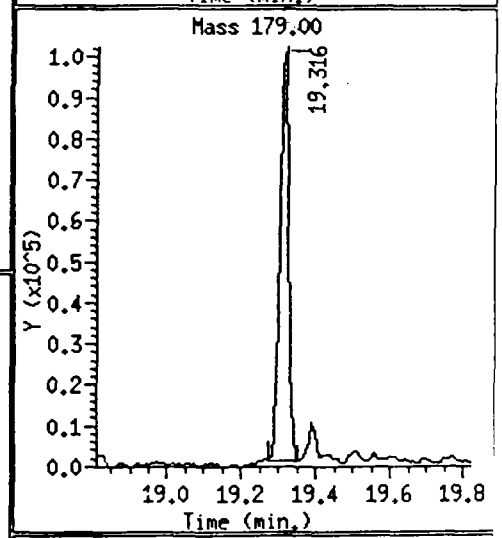


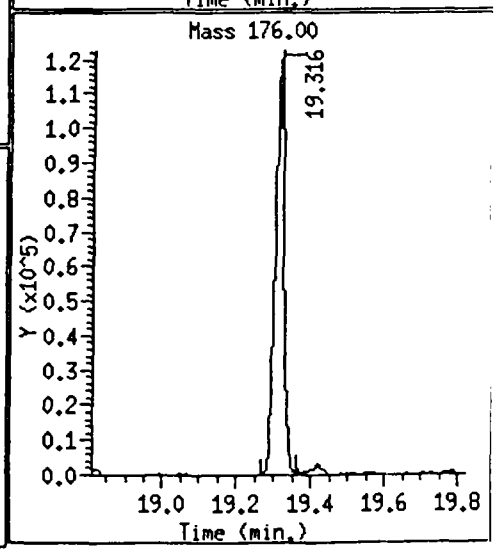












Data File: /chem/V.i/06-08-98.b/VH279.d

Date : 08-JUN-1998 23:58

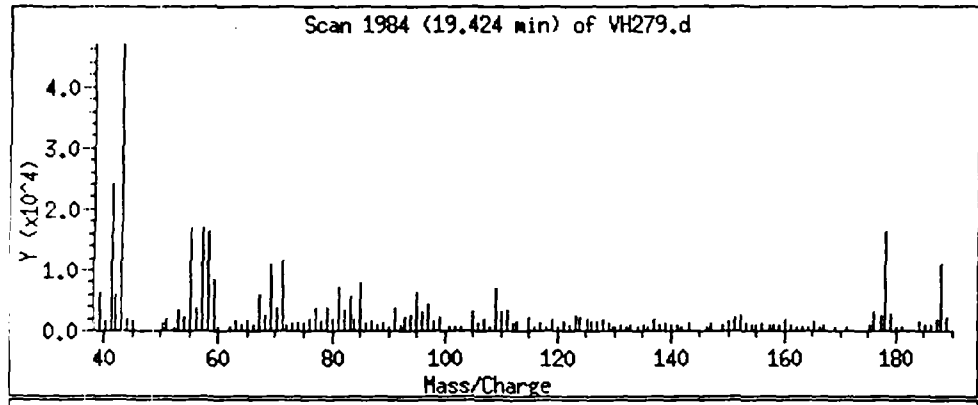
Instrument : V.i

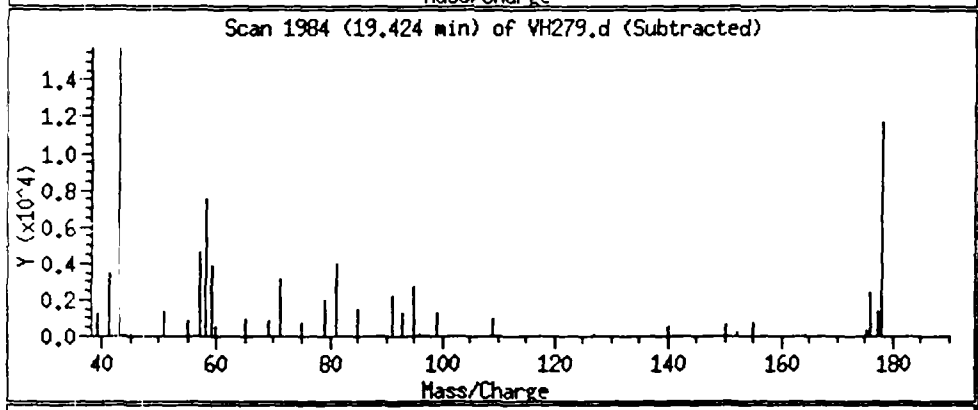
Sample ID : SITE#3 5-27-98 24HR

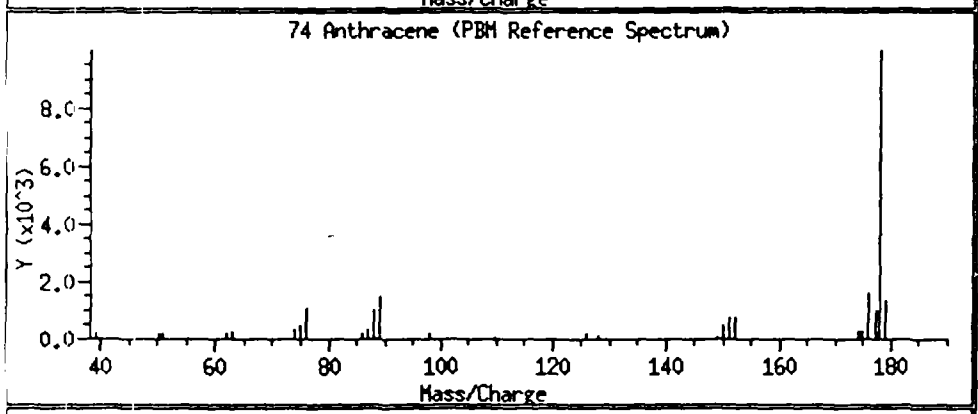
Column phase : DB-5 Column diameter : 0.32

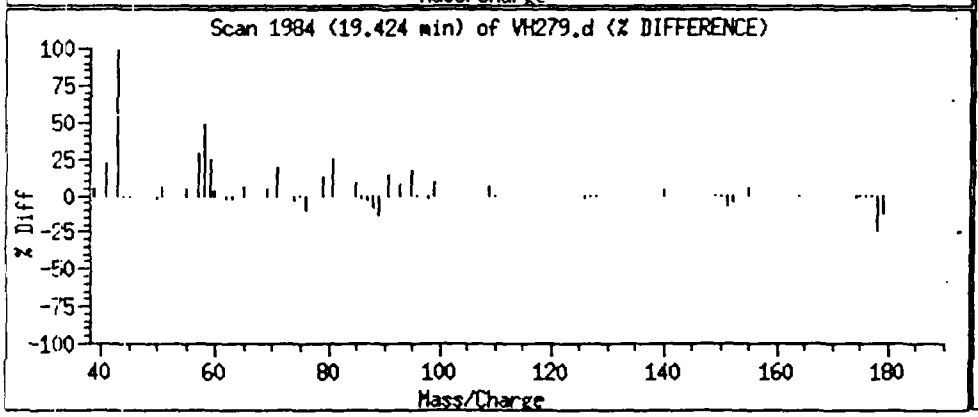
Volume Injected (uL) : 1.0

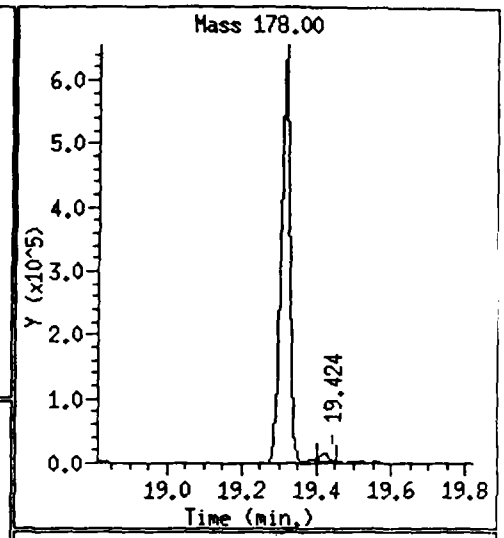
74 Anthracene

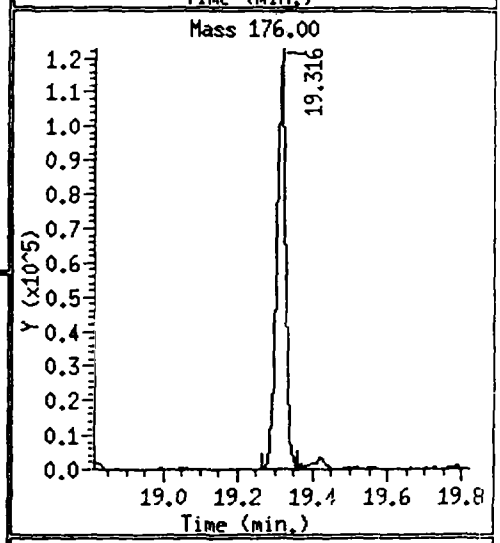


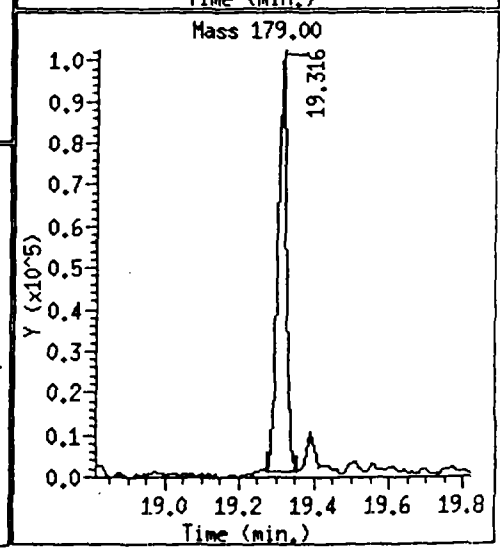












Data File: /chem/V.i/06-08-98.b/VH279.d

Date : 08-JUN-1998 23:58

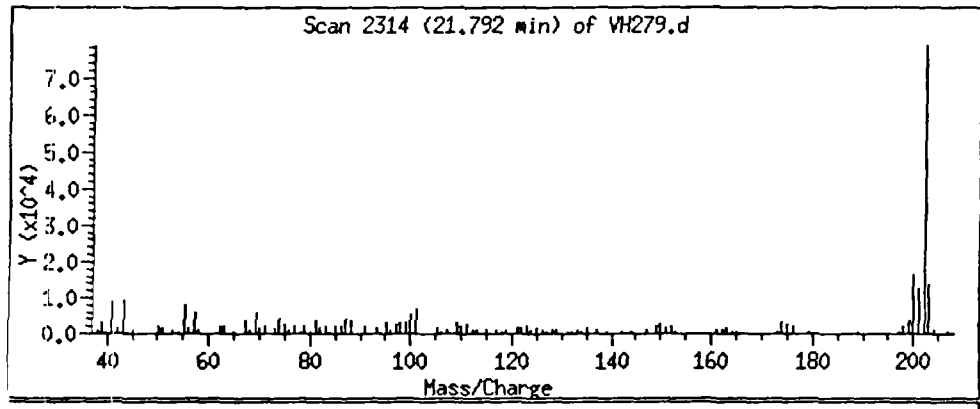
Instrument : V.i

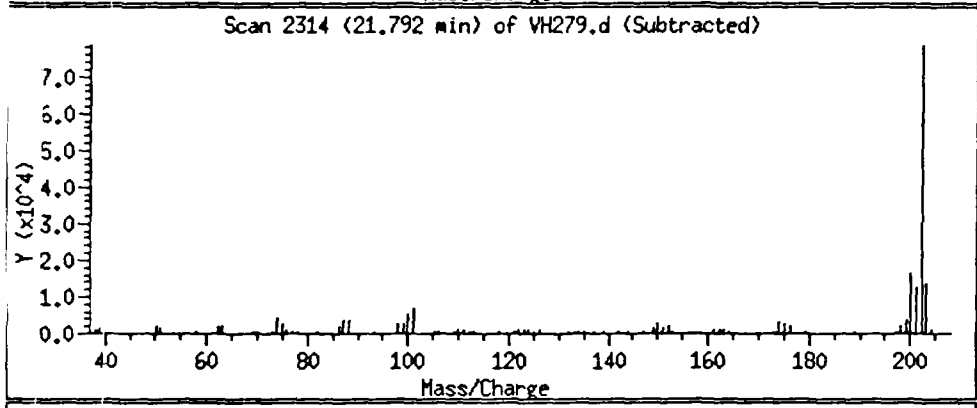
Sample ID : SITE#3 5-27-98 24HR

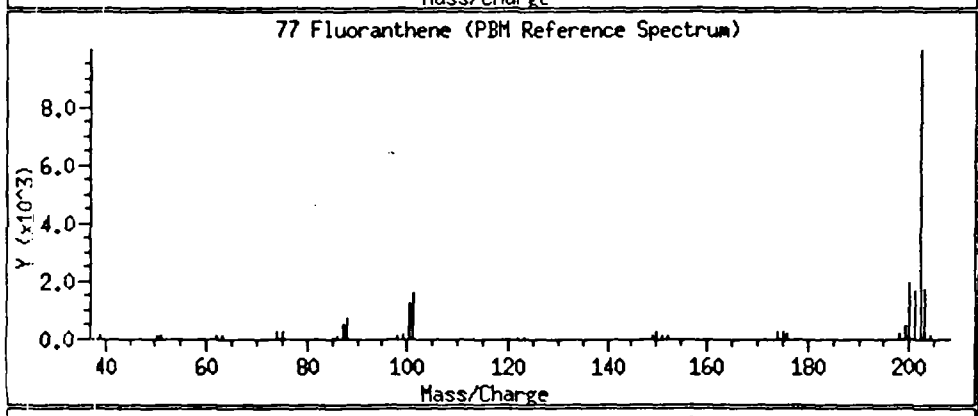
Column phase : DB-5 Column diameter : 0.32

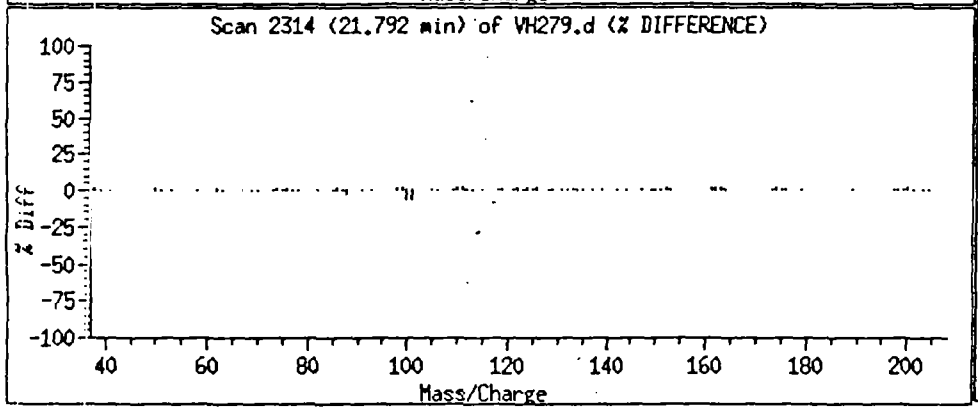
Volume Injected (uL) : 1.0

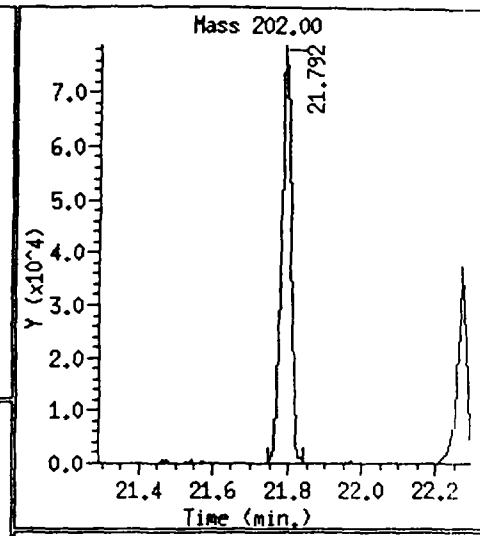
77 Fluoranthene

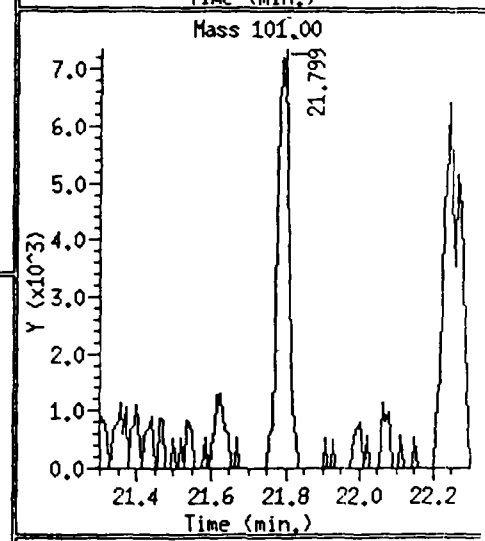


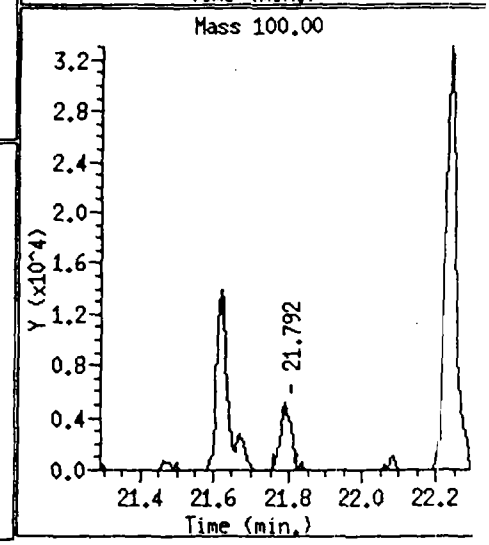












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH275

Method 8270A PUF
Sample ID: SITE#3 5-28-98 8hour

Client Project: Hull & Assoc
TLI ID: 209-67-3,7

Date Received: 06/02/98
Date Extracted: 06/04/98
Date Analyzed : 06/08/98

Response File: VH267

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	2.68	BJ	12.56		10
Acenaphthene-d_{10}		IS 3	16.15		
Fluorene	1.54	J	17.29		10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene	4.85	BJ	19.31		10
Anthracene	0.25	J	19.41		10
Fluoranthene	1.01	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		0.97	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	88.80	19.38	4	89
Pyrene-d_{10}	109.52	22.24	5	110
Terphenyl-d_{14}	98.91	22.60	5	99

Reviewed by PWB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit
IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

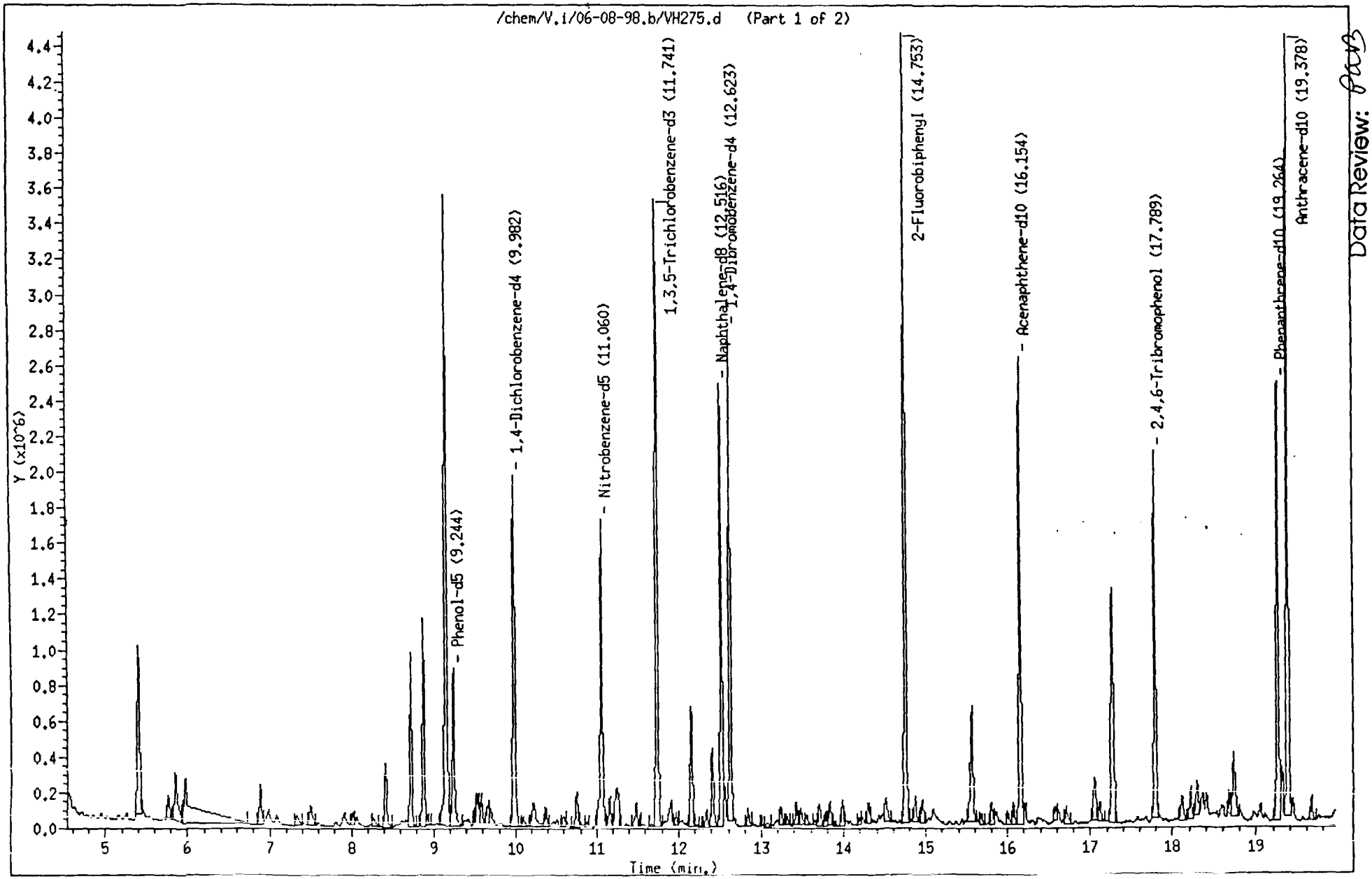
Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

Savar v3.7
Printed: 15:15 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH275.d
Date : 08-JUN-1998 20:43
Instrument : V.i
Sample ID : SITE#3 5-28-98 8HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



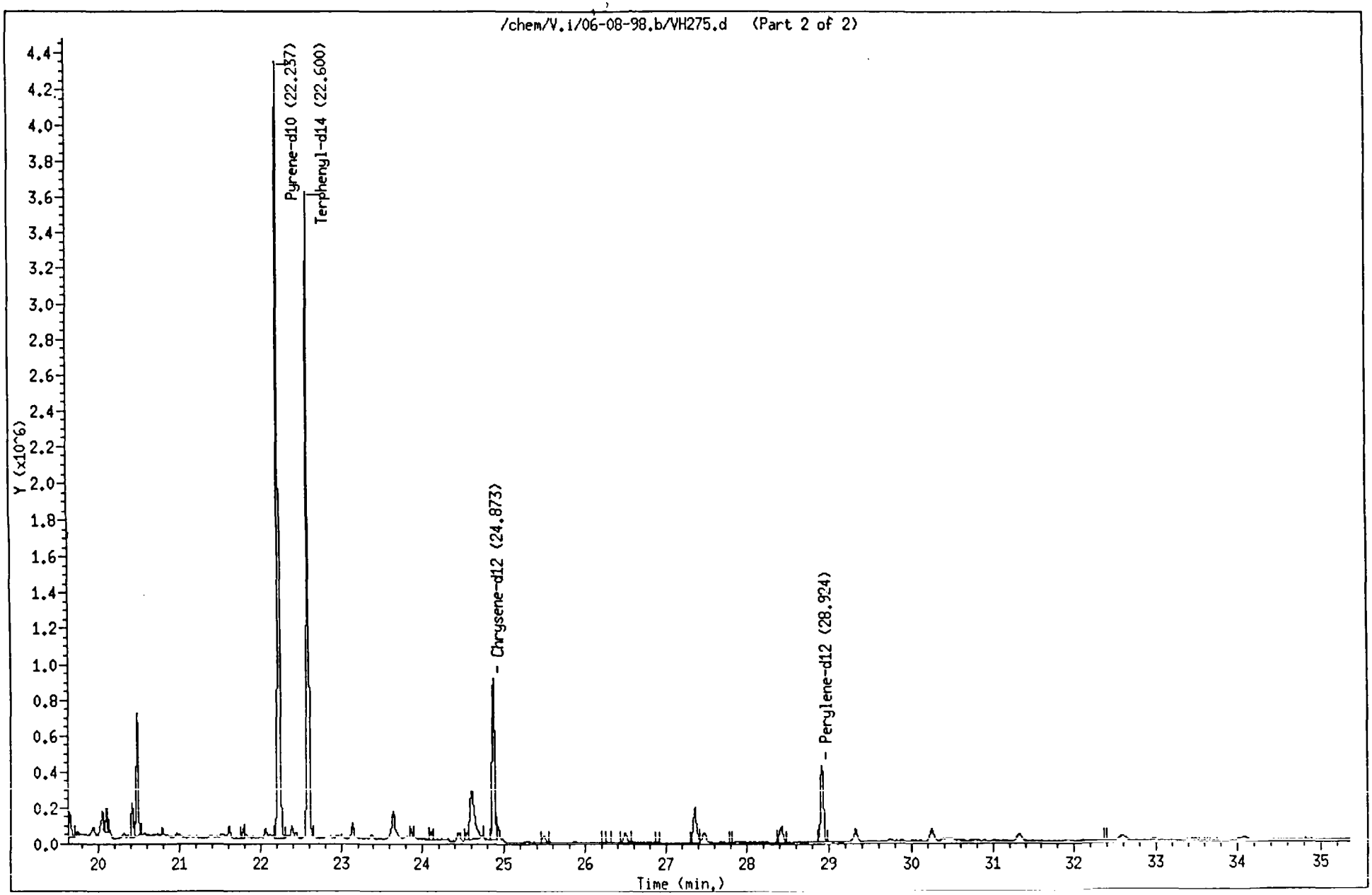
Data Review: [illegible]
Date: 6/12/98

119

Data File: /chem/V.i/06-08-98.b/VH275.d
Date : 08-JUN-1998 20:43
Instrument : V.i
Sample ID : SITE#3 5-28-98 8HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH275.d
Lab. Id. : 45869
Inj Date : 08-JUN-1998 20:43 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#3 5-28-98 8HOUR
Misc Info : 209-67-3,7
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 8
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.982(1.000)	480999	40.00	
*	2 Naphthalene-d8	136.00	12.516(1.000)	1745412	40.00	
*	3 Acenaphthene-d10	164.00	16.154(1.000)	940029	40.00	
*	4 Phenanthrene-d10	188.00	19.264(1.000)	1570389	40.00	
*	5 Chrysene-d12	240.00	24.873(1.000)	542571	40.00	
*	6 Perylene-d12	264.00	28.924(1.000)	417327	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.244(0.926)	398432	27.44	27.44
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.060(0.884)	811181	54.71	54.71
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.741(0.938)	966972	64.17	64.17
$	13 1,4-Dibromobenzene-d4	240.00	12.623(1.265)	774720	71.53	71.53
$	14 2-Fluorobiphenyl	172.00	14.753(0.913)	1977912	73.29	73.29
$	15 2,4,6-Tribromophenol	329.80	17.797(1.102)	334895	62.04	62.04
$	16 Anthracene-d10	188.00	19.378(1.006)	2865585	88.80	88.80
$	17 Pyrene-d10	212.00	22.237(0.894)	2715627	109.50	109.50
$	18 Terphenyl-d14	244.00	22.600(0.909)	1538869	98.93	98.93
	42 Naphthalene	128.00	12.558(1.003)	112396	2.68	2.68(a)
	46 2-Methylnaphthalene	142.00	13.985(1.117)	45421	1.75	1.75(a)
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	154.00	16.218(1.004)	25220	1.12	1.12(a)
	65 Fluorene	166.00	17.292(1.070)	46170	1.54	1.54(a)
	73 Phenanthrene	178.00	19.307(1.002)	174538	4.85	4.85(a)
	74 Anthracene	178.00	19.41 ~~19.307(1.002)~~	~~174538~~ 8448	~~5.20~~	~~5.20(a)~~ (M) PAB
	77 Fluoranthene	~~Compound Not Detected.~~ 21.789			37244	(M) PAB
	78 Pyrene	Compound Not Detected.				
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-08-98.b/VH275.d

Date : 08-JUN-1998 20:43

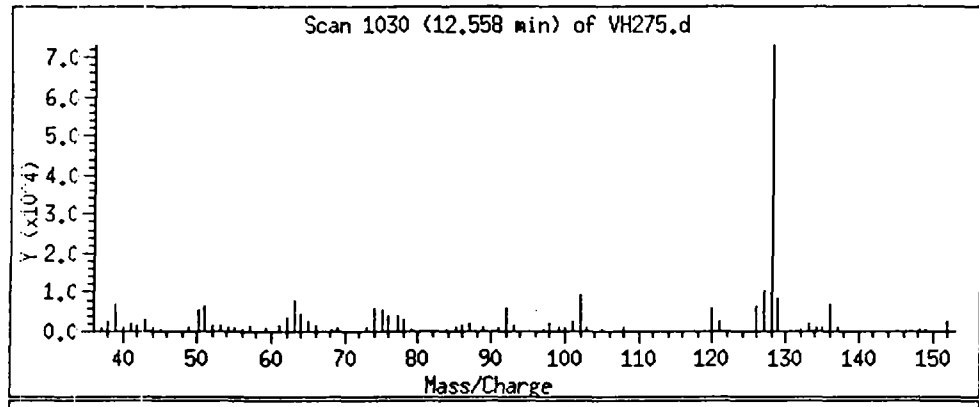
Instrument : V.i

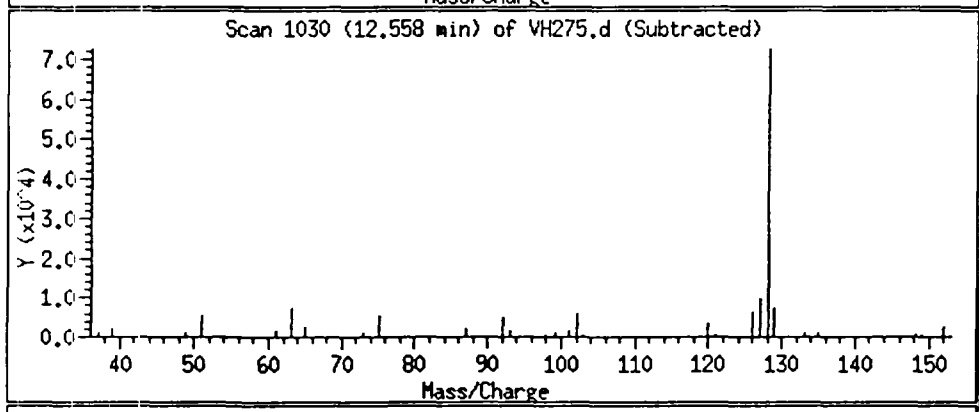
Sample ID : SITE#3 5-28-98 8HR

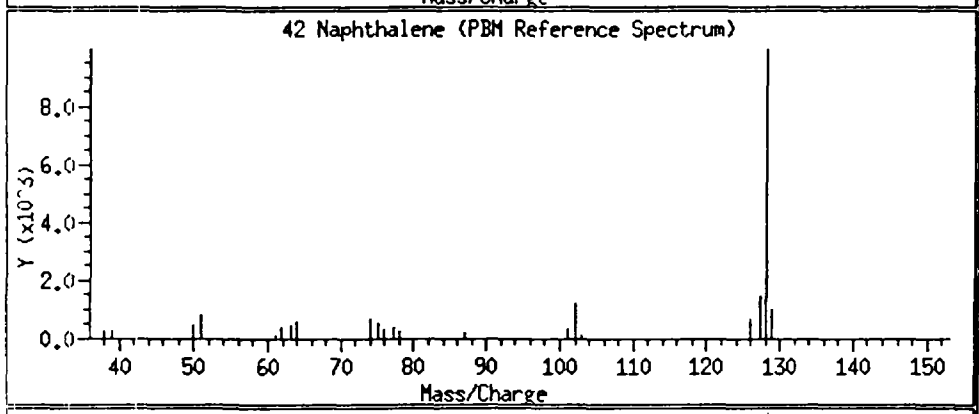
Column phase : DB-5 Column diameter : 0.32

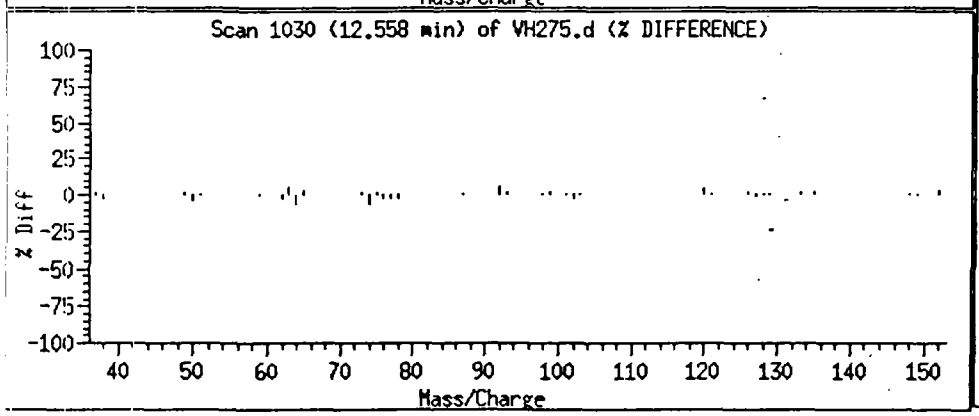
Volume Injected (uL) : 1.0

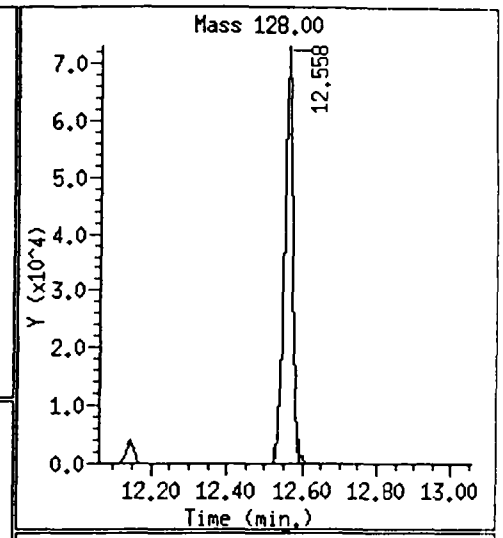
42 Naphthalene

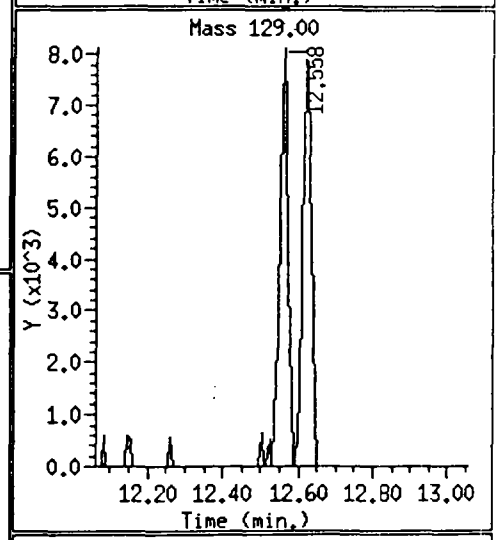


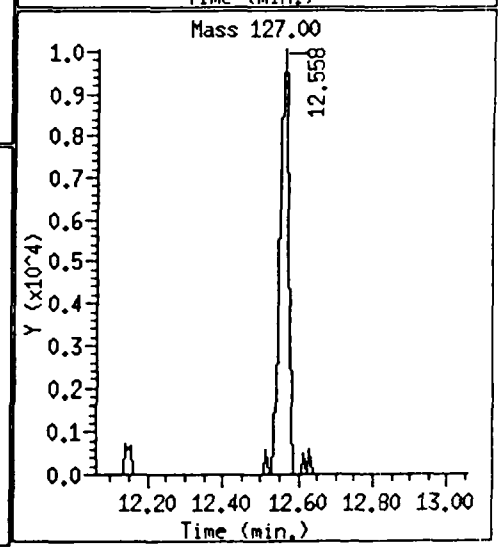












Data File: /chem/V.i/06-08-98.b/VH275.d

Date : 08-JUN-1998 20:43

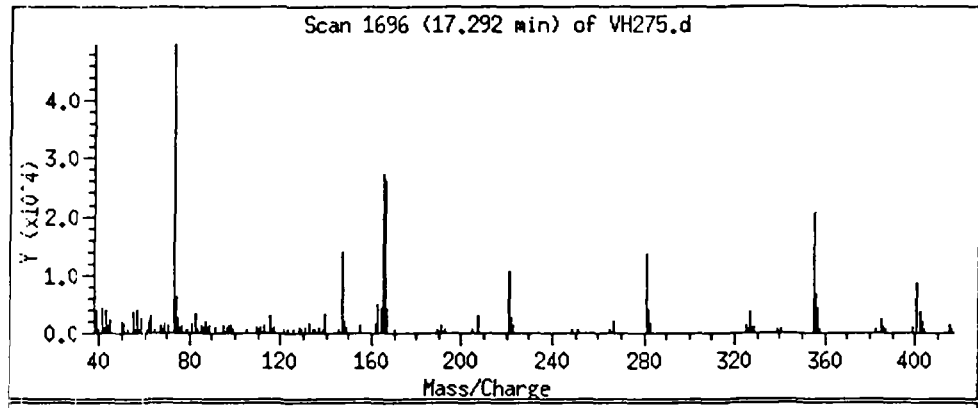
Instrument : V.i

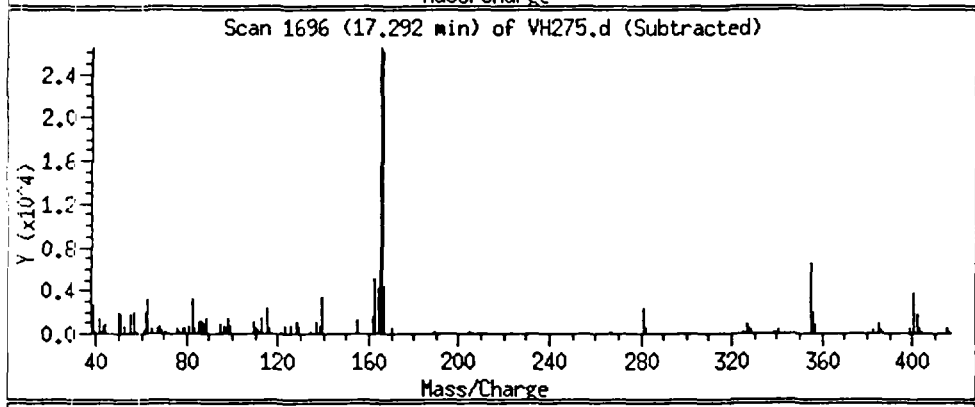
Sample ID : SITE#3 5-28-98 8HR

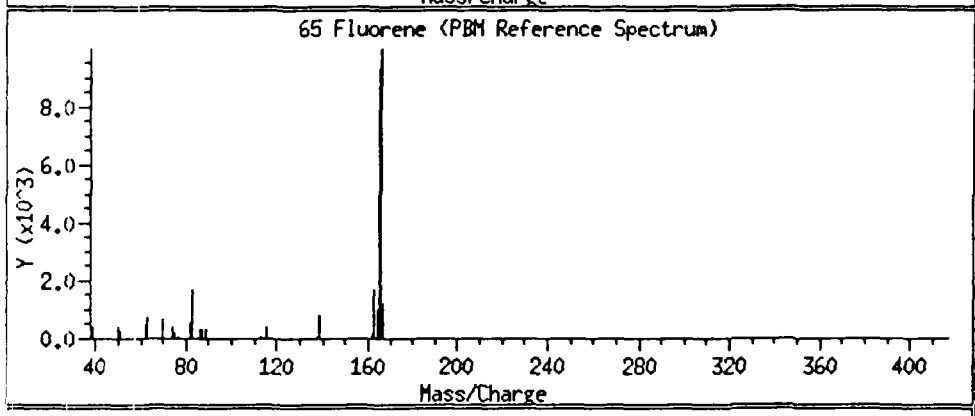
Column phase : DB-5 Column diameter : 0.32

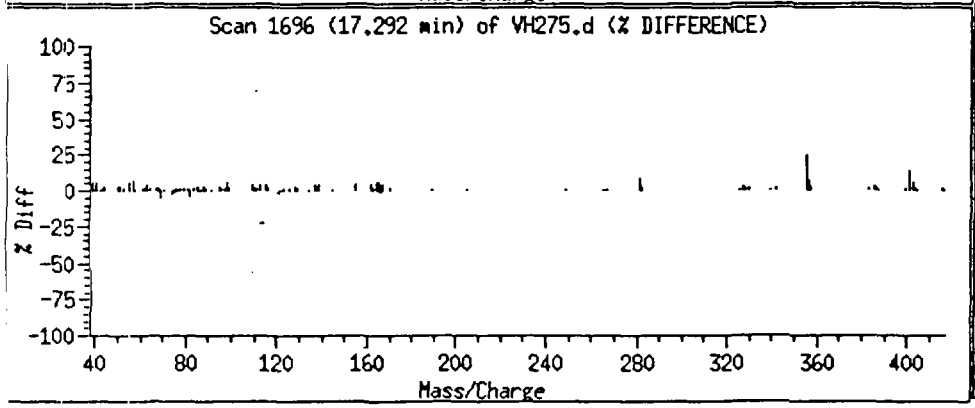
Volume Injected (uL) : 1.0

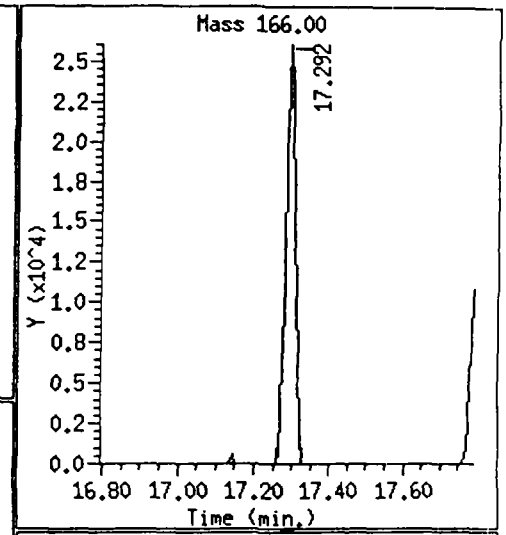
65 Fluorene

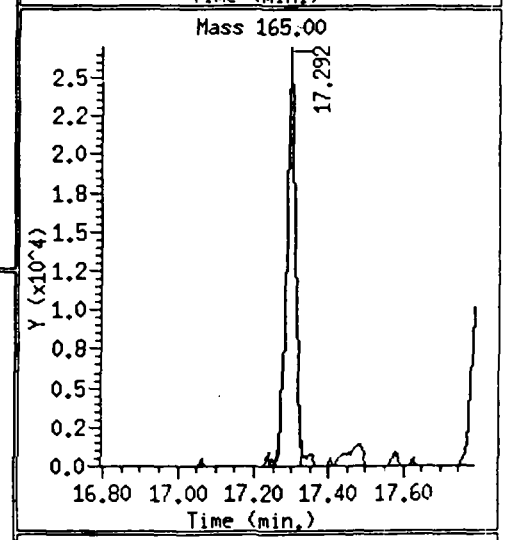


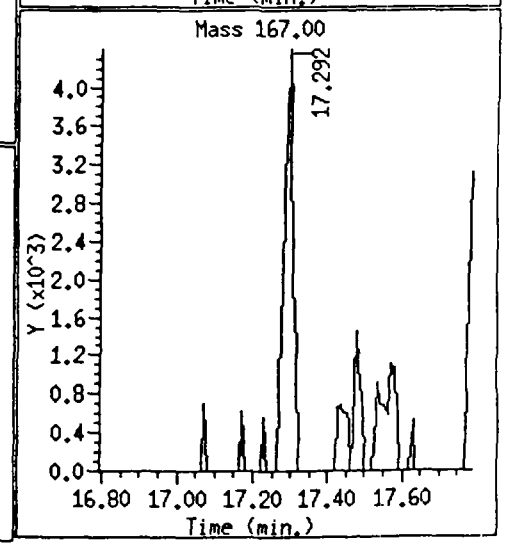












Data File: /chem/V.i/06-08-98.b/VH275.d

Date : 08-JUN-1998 20:43

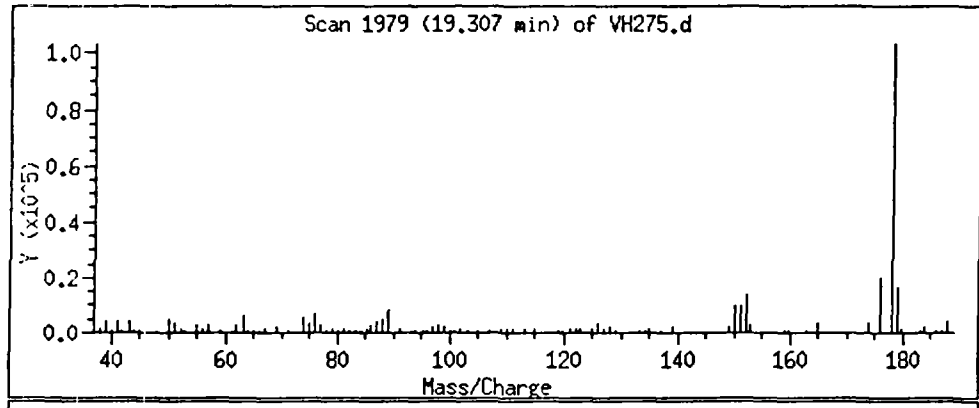
Instrument : V.i

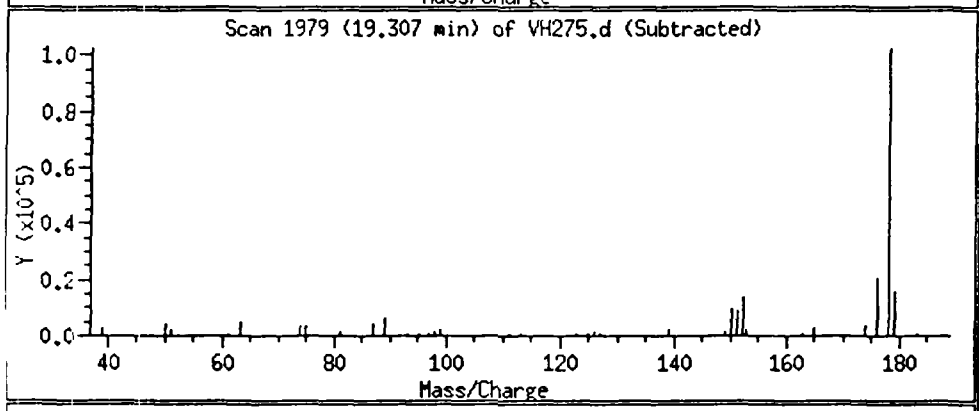
Sample ID : SITE#3 5-28-98 8HR

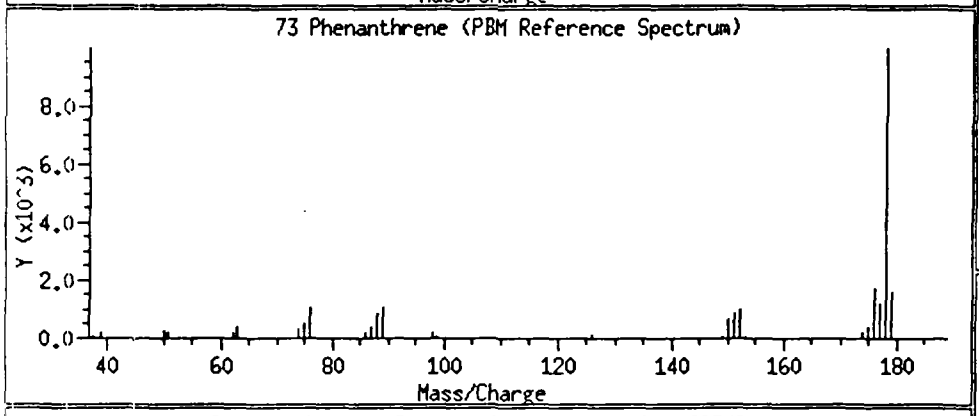
Column phase : DB-5 Column diameter : 0.32

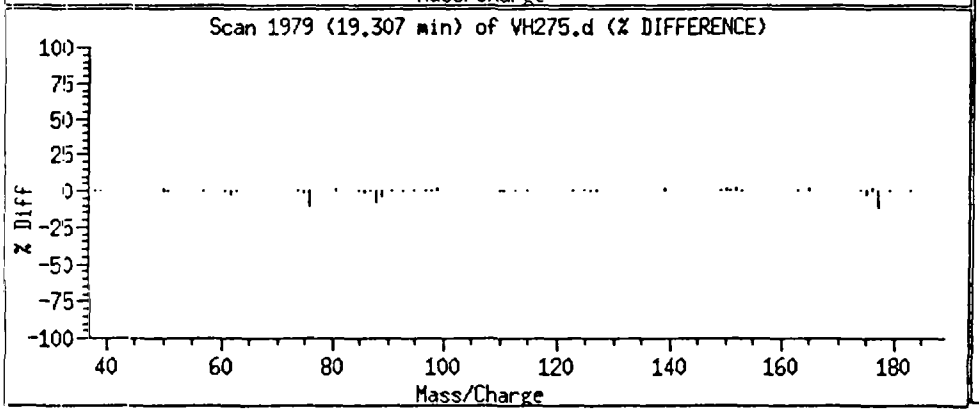
Volume Injected (uL) : 1.0

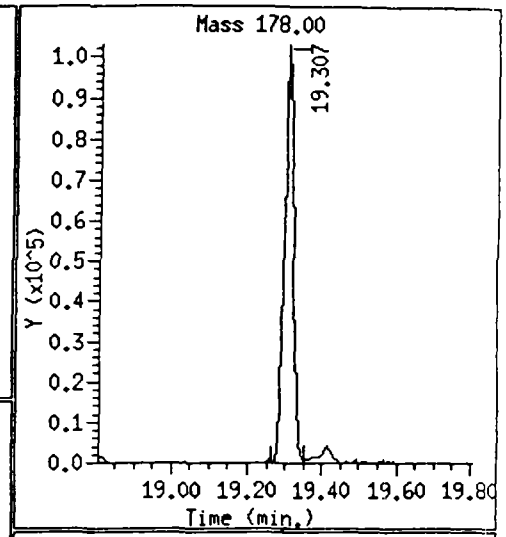
73 Phenanthrene

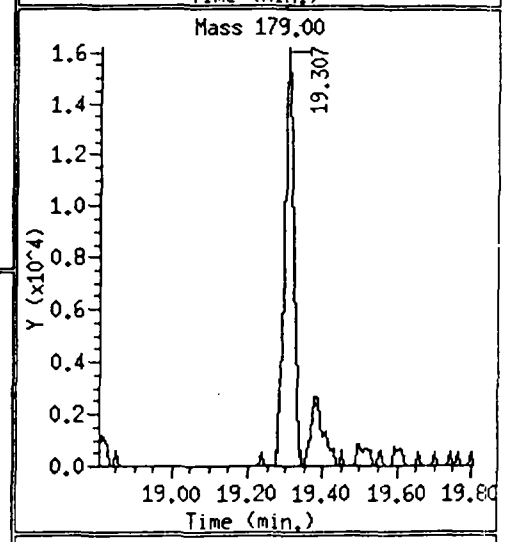


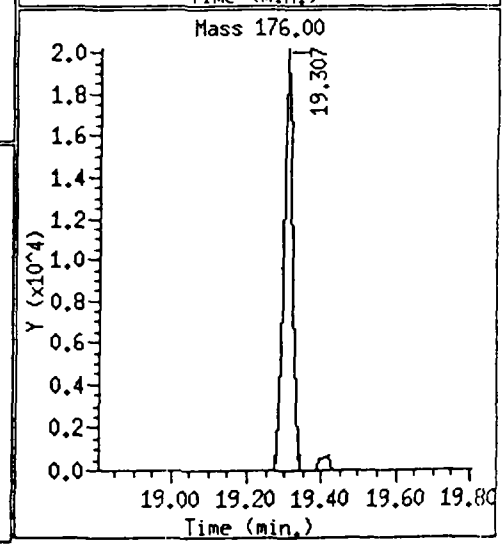












Data File: /chem/V.i/06-08-98.b/VH275.d

Date : 08-JUN-1998 20:43

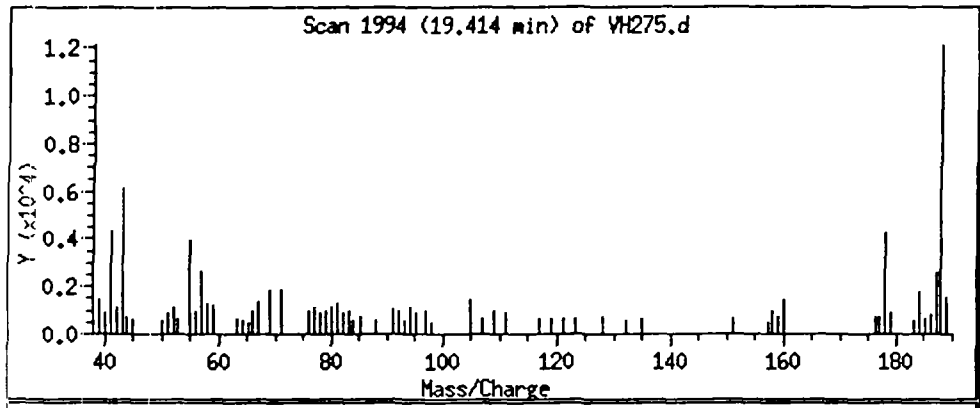
Instrument : V.i

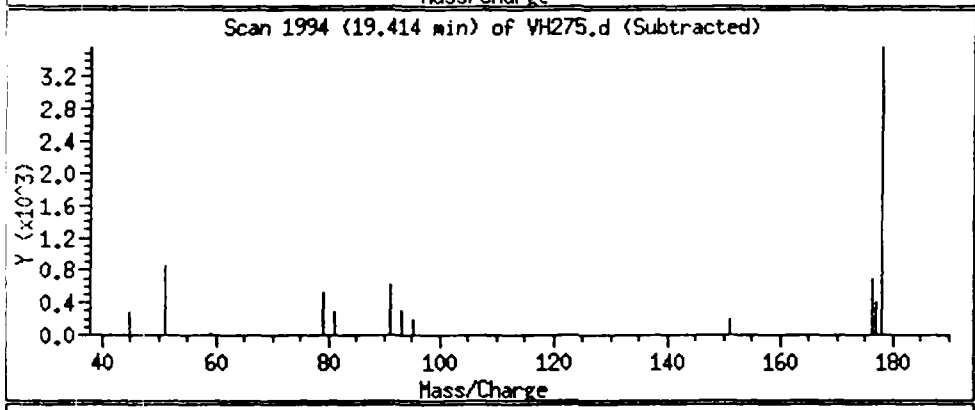
Sample ID : SITE#3 5-28-98 8HR

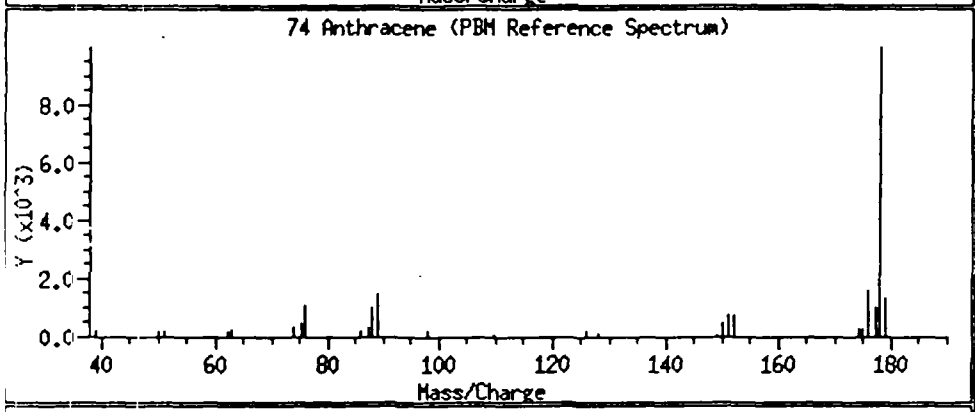
Column phase : DB-5 Column diameter : 0.32

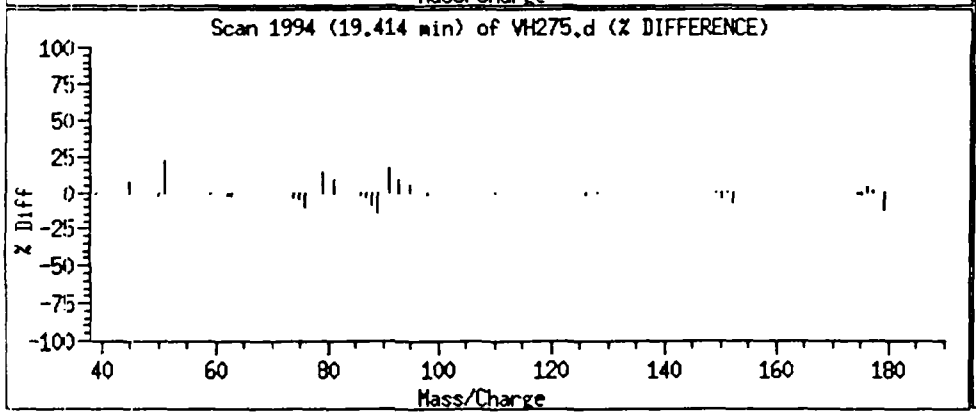
Volume Injected (uL) : 1.0

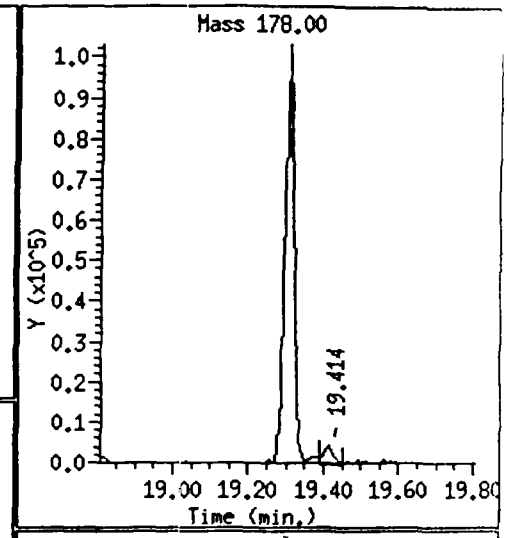
74 Anthracene

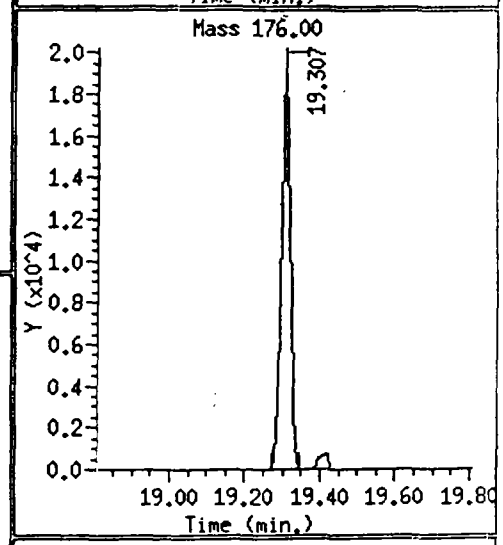


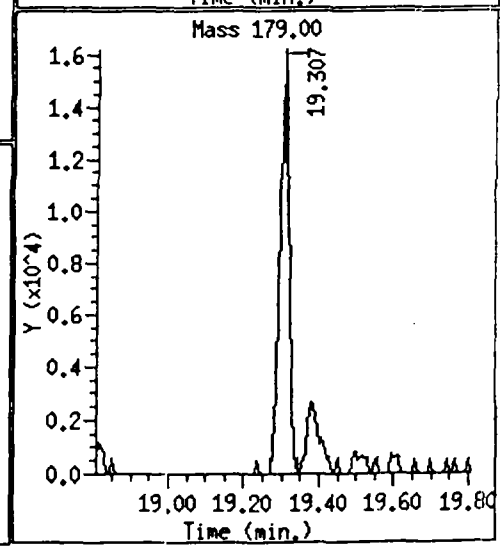












Data File: /chem/V.i/06-08-98.b/VH275.d

Date : 08-JUN-1998 20:43

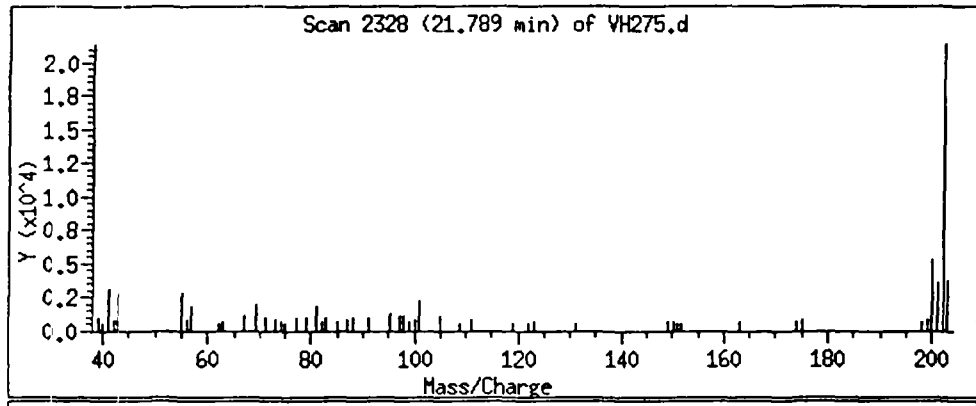
Instrument : V.i

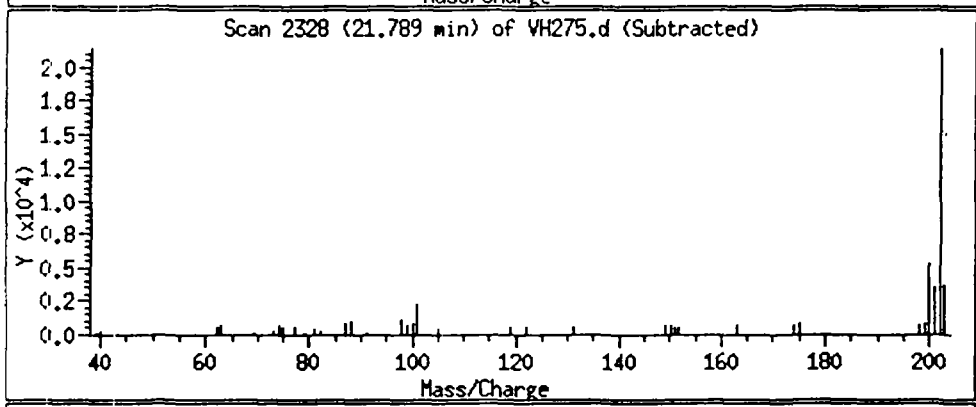
Sample ID : SITE#3 5-28-98 8HR

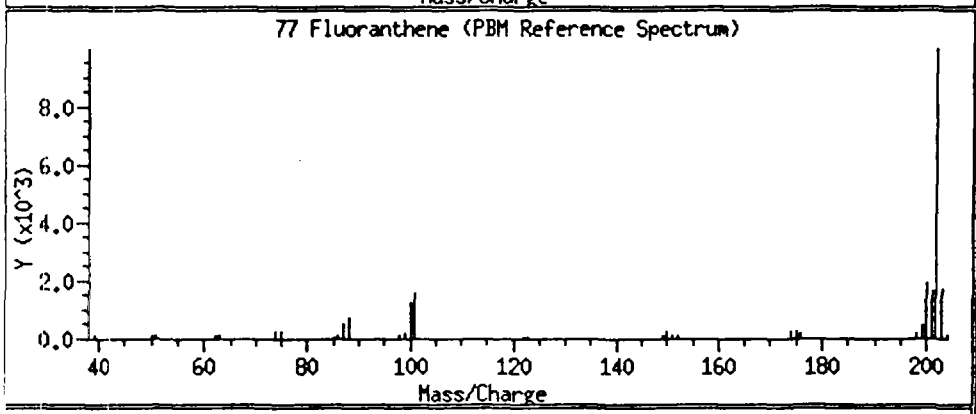
Column phase : DB-5 Column diameter : 0.32

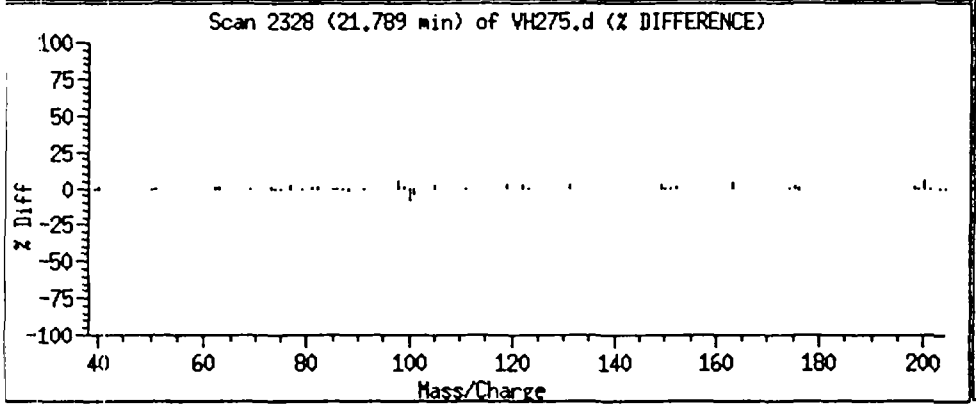
Volume Injected (uL) : 1.0

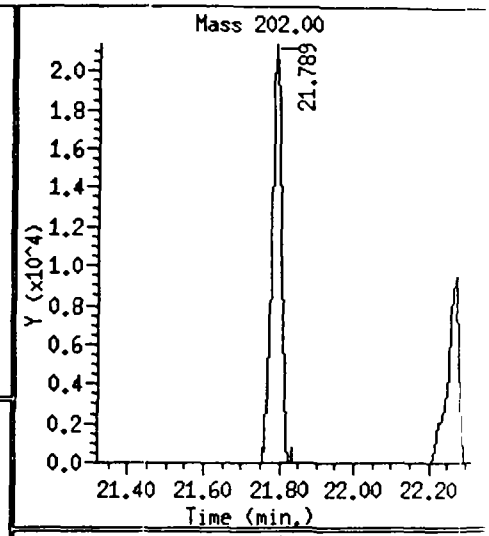
77 Fluoranthene

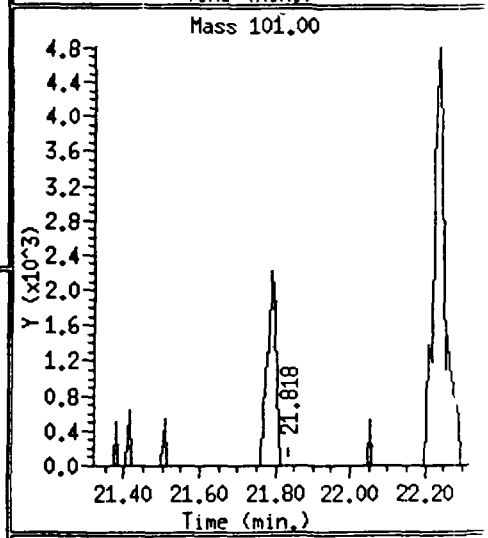


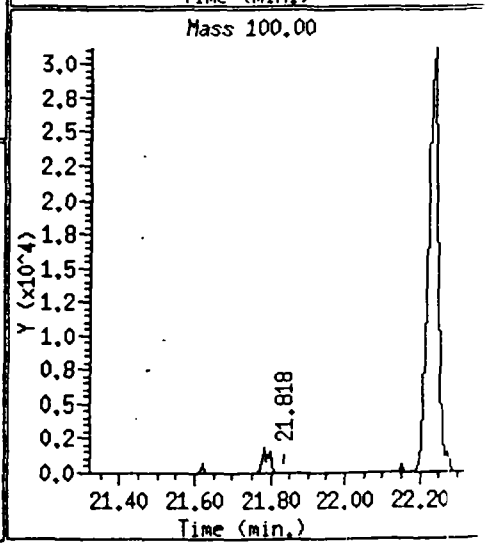












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH280

Method 8270A PUF
Sample ID: SITE#3 5-29-98 24hour

Client Project: Hull & Assoc
TLI ID: 209-67-12,23

Date Received: 06/02/98
Date Extracted: 06/04/98
Date Analyzed : 06/09/98

Response File: VH267

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	73.76	B	12.57		10
Acenaphthene-d_{10}		IS 3	16.16		
Fluorene	21.03		17.30		10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene	65.49	B	19.32		10
Anthracene	2.87	J	19.42		10
Fluoranthene	8.41	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.04	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	98.32	19.39	4	98
Pyrene-d_{10}	118.67	22.24	5	119
Terphenyl-d_{14}	109.69	22.60	5	110

Reviewed by PAB Date 6/12/98

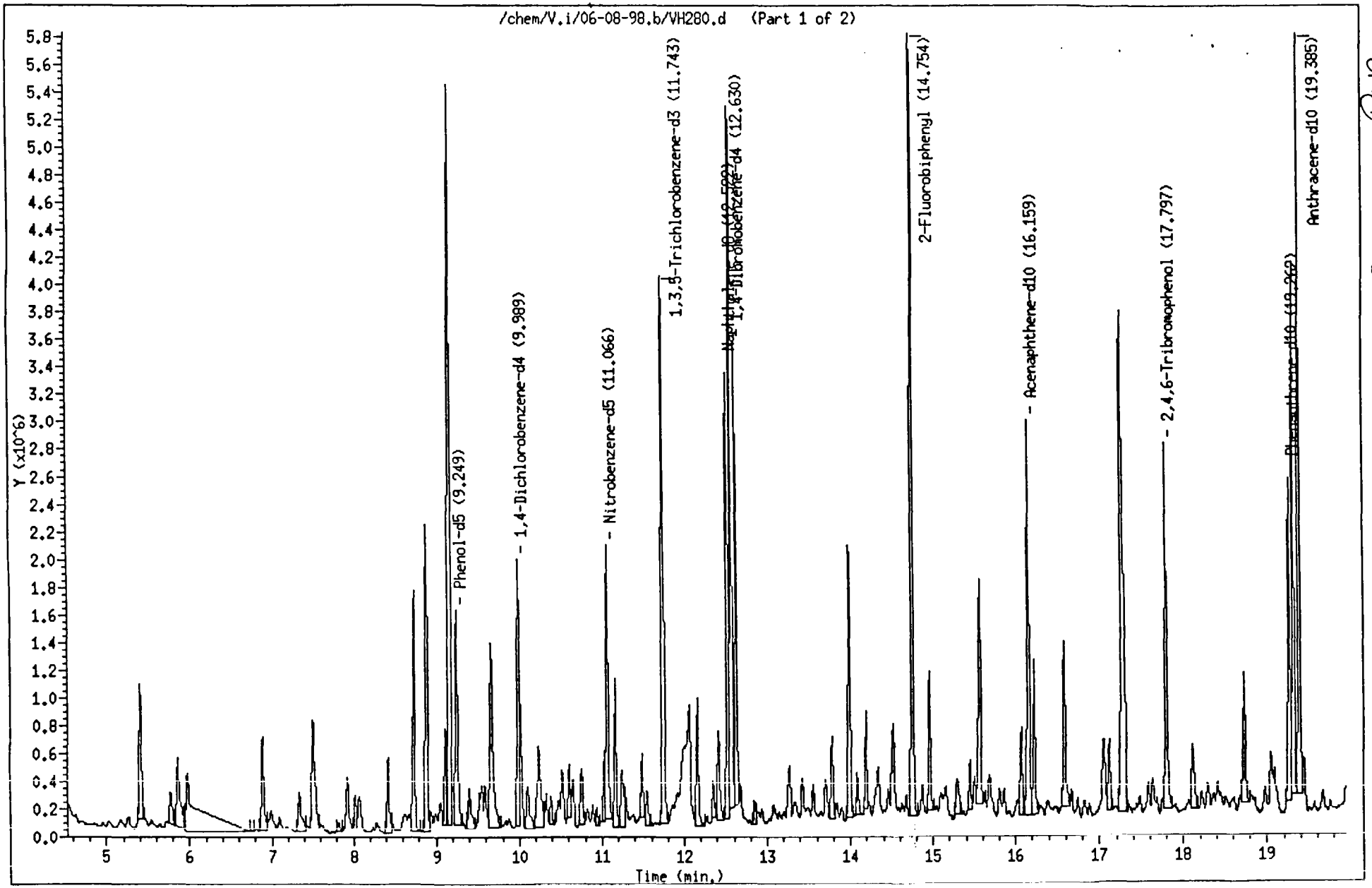
NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Data File: /chem/V.i/06-08-98.b/VH280.d
Date : 09-JUN-1998 00:47
Instrument : V.i
Sample ID : SITE#3 5-27 FRI24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



Data Review: PAB
Date: 6/12/98

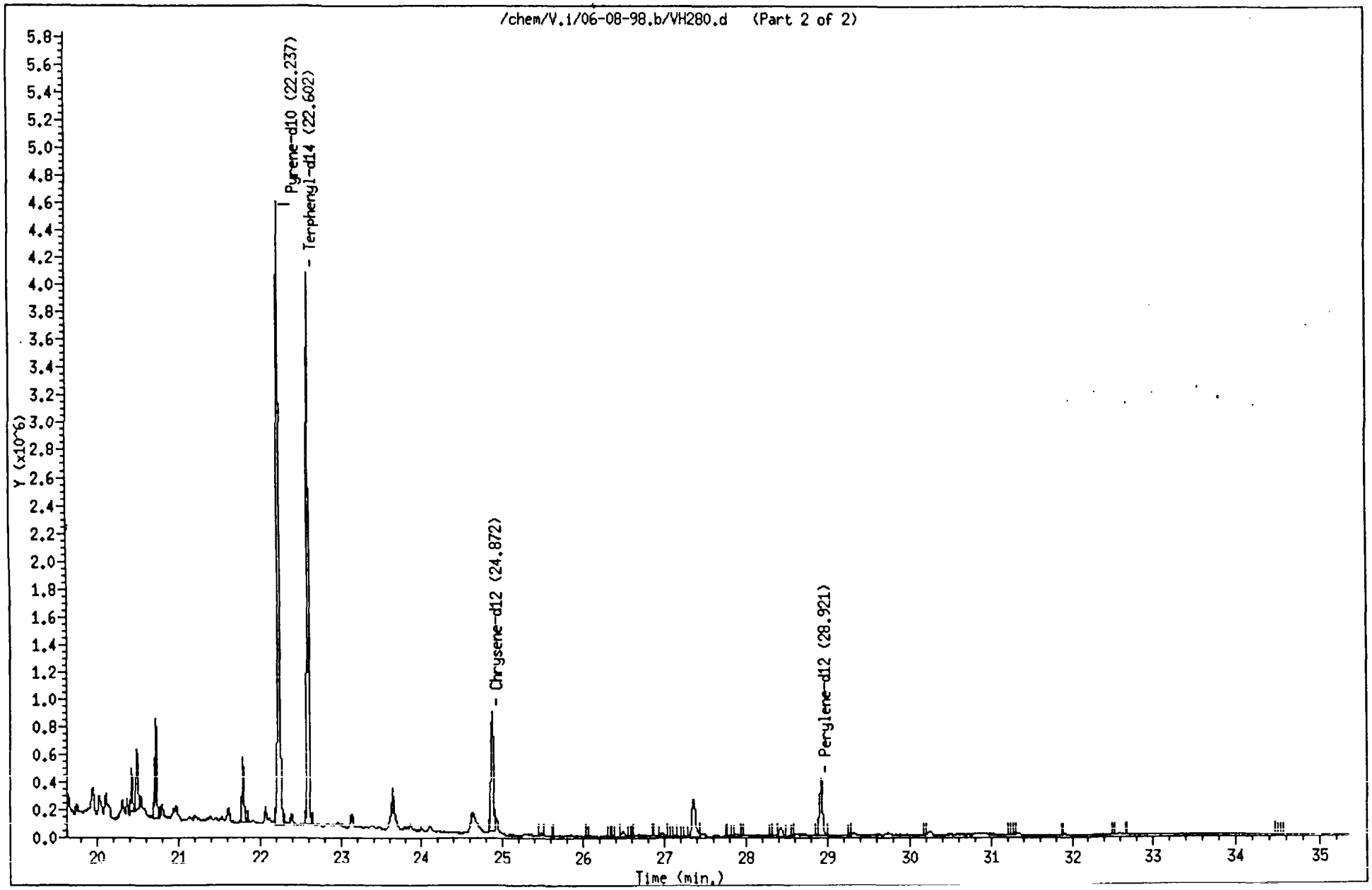
128

129

Data File: /chem/V.i/06-08-98.b/VH280.d
Date : 09-JUN-1998 00:47
Instrument : V.i
Sample ID : SITE#3 5-27 FRI24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH280.d
Lab. Id. : 45869
Inj Date : 09-JUN-1998 00:47 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#3 5-27-98 FRI24HOUR
Misc Info : 209-67-12,23
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 13
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.989(1.000)	496447	40.00	
*	2 Naphthalene-d8	136.00	12.522(1.000)	1840742	40.00	
*	3 Acenaphthene-d10	164.00	16.159(1.000)	954647	40.00	
*	4 Phenanthrene-d10	188.00	19.262(1.000)	1613690	40.00	
*	5 Chrysene-d12	240.00	24.872(1.000)	518591	40.00	
*	6 Perylene-d12	264.00	28.921(1.000)	390510	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.249(0.926)	514539	34.33	34.33
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.066(0.884)	915294	58.54	58.54
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.743(0.938)	1114487	70.13	70.13
$	13 1,4-Dibromobenzene-d4	240.00	12.630(1.264)	891725	79.77	79.77
$	14 2-Fluorobiphenyl	172.00	14.754(0.913)	2451767	89.46	89.46
$	15 2,4,6-Tribromophenol	329.80	17.797(1.101)	385349	70.29	70.29
$	16 Anthracene-d10	188.00	19.385(1.006)	3260426	98.32	98.32
$	17 Pyrene-d10	212.00	22.237(0.894)	2812441	118.65	118.65
$	18 Terphenyl-d14	244.00	22.602(0.909)	1631163	109.72	109.72
	42 Naphthalene	128.00	12.572(1.004)	3265190	73.79	73.79
	46 2-Methylnaphthalene	142.00	13.988(1.117)	794804	29.08	29.08
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	154.00	16.224(1.004)	349919	15.29	15.29
	65 Fluorene	166.00	17.301(1.071)	640065	21.04	21.04
	73 Phenanthrene	178.00	19.320(1.003)	2422684 98866	65.51	65.51
	74 Anthracene	178.00 19.42	~~19.320(1.003)~~	~~2422684~~	~~70.22~~	~~70.22~~ (a) PaB
	77 Fluoranthene	202.00	21.794(1.131)	317436	8.41	8.41(a)
	78 Pyrene	202.00	22.273(0.895)	146126	4.85	4.85(a)
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review. PaB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-08-98.b/VH280.d

Date : 09-JUN-1998 00:47

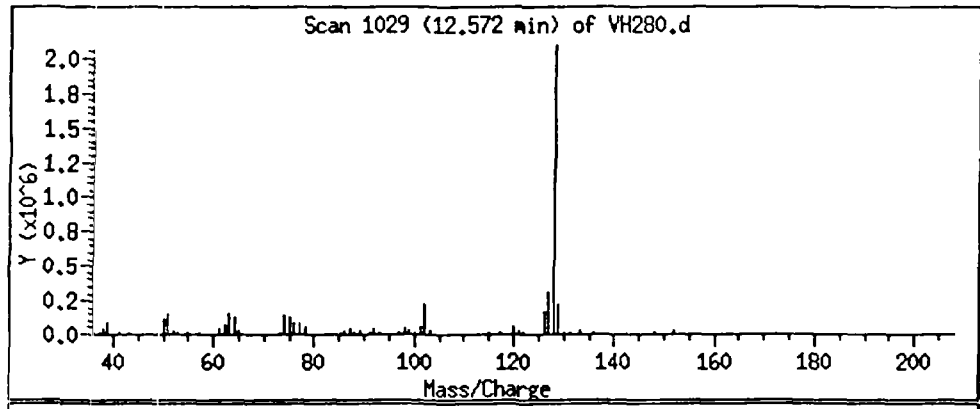
Instrument : V.i

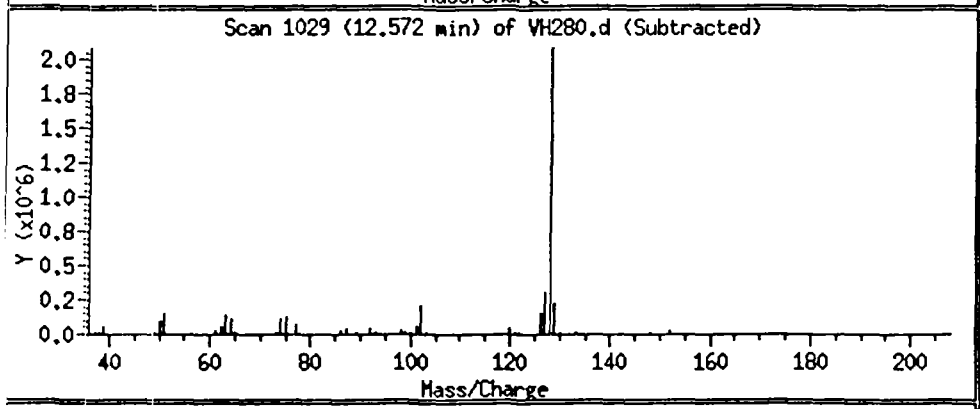
Sample ID : SITE#3 5-27 FRI24HR

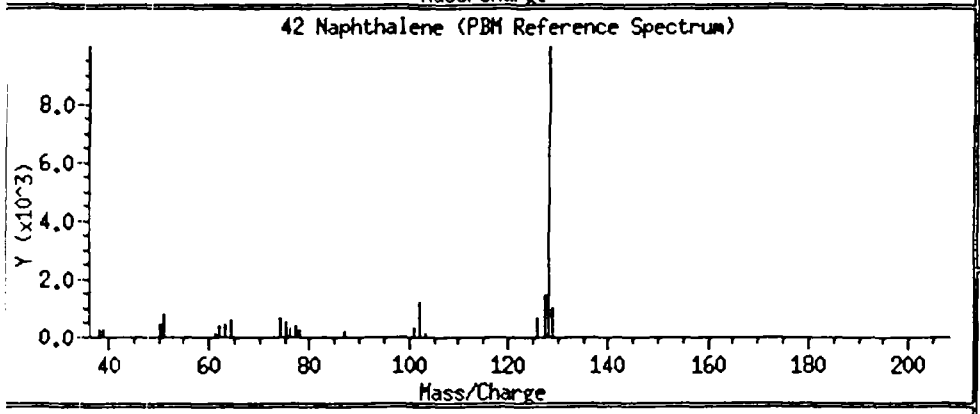
Column phase : DB-5 Column diameter : 0.32

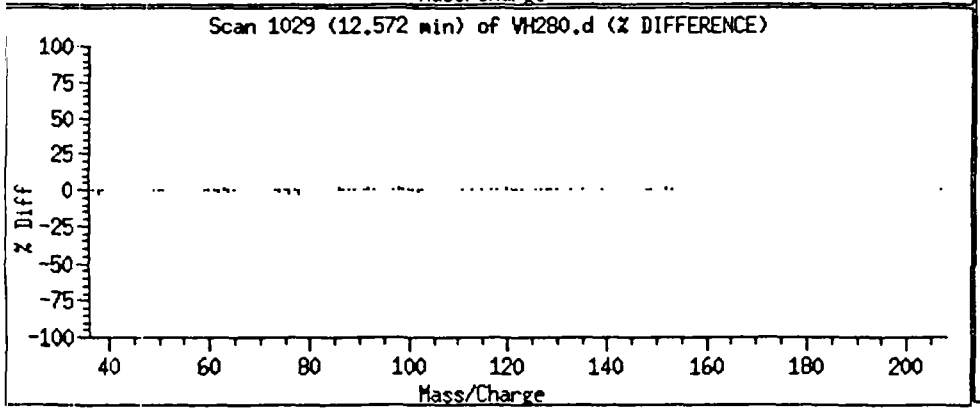
Volume Injected (uL) : 1.0

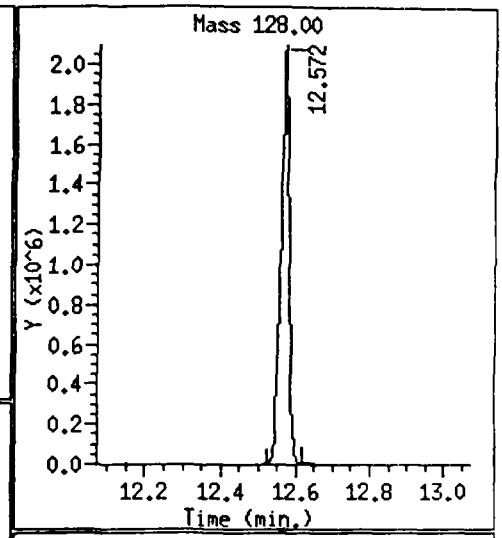
42 Naphthalene

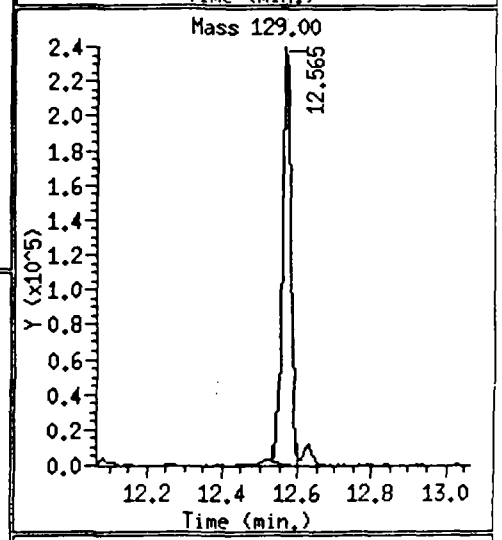


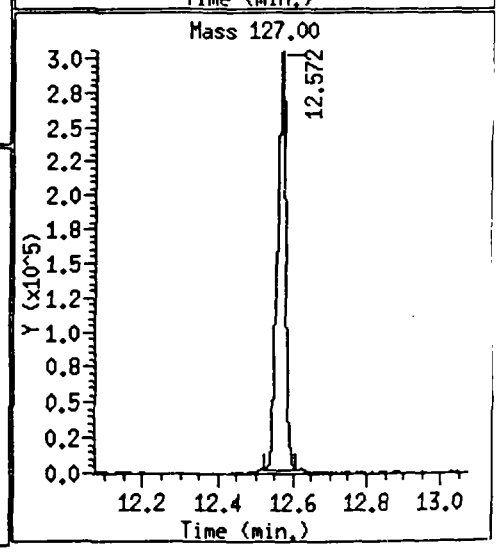












Data File: /chem/V.i/06-08-98.b/VH280.d

Date : 09-JUN-1998 00:47

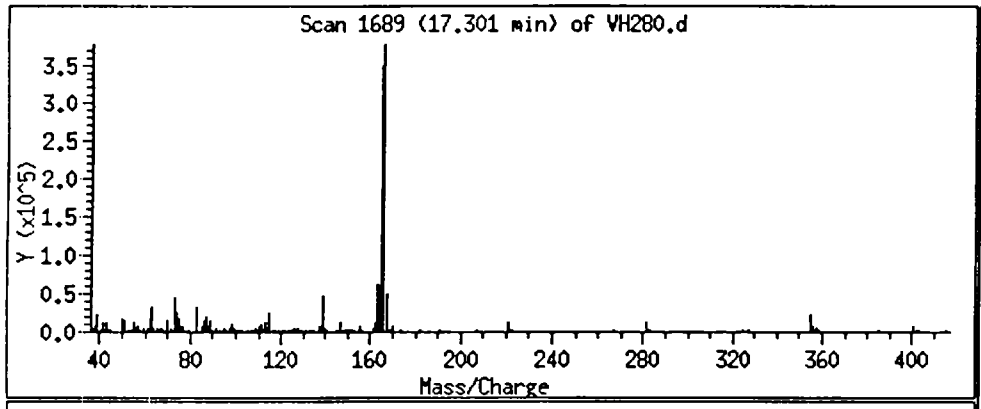
Instrument : V.i

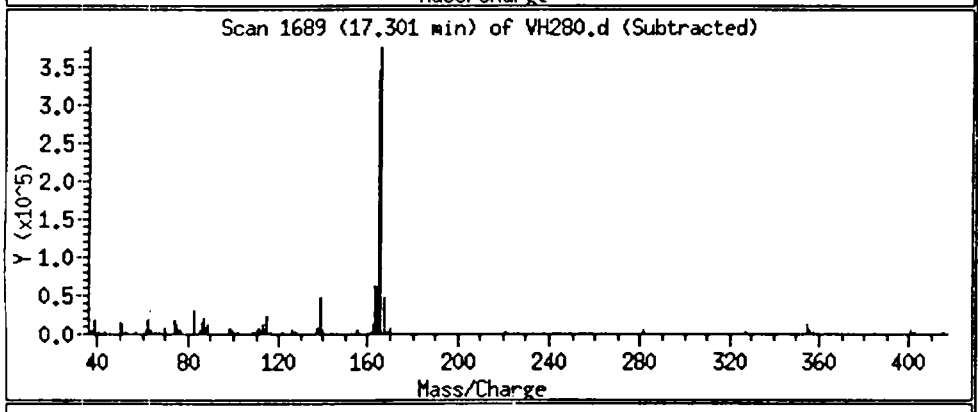
Sample ID : SITE#3 5-27 FRI24HR

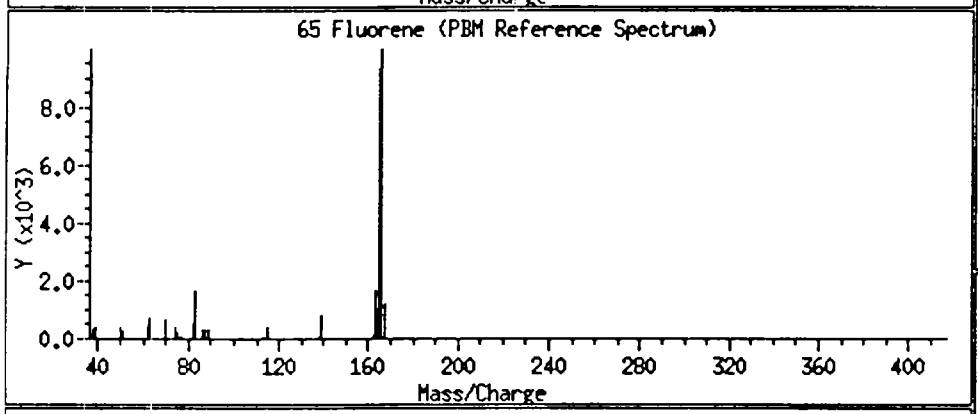
Column phase : DB-5 Column diameter : 0.32

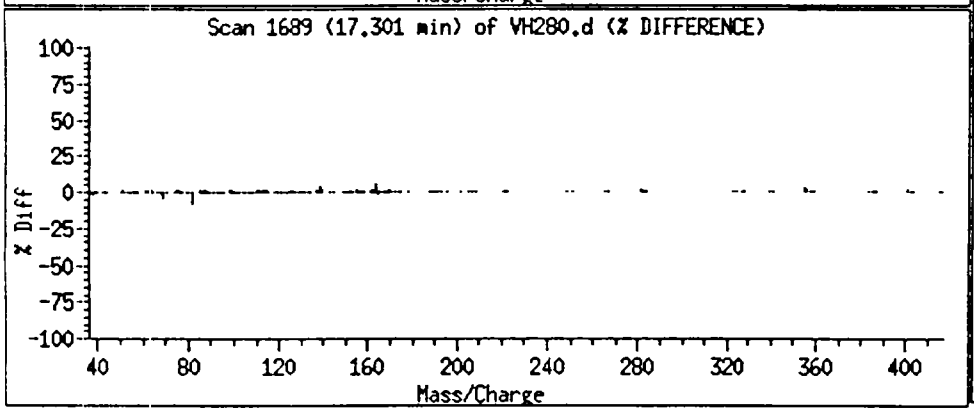
Volume Injected (uL) : 1.0

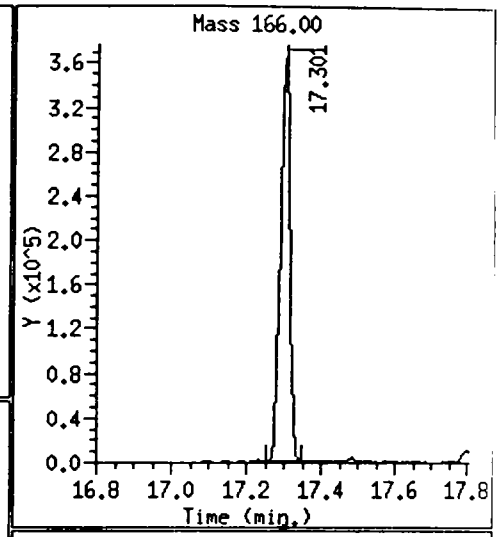
65 Fluorene

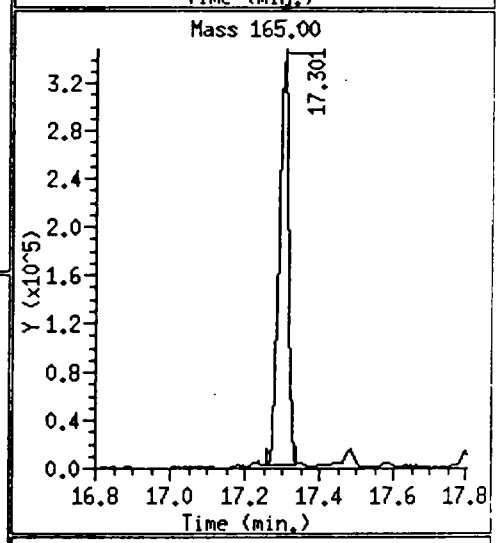


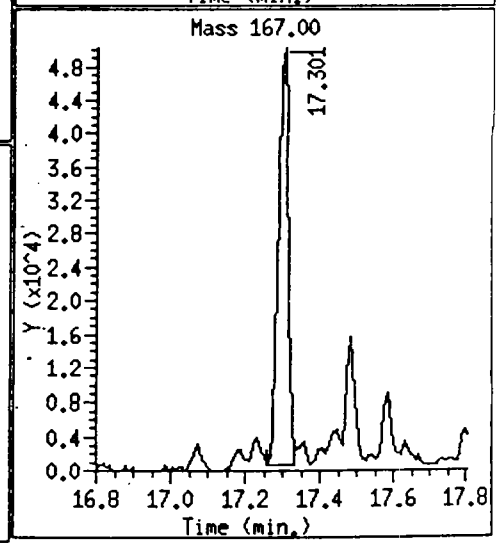












Data File: /chem/V.i/06-08-98.b/VH280.d

Date : 09-JUN-1998 00:47

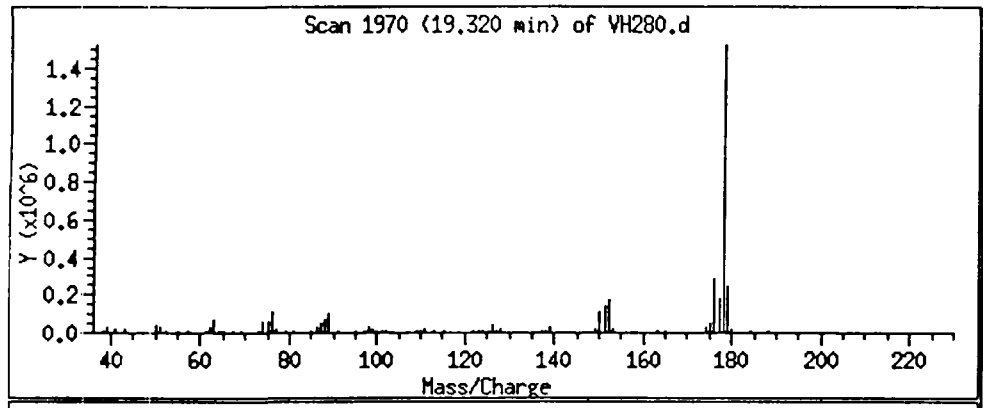
Instrument : V.i

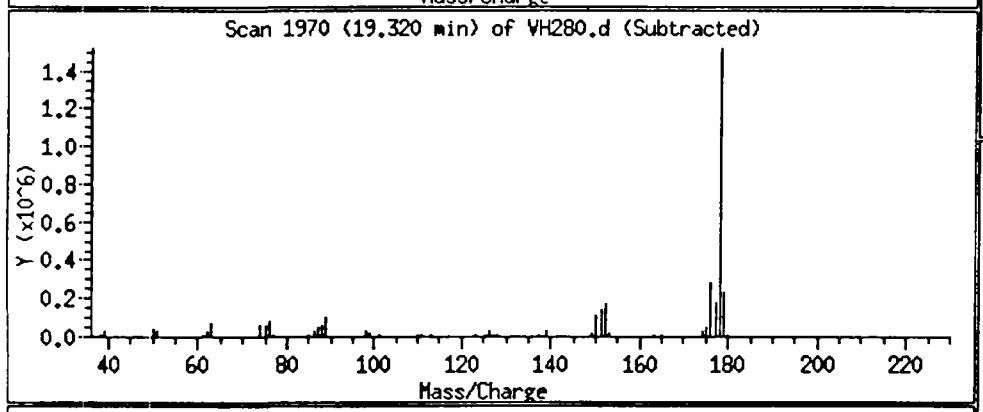
Sample ID : SITE#3 5-27 FRI24HR

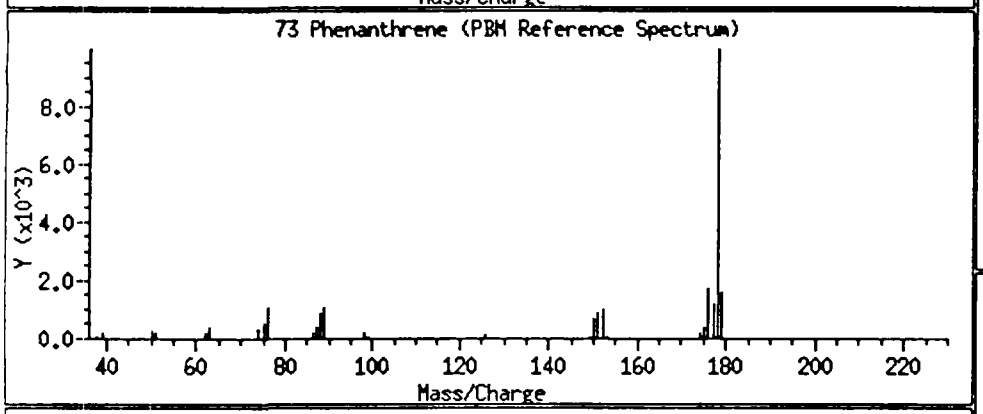
Column phase : DB-5 Column diameter : 0.32

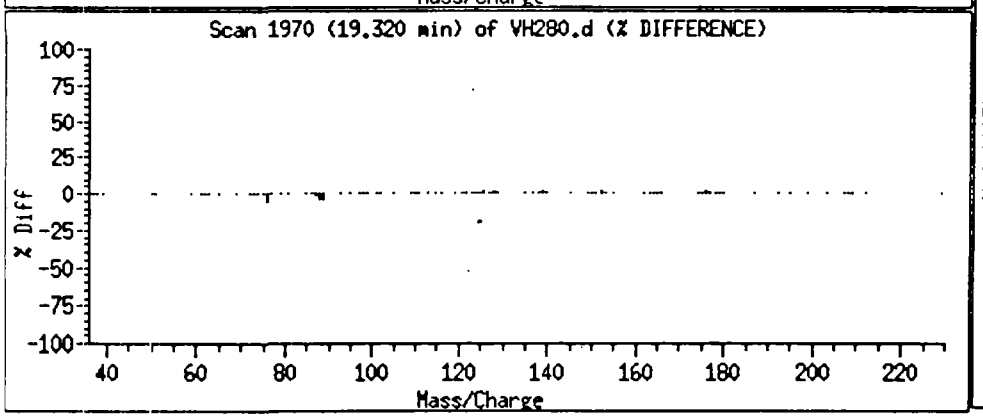
Volume Injected (uL) : 1.0

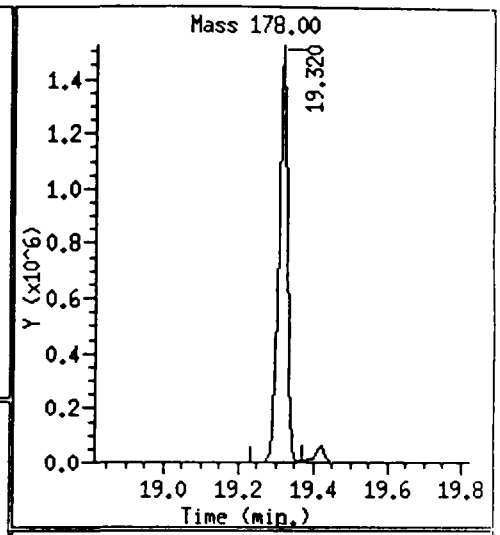
73 Phenanthrene

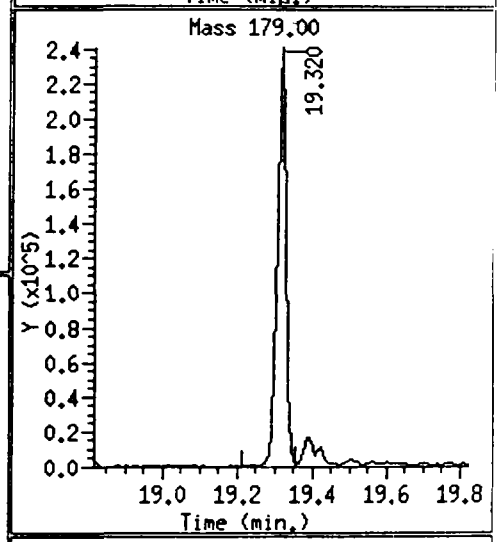


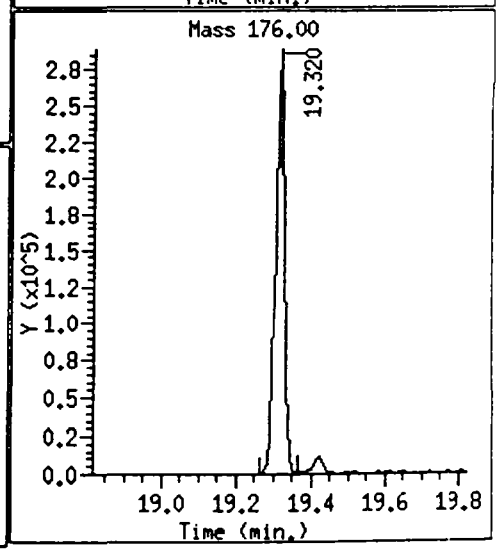












Data File: /chem/V.i/06-08-98.b/VH280.d

Date : 09-JUN-1998 00:47

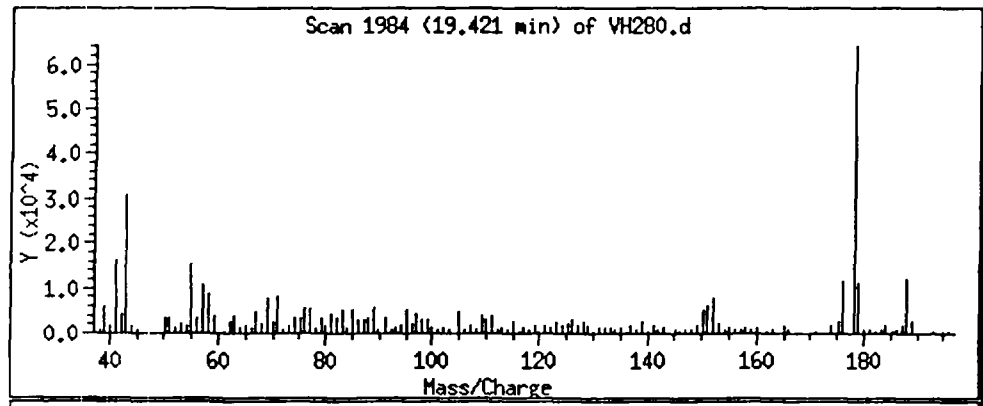
Instrument : V.i

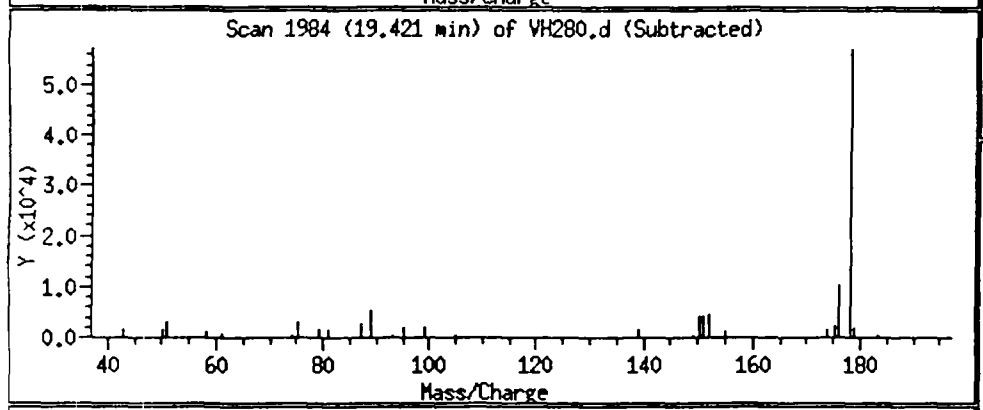
Sample ID : SITE#3 5-27 FRI24HR

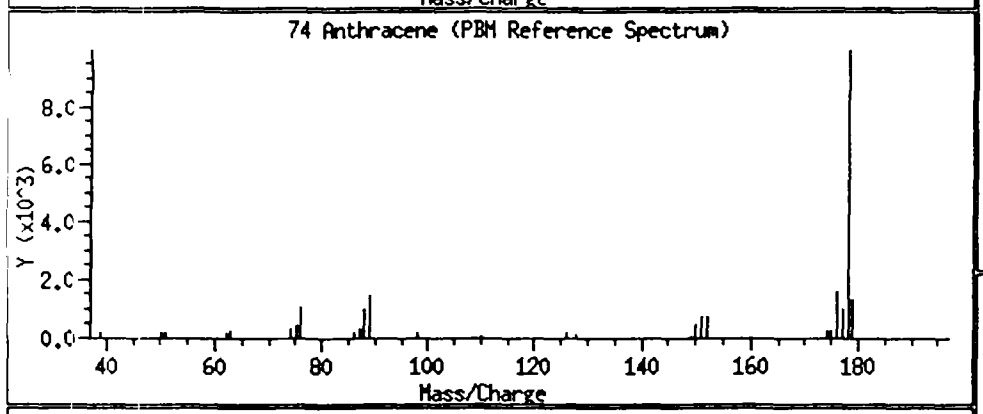
Column phase : DB-5 Column diameter : 0.32

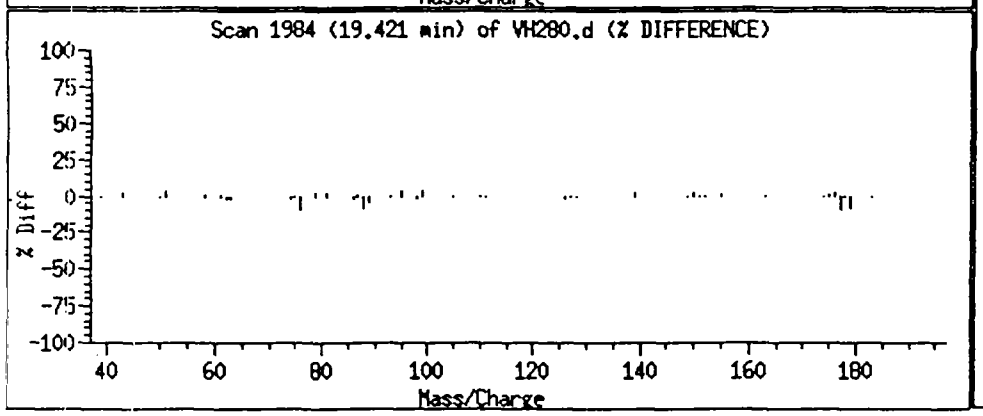
Volume Injected (uL) : 1.0

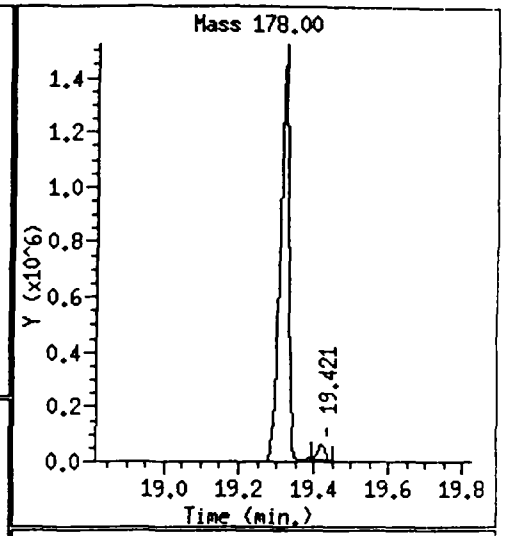
74 Anthracene

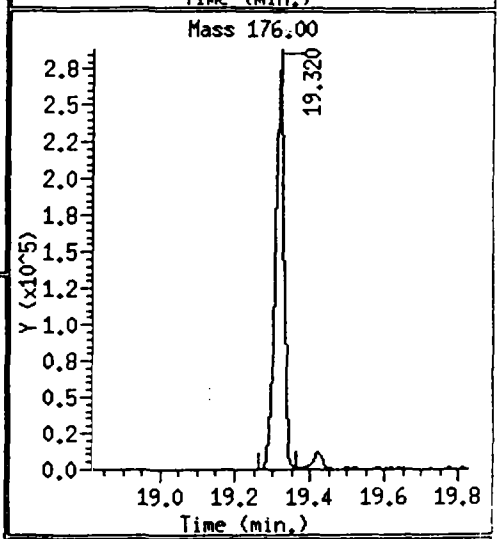


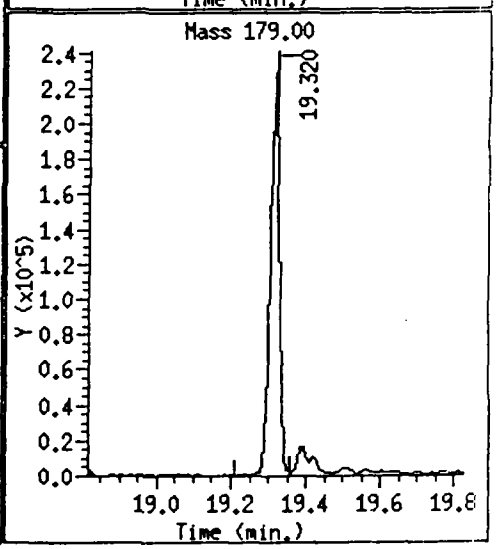












Data File: /chem/V.i/06-08-98.b/VH280.d

Date : 09-JUN-1998 00:47

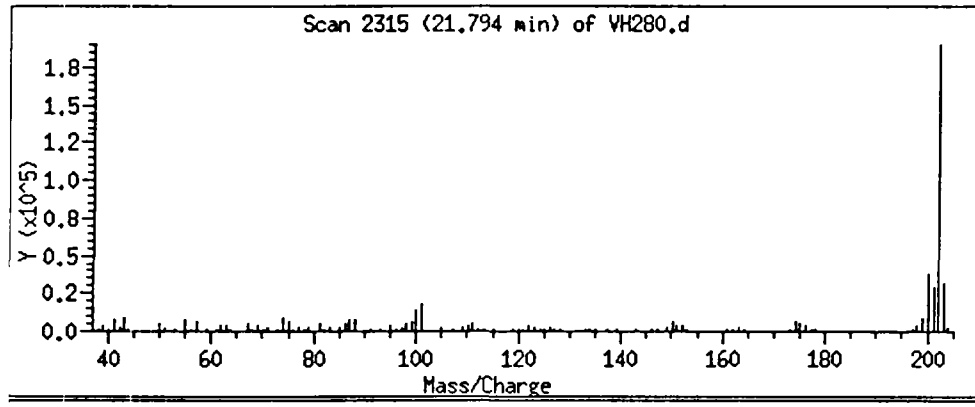
Instrument : V.i

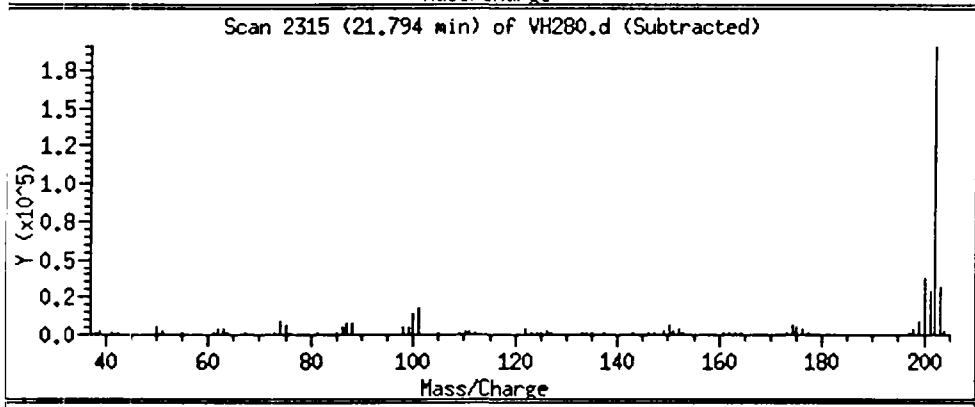
Sample ID : SITE#3 5-27 FRI24HR

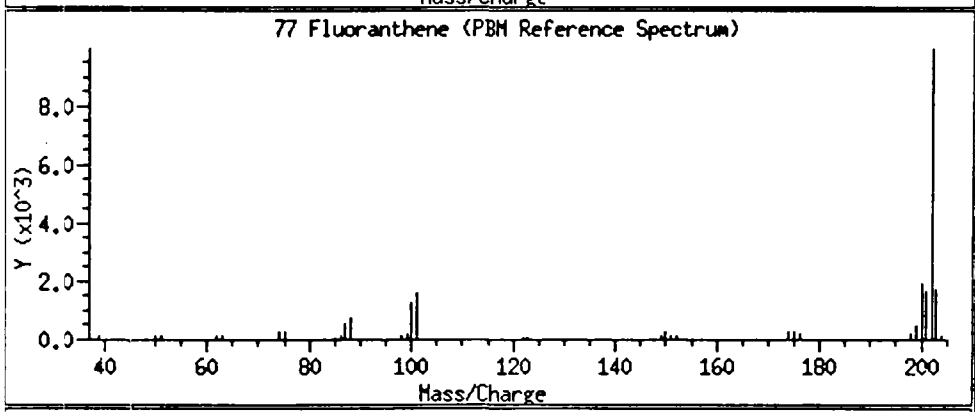
Column phase : DB-5 Column diameter : 0.32

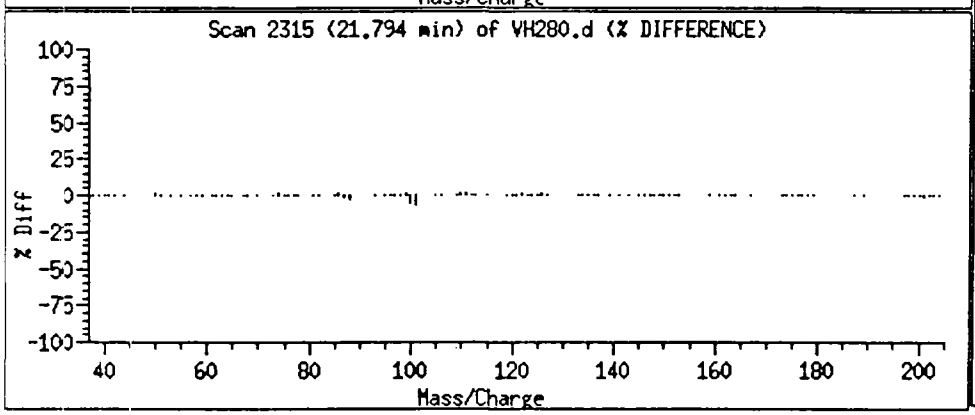
Volume Injected (uL) : 1.0

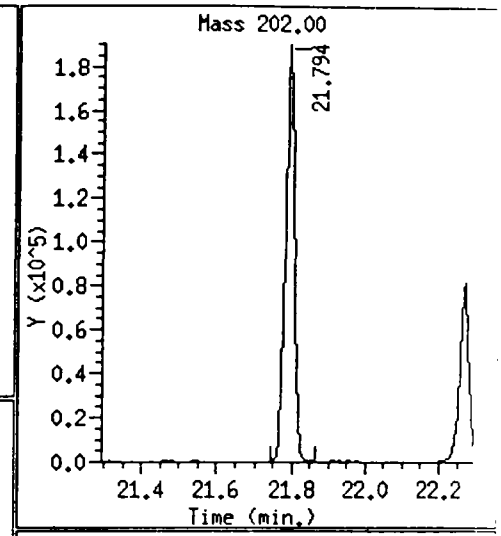
77 Fluoranthene

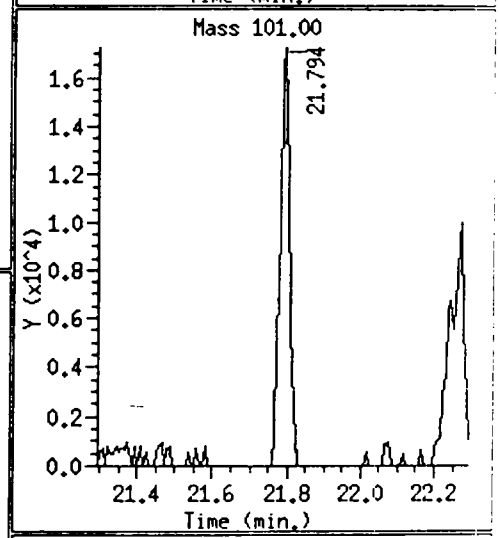


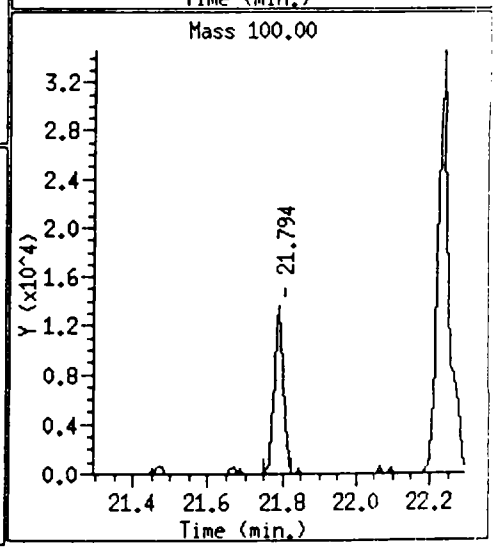












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH290

Method 8270A PUF
Sample ID: SITE#4 5-27-98 24hour

Client Project: Hull & Assoc
TLI ID: 209-67-13,17

Date Received: 06/02/98
Date Extracted: 06/04/98
Date Analyzed : 06/09/98

Response File: VH284

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	6.73	BJ	12.56		10
Acenaphthene-d_{10}		IS 3	16.15		
Fluorene	4.90	J	17.30		10
Phenanthrene-d_{10}		IS 4	19.27		
Phenanthrene	25.96	B	19.31		10
Anthracene	0.70	J	19.42		10
Fluoranthere	3.65	J	21.79		10
Chrysene-d_{12}		IS 5	24.88		
Perylene-d_{12}		IS 6	28.93		
Benzo(a)pyrene		U		1.75	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	90.80	19.38	4	91
Pyrene-d_{10}	114.08	22.24	5	114
Terphenyl-d_{14}	93.87	22.61	5	94

Reviewed by PAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

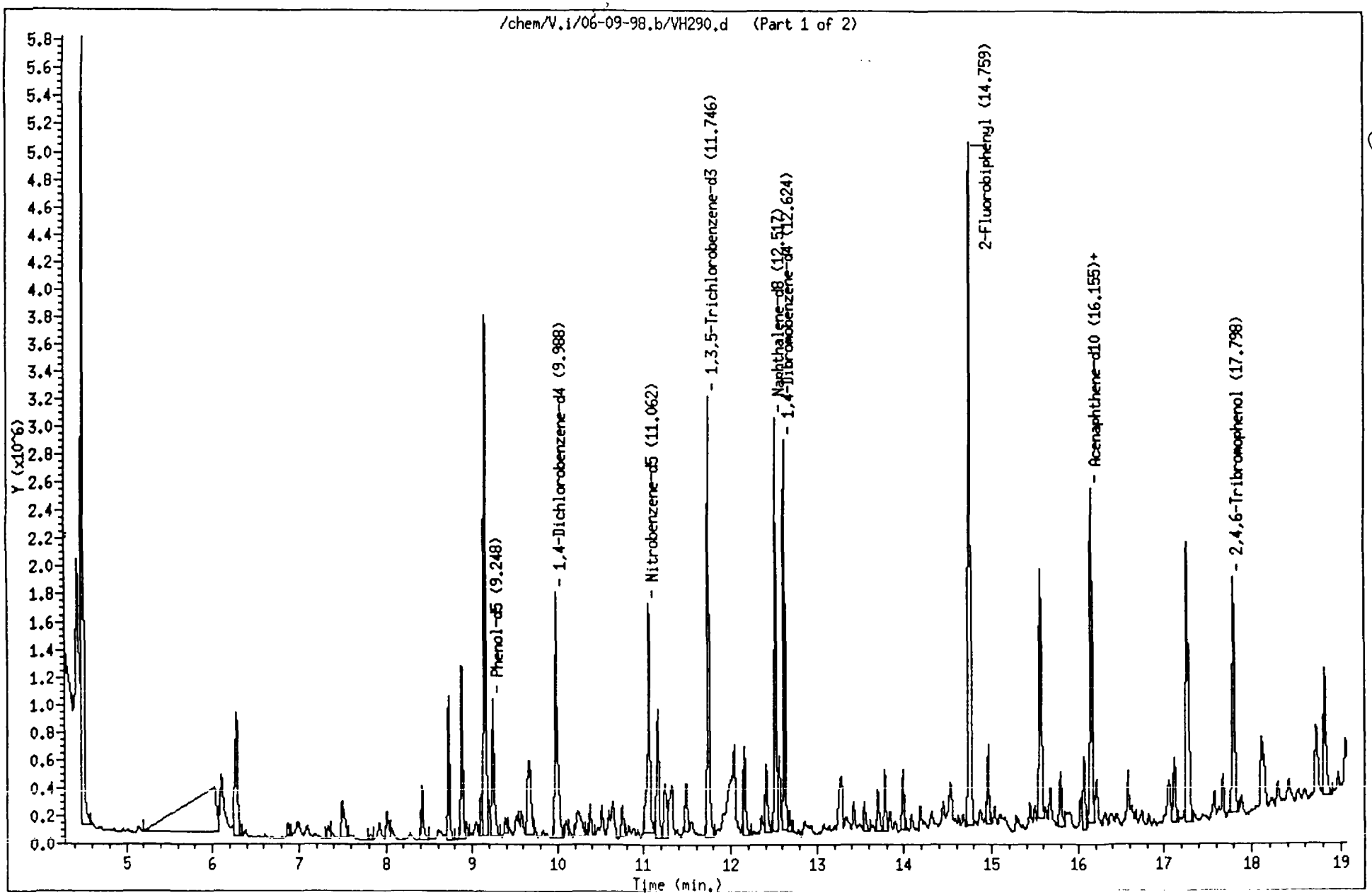
Savar v3.7
Printed: 15:16 06/12/1998

Data File: /chem/V.i/06-09-98.b/VH290.d
Date : 09-JUN-1998 16:16
Instrument : V.i
Sample ID : SITE#4 5-27-98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

138



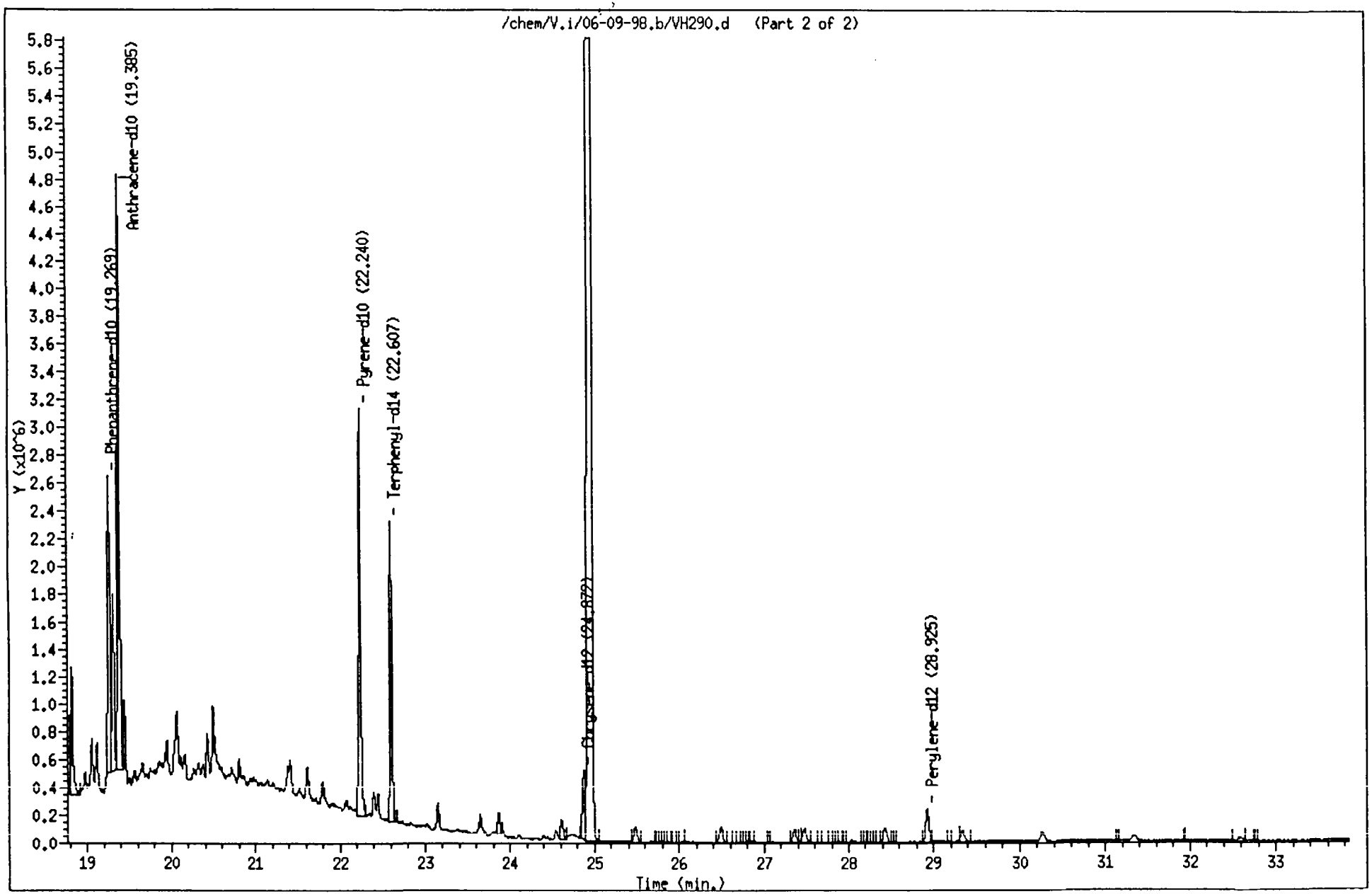
Data Review: [illegible]
Date: 6/12/98

139

Data File: /chem/V.i/06-09-98.b/VH290.d
Date : 09-JUN-1998 16:16
Instrument : V.i
Sample ID : SITE#4 5-27-98 24HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-09-98.b/VH290.d
Lab. Id. : 45869
Inj Date : 09-JUN-1998 16:16 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#4 5-27-98 24HOUR
Misc Info : 209-67-13,17
Comment :
Method : /chem/V.i/06-09-98.b/cclist.m
Meth Date : 09-Jun-1998 12:27 target
Cal Date : 09-JUN-1998 10:31 Cal File: VH284.d
Als bottle: 6
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.988(1.000)	428430	40.00	
*	2 Naphthalene-d8	136.00	12.517(1.000)	1607247	40.00	
*	3 Acenaphthene-d10	164.00	16.155(1.000)	841351	40.00	
*	4 Phenanthrene-d10	188.00	19.269(1.000)	1381801	40.00	
*	5 Chrysene-d12	240.00	24.879(1.000)	301155	40.00	
*	6 Perylene-d12	264.00	28.925(1.000)	230596	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.248(0.926)	431685	32.67	32.67
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.062(0.884)	798240	59.03	59.03
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.746(0.938)	885351	60.79	60.79
$	13 1,4-Dibromobenzene-d4	240.00	12.624(1.264)	708561	65.86	65.86
$	14 2-Fluorobiphenyl	172.00	14.759(0.914)	2020057	84.38	84.38
$	15 2,4,6-Tribromophenol	329.80	17.798(1.102)	243938	62.51	62.51
$	16 Anthracene-d10	188.00	19.385(1.006)	2578460	90.78	90.78
$	17 Pyrene-d10	212.00	22.240(0.894)	1766814	114.08	114.08
$	18 Terphenyl-d14	244.00	22.607(0.909)	882667	93.85	93.85
	42 Naphthalene	128.00	12.560(1.003)	268009	6.73	6.73(a)
	46 2-Methylnaphthalene	142.00	13.987(1.117)	156303	6.40	6.40(a)
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	152.00	~~16.140(0.999)~~	~~8388~~	~~0.24~~	~~0.24(a)~~ NA PaB
	58 Acenaphthene	154.00	16.220(1.004)	71202	3.49	3.49(a)
	65 Fluorene	166.00	17.295(1.071)	140217	4.90	4.90(a)
	73 Phenanthrene	178.00	19.312(1.002)	806163	25.95	25.95
	74 Anthracene	178.00	19.42 ~~19.312(1.002)~~	~~806163~~ 20482	~~27.66~~	~~27.66~~ NA PaB
	77 Fluoranthene	202.00	21.793(1.131)	111468	3.64	3.64(a)
	78 Pyrene	Compound Not Detected.				
	83 Benzo(a)anthracene	228.00	~~24.951(1.005)~~	~~3225~~	~~0.33~~	~~0.33(a)~~ 7P PaB
	84 Chrysene	228.00	~~24.951(1.003)~~	~~3225~~	~~0.39~~	~~0.39(a)~~ 7P PaB
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PaB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-09-98.b/VH290.d

Date : 09-JUN-1998 16:16

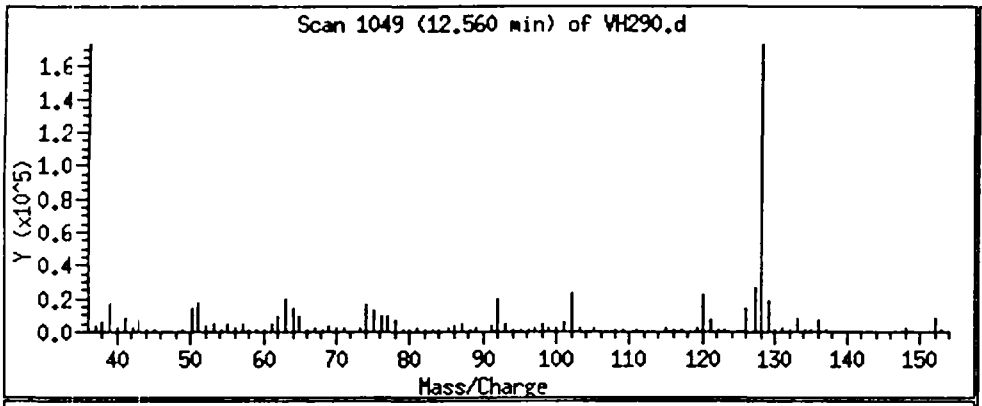
Instrument : V.i

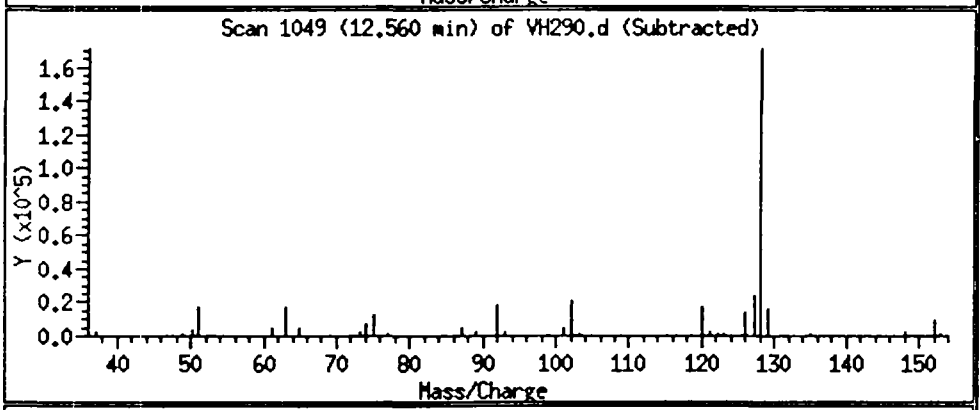
Sample ID : SITE#4 5-27-98 24HR

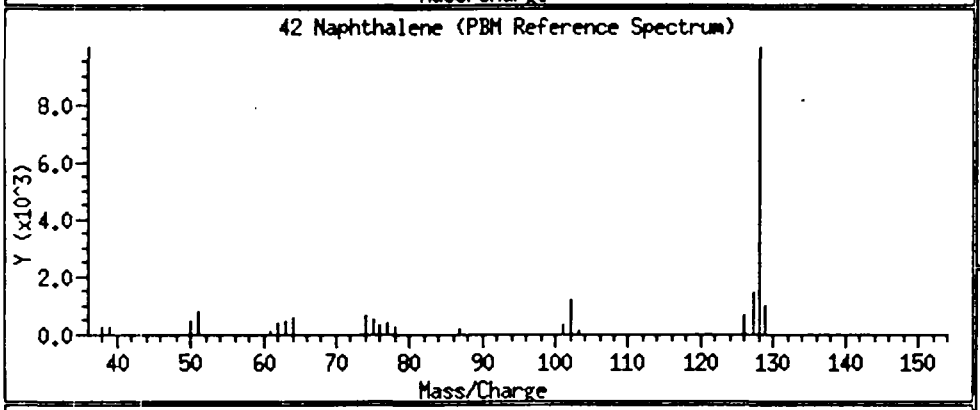
Column phase : DB-5 Column diameter : 0.32

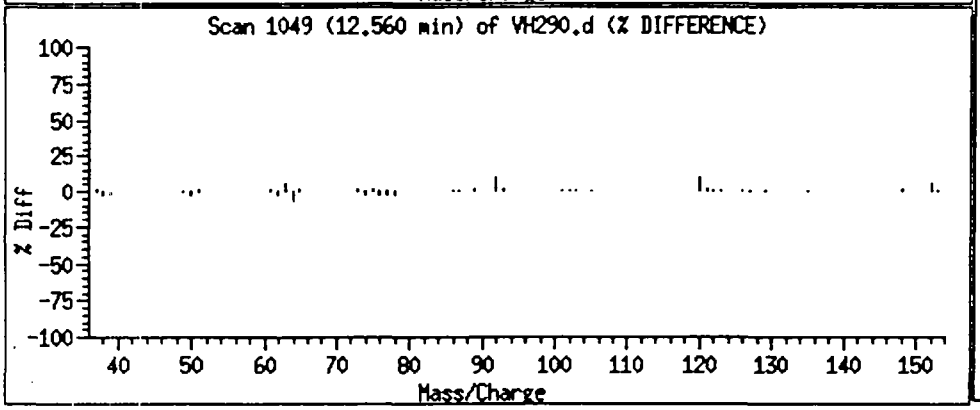
Volume Injected (uL) : 1.0

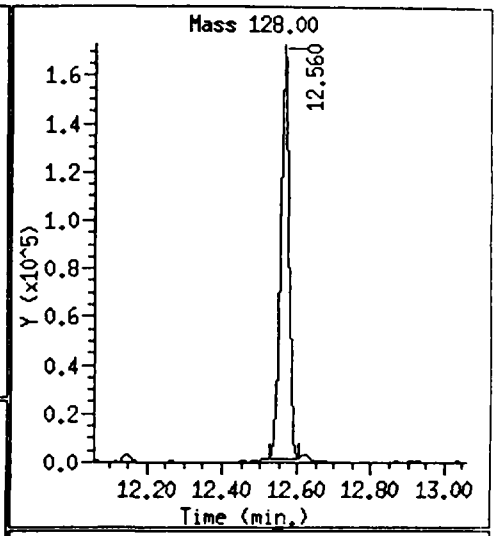
42 Naphthalene

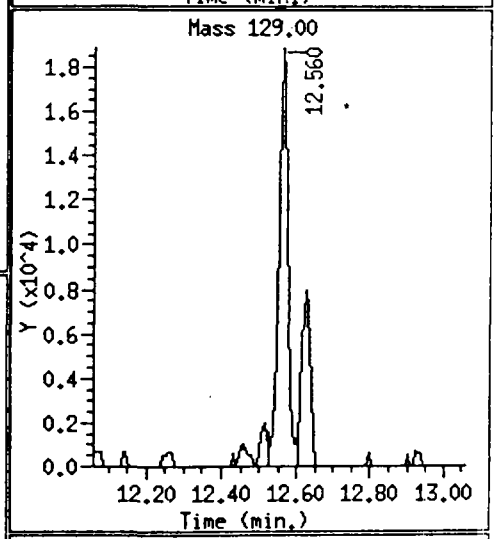


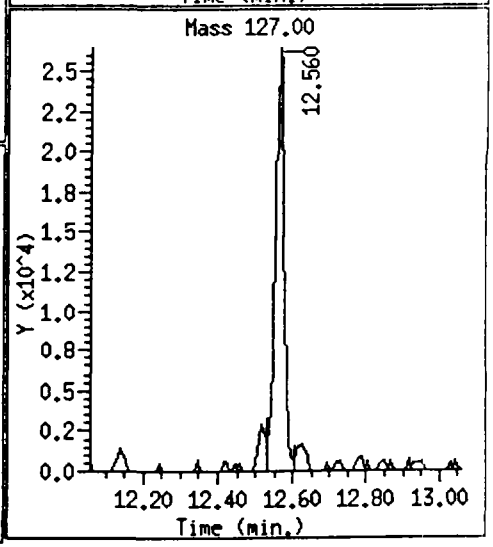












Data File: /chem/V.i/06-09-98.b/VH290.d

Date : 09-JUN-1998 16:16

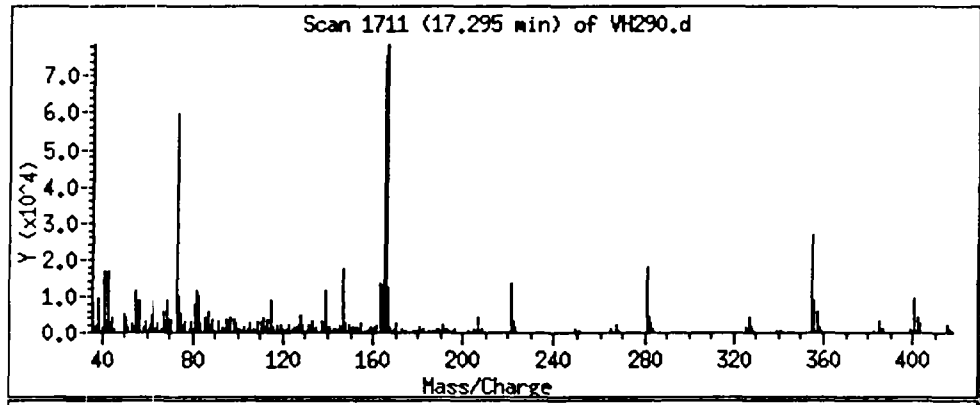
Instrument : V.i

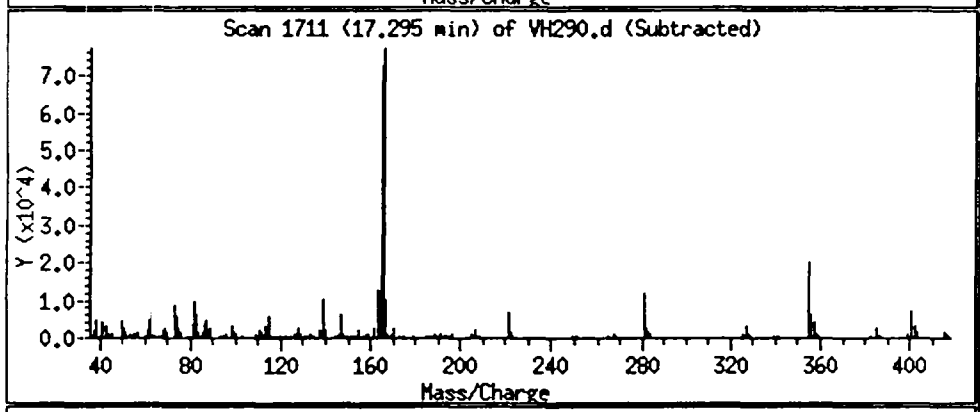
Sample ID : SITE#4 5-27-98 24HR

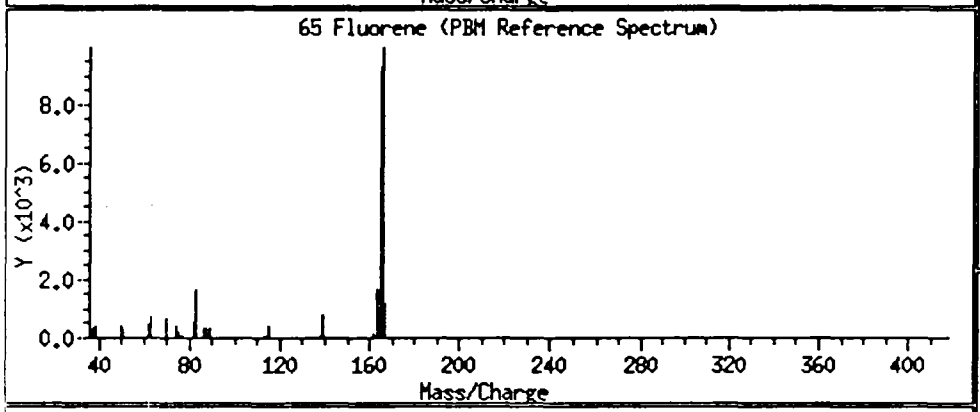
Column phase : DB-5 Column diameter : 0.32

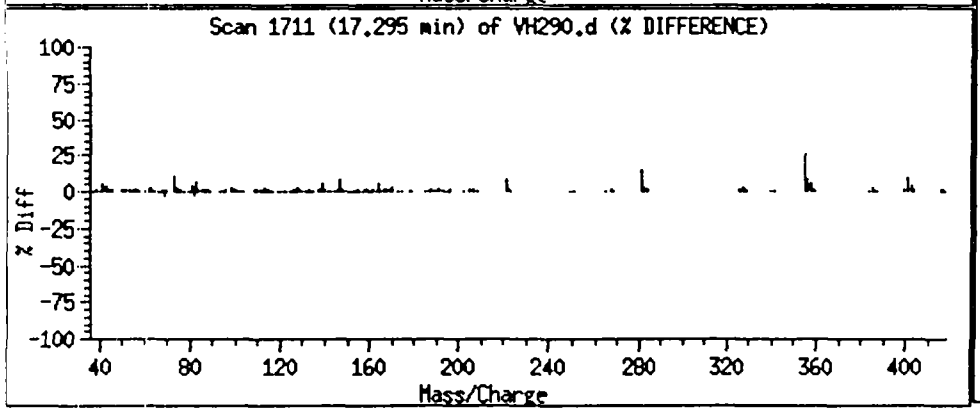
Volume Injected (uL) : 1.0

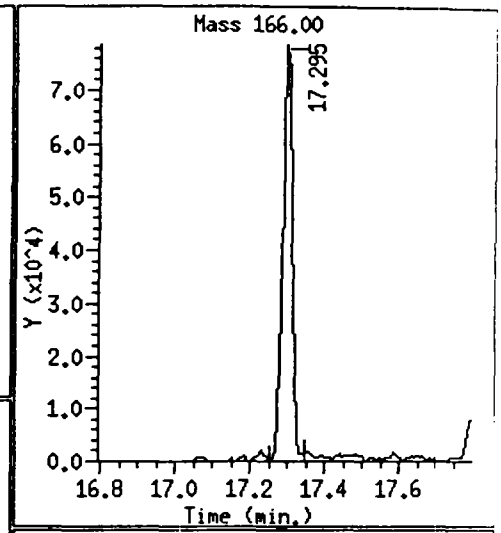
65 Fluorene

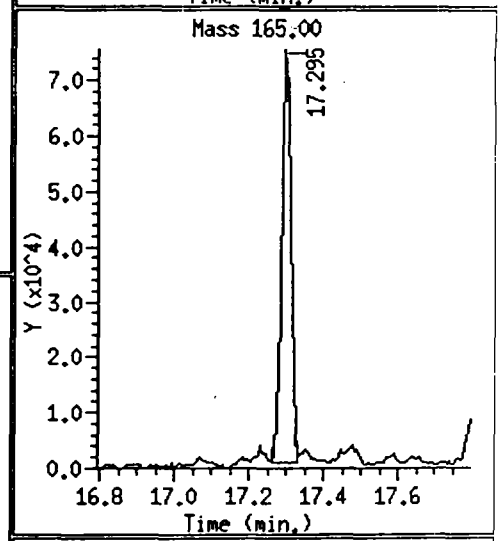


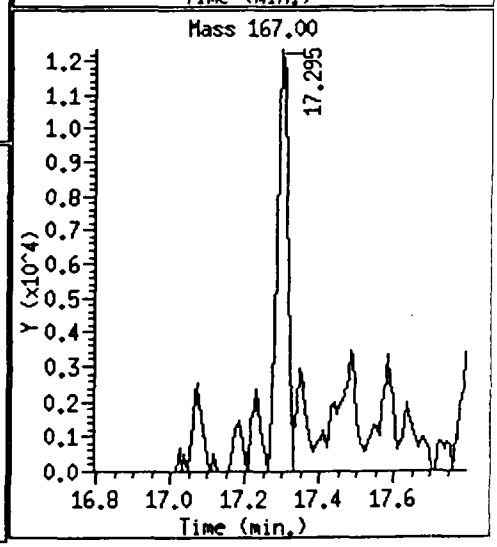












Data File: /chem/V.i/06-09-98.b/VH290.d

Date : 09-JUN-1998 16:16

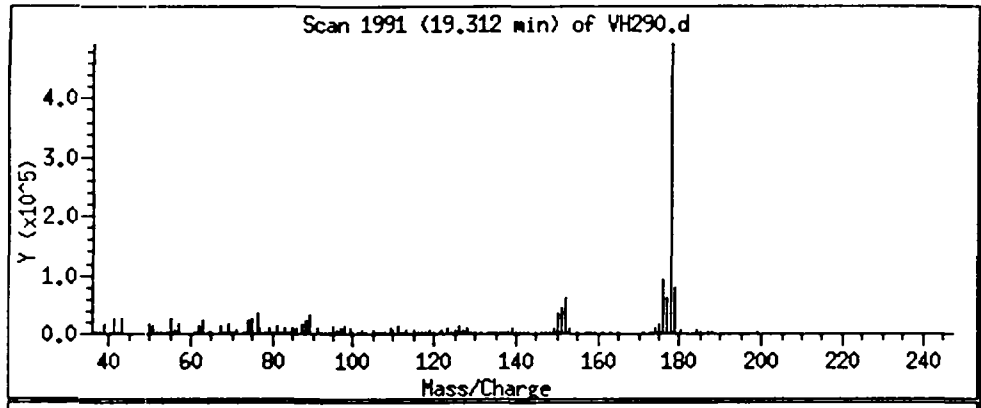
Instrument : V.i

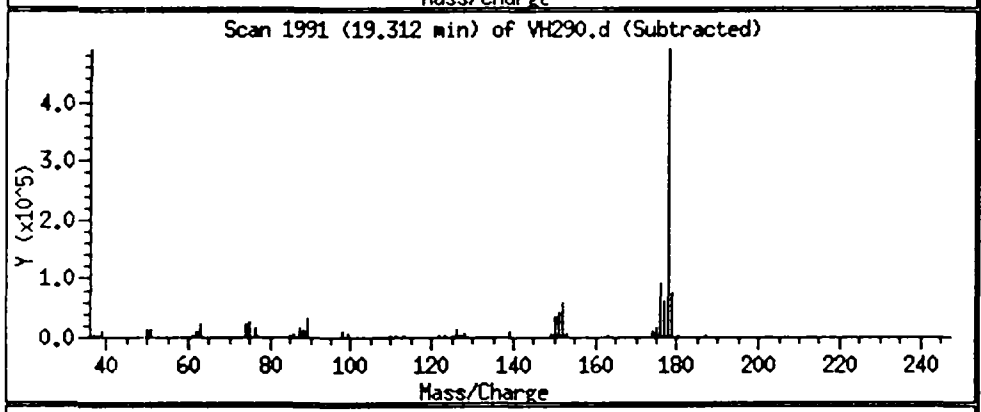
Sample ID : SITE#4 5-27-98 24HR

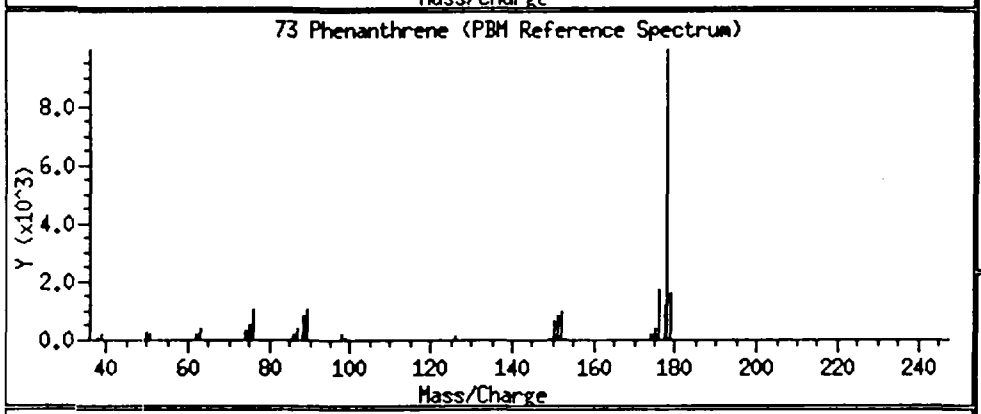
Column phase : DB-5 Column diameter : 0.32

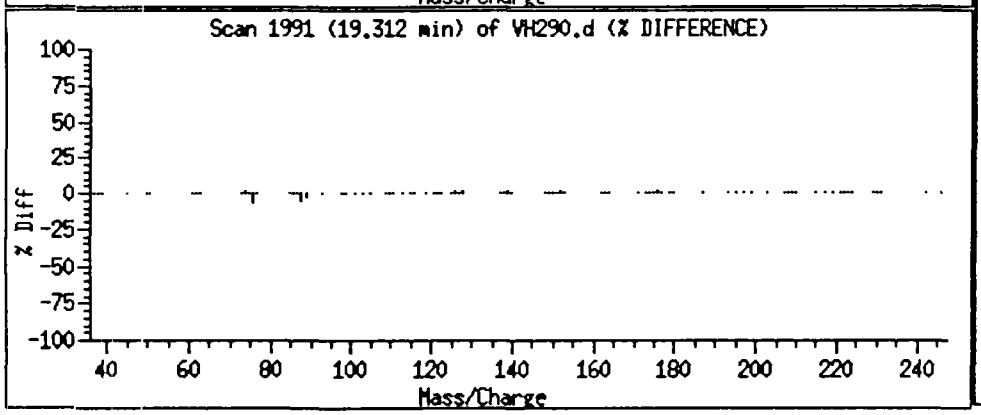
Volume Injected (uL) : 1.0

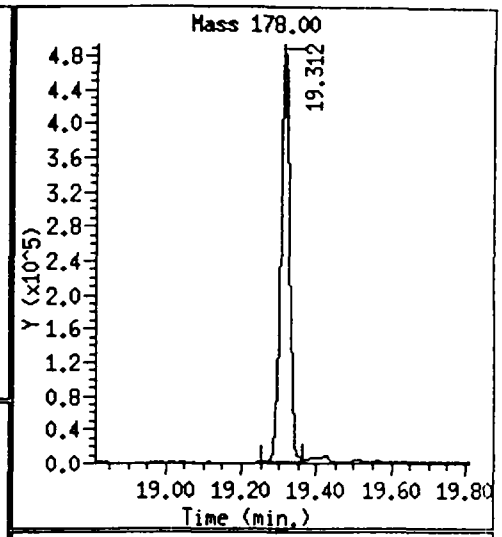
73 Phenanthrene

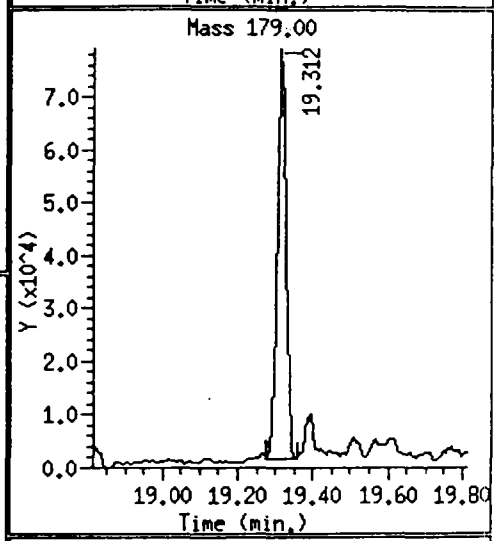


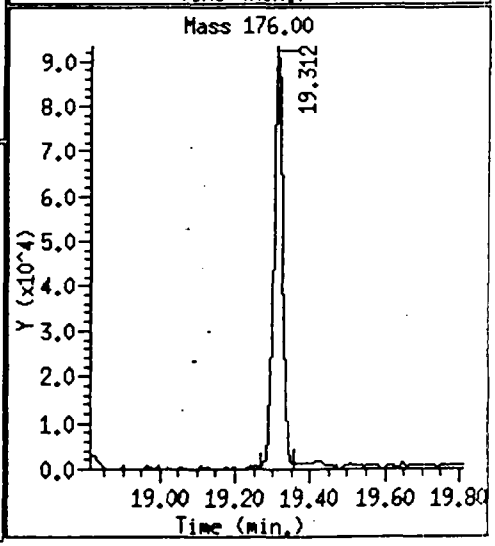












Data File: /chem/V.i/06-09-98.b/VH290.d

Date : 09-JUN-1998 16:16

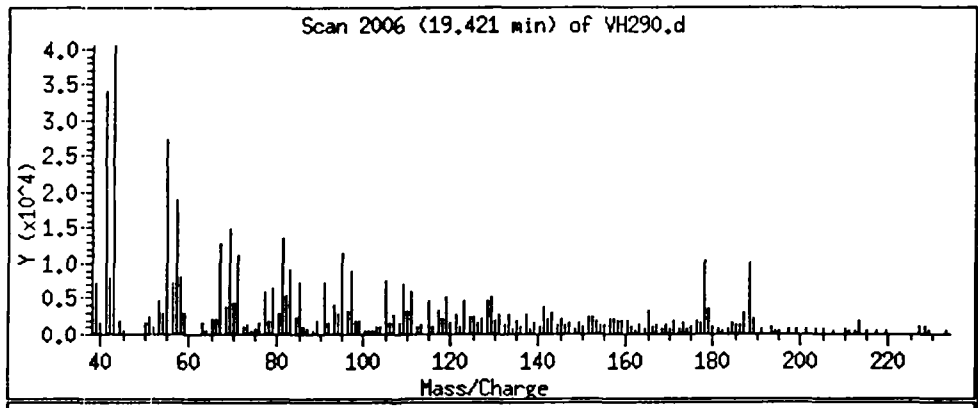
Instrument : V.i

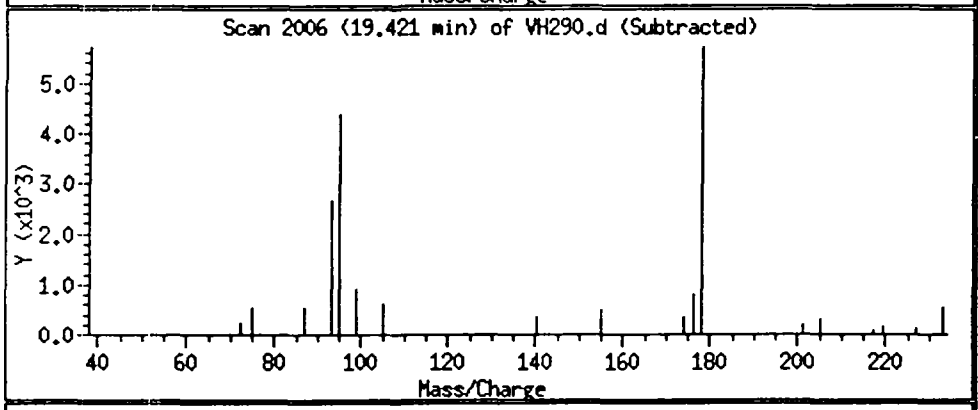
Sample ID : SITE#4 5-27-98 24HR

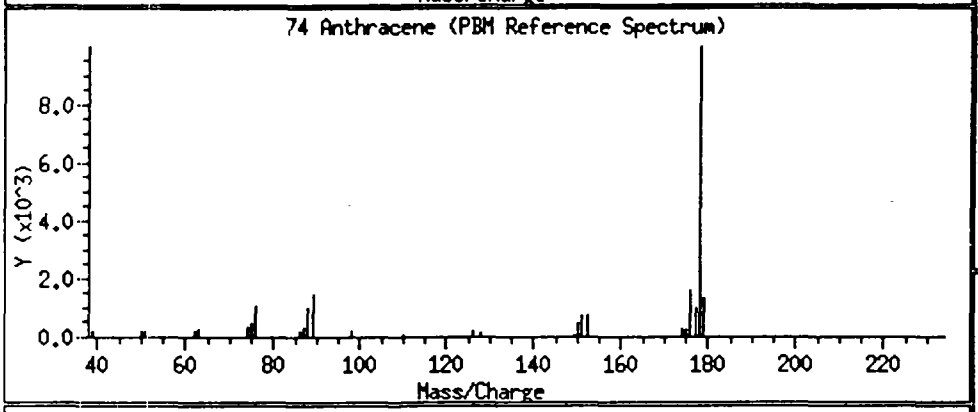
Column phase : DB-5 Column diameter : 0.32

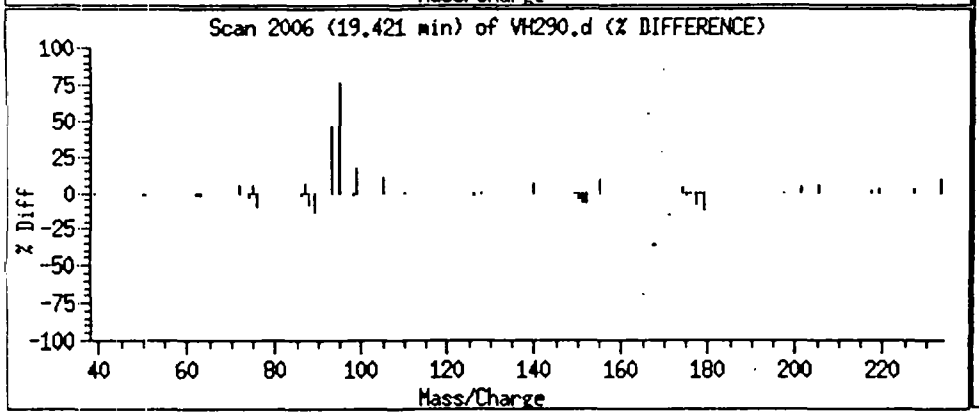
Volume Injected (uL) : 1.0

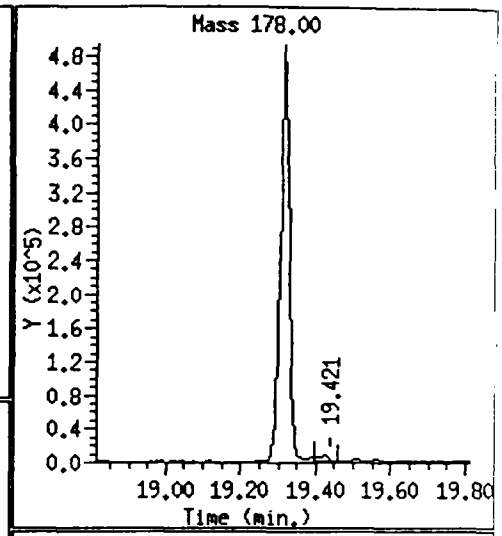
74 Anthracene

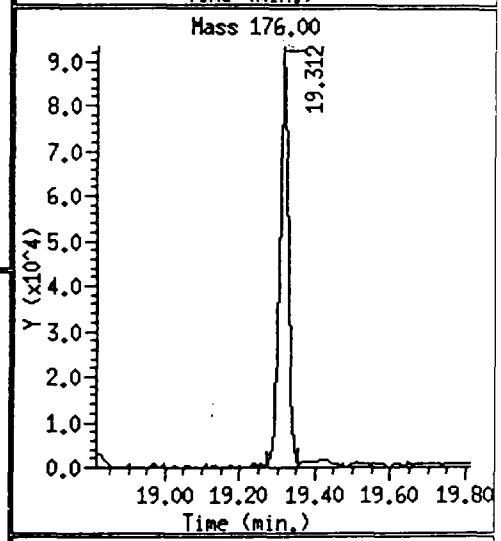


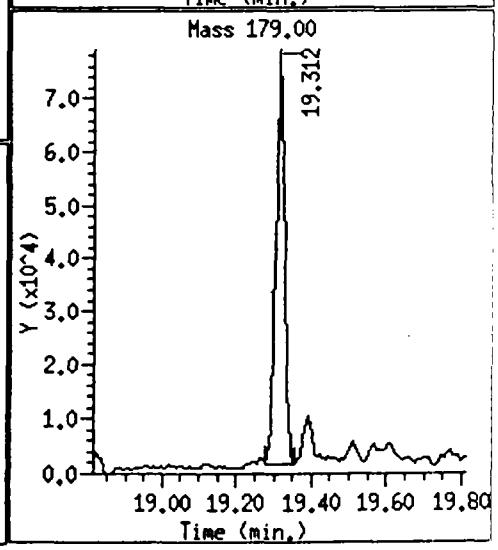












Data File: /chem/V.i/06-09-98.b/VH290.d

Date : 09-JUN-1998 16:16

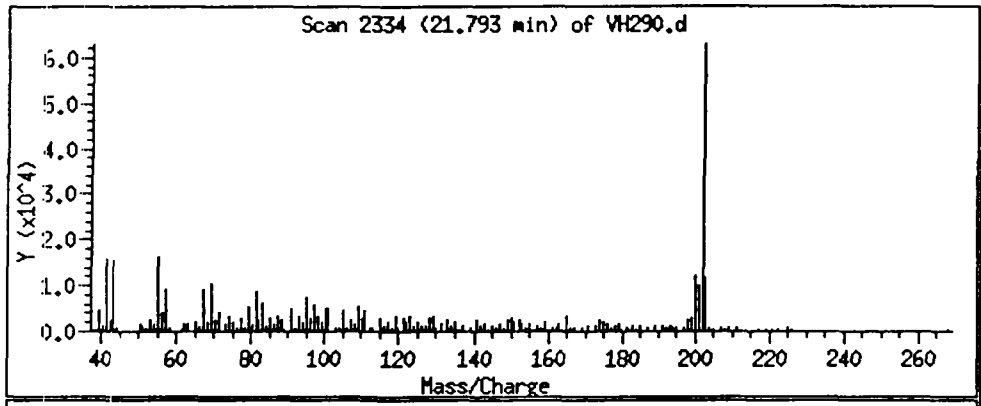
Instrument : V.i

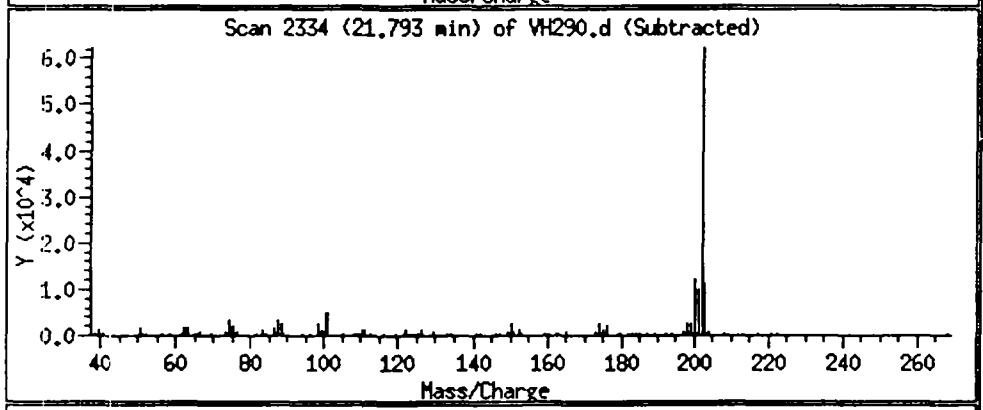
Sample ID : SITE#4 5-27-98 24HR

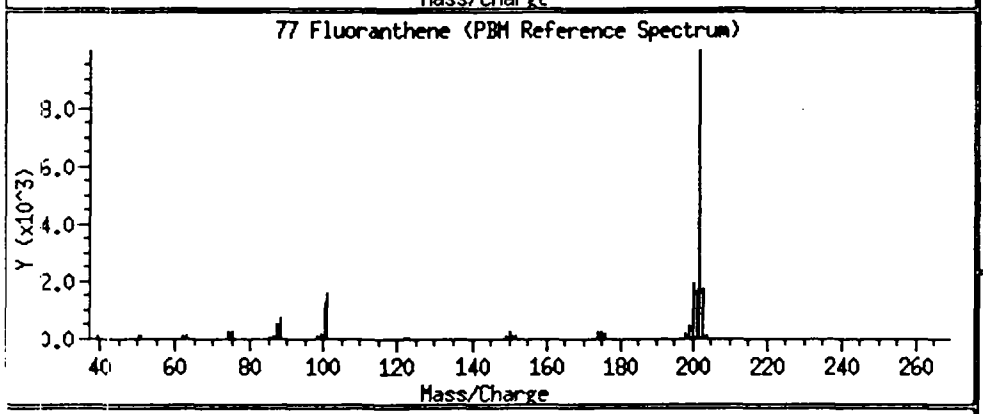
Column phase : DB-5 Column diameter : 0.32

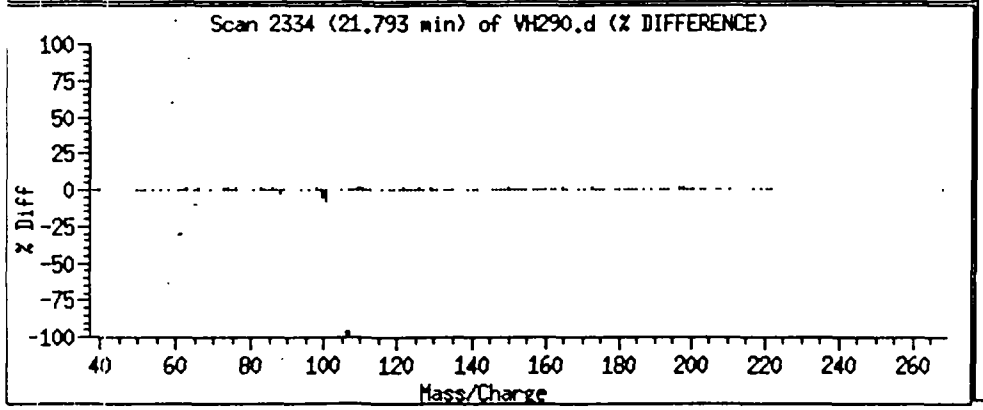
Volume Injected (uL) : 1.0

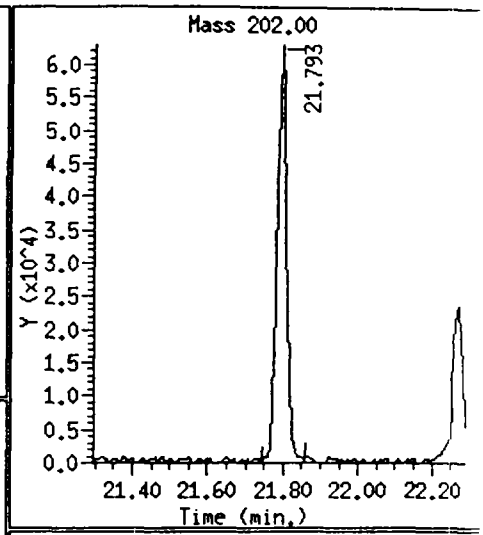
77 Fluoranthene

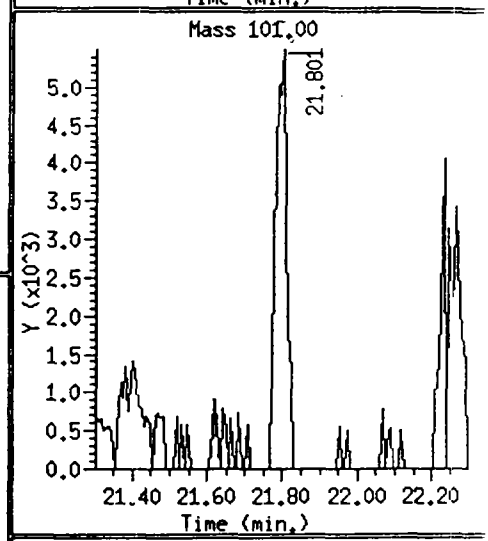


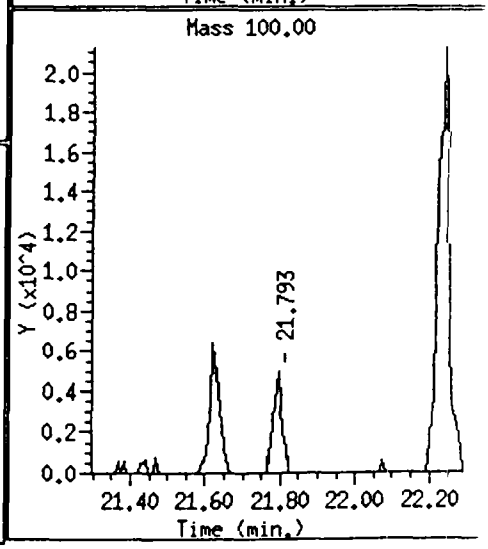












Derenzo and Associates, Inc.

Project Number: 45869 | Method 8270A PUF
Sample File: VH289 | Sample ID: SITE#4 5-28-98 8hour

Client Project: Hull & Assoc | Date Received: 06/02/98 | Response File: VH284
TLI ID: 209-67-4,8 | Date Extracted: 06/04/98
Date Analyzed : 06/09/98

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	1.51	BJ	12.56		10
Acenaphthene-d_{10}		IS 3	16.16		
Fluorene	1.02	J	17.29		10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene	6.86	BJ	19.31		10
Anthracene	0.23	J	19.42		10
Fluoranthene	1.66	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.60	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	100.76	19.38	4	101
Pyrene-d_{10}	126.96	22.24	5	127
Terphenyl-d_{14}	114.73	22.60	5	115

Reviewed by PaB Date 6/12/98

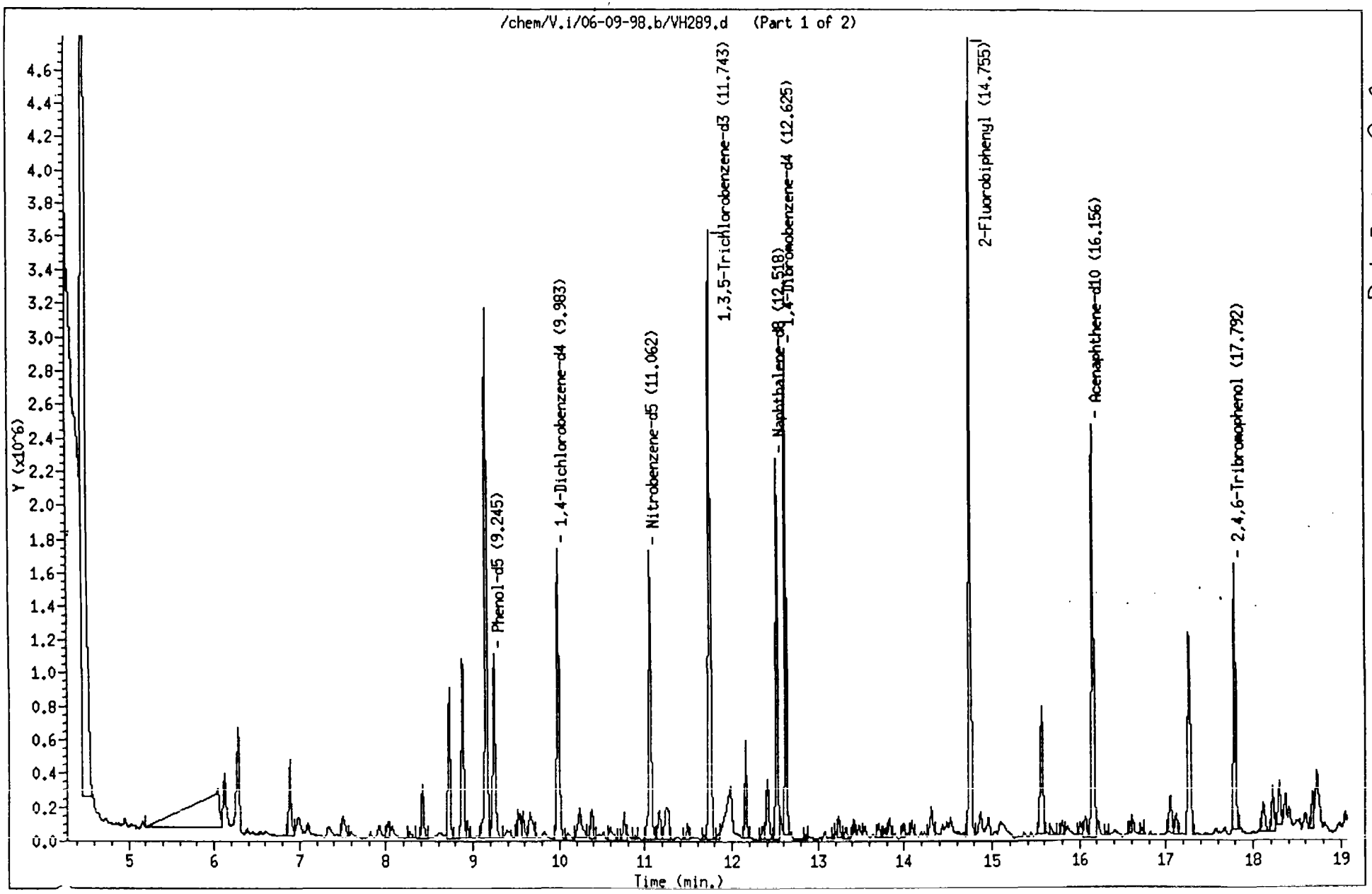
NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit
IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Data File: /chem/V.i/06-09-98.b/VH289.d
Date : 09-JUN-1998 15:29
Instrument : V.i
Sample ID : SITE#4 5-28-98 8HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

148

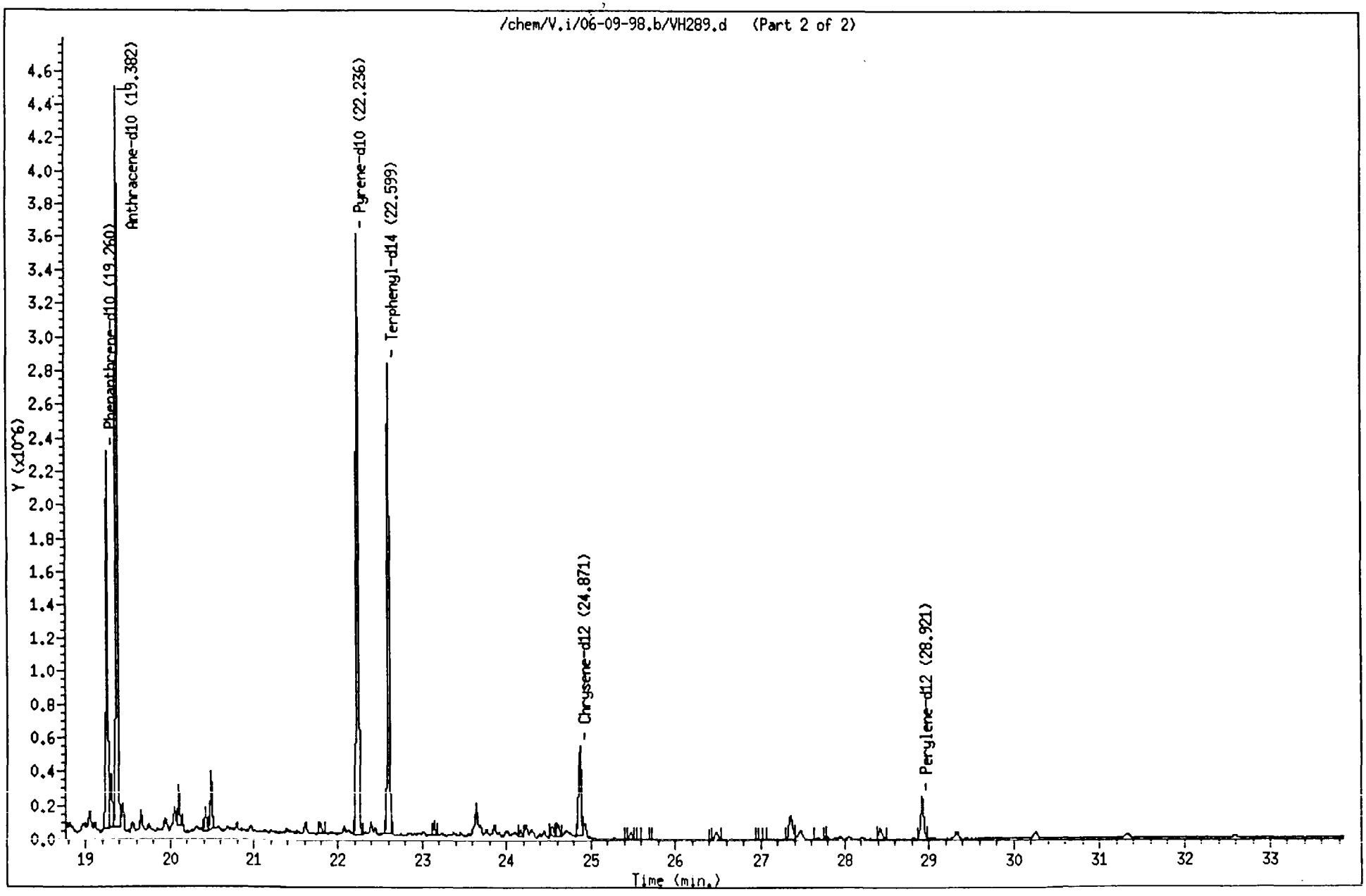


Data Review: [illegible]
Date: 6/12/98

Data File: /chem/V.i/06-09-98.b/VH289.d
Date : 09-JUN-1998 15:29
Instrument : V.i
Sample ID : SITE#4 5-28-98 8HR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-09-98.b/VH289.d
Lab. Id. : 45869
Inj Date : 09-JUN-1998 15:29 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SITE#4 5-28-98 8HOUR
Misc Info : 209-67-4,8
Comment :
Method : /chem/V.i/06-09-98.b/cclist.m
Meth Date : 09-Jun-1998 12:27 target
Cal Date : 09-JUN-1998 10:31 Cal File: VH284.d
Als bottle: 5
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.983(1.000)	420237	40.00	
*	2 Naphthalene-d8	136.00	12.518(1.000)	1520178	40.00	
*	3 Acenaphthene-d10	164.00	16.156(1.000)	825868	40.00	
*	4 Phenanthrene-d10	188.00	19.260(1.000)	1318048	40.00	
*	5 Chrysene-d12	240.00	24.871(1.000)	323061	40.00	
*	6 Perylene-d12	264.00	28.921(1.000)	251617	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.245(0.926)	489259	37.75	37.75
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.062(0.884)	794861	62.15	62.15
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.743(0.938)	985985	71.58	71.58
$	13 1,4-Dibromobenzene-d4	240.00	12.625(1.265)	772777	73.23	73.23
$	14 2-Fluorobiphenyl	172.00	14.755(0.913)	2080134	88.52	88.52
$	15 2,4,6-Tribromophenol	329.80	17.792(1.101)	236490	61.74	61.74
$	16 Anthracene-d10	188.00	19.382(1.006)	2729217	100.73	100.73
$	17 Pyrene-d10	212.00	22.236(0.894)	2109240	126.96	126.96(R)
$	18 Terphenyl-d14	244.00	22.599(0.909)	1157348	114.71	114.71
	42 Naphthalene	128.00	12.560(1.003)	56789	1.51	1.51(a)
	46 2-Methylnaphthalene	142.00	13.987(1.117)	26777	1.16	1.16(a)
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	154.00	16.221(1.004)	12423	0.62	0.62(a)
	65 Fluorene	166.00	17.294(1.070)	28737	1.02	1.02(a)
	73 Phenanthrene	178.00	19.310(1.003)	203125	6.86	6.86(a)
	74 Anthracene	178.00	19.42 ~~19.310(1.003)~~	~~203125~~ 6523	~~7.31~~	~~7.31(a)~~ M PaB
	77 Fluoranthene	202.00	21.788(1.131)	48469	1.66	1.66(a)
	78 Pyrene	202.00	~~21.788(0.876)~~	~~34295~~	~~1.58~~	~~1.58(a)~~ NA PaB
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PaB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount Below Limit Of Quantitation(BLOQ).
R - Spike/Surrogate failed recovery limits.

Data File: /chem/V.i/06-09-98.b/VH289.d

Date : 09-JUN-1998 15:29

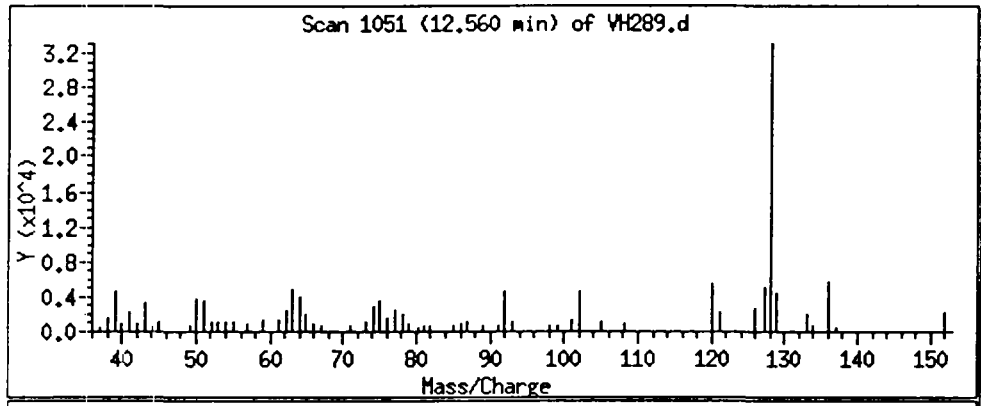
Instrument : V.i

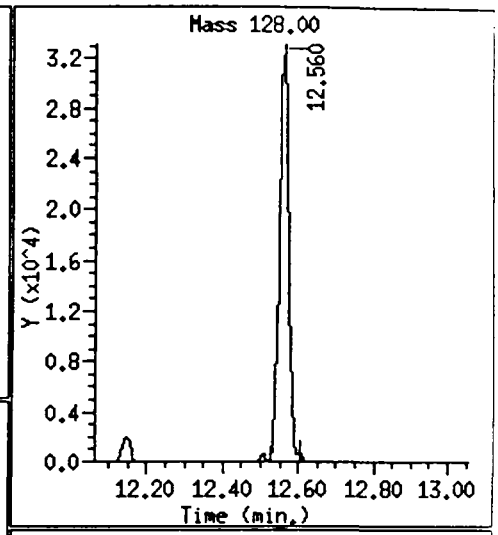
Sample ID : SITE#4 5-28-98 8HR

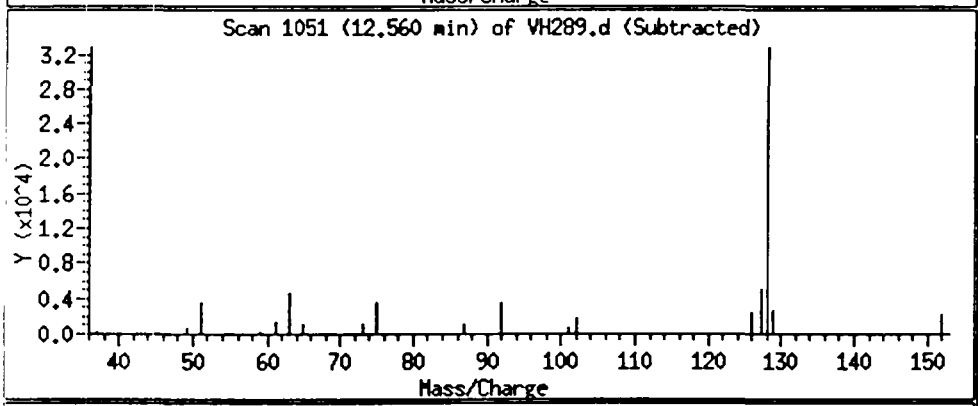
Column phase : DB-5 Column diameter : 0.32

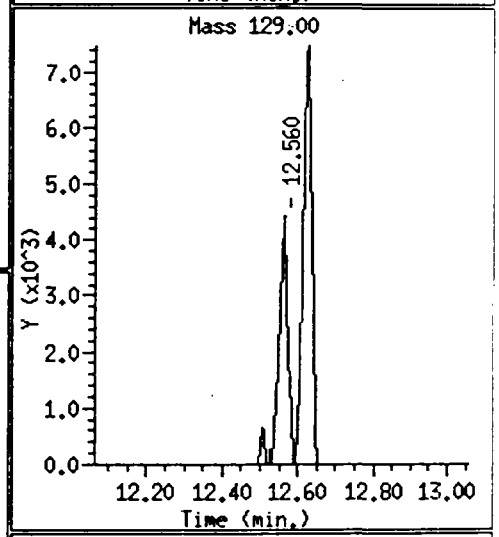
Volume Injected (uL) : 1.0

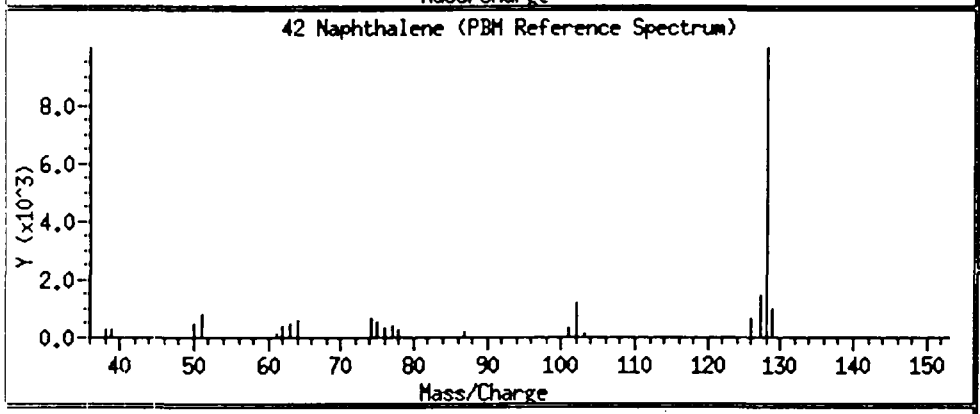
42 Naphthalene

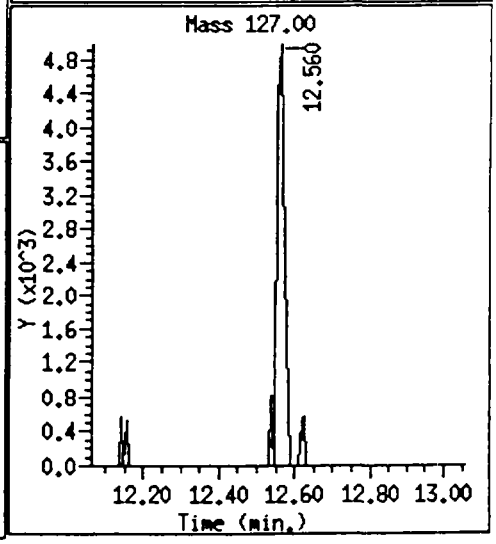


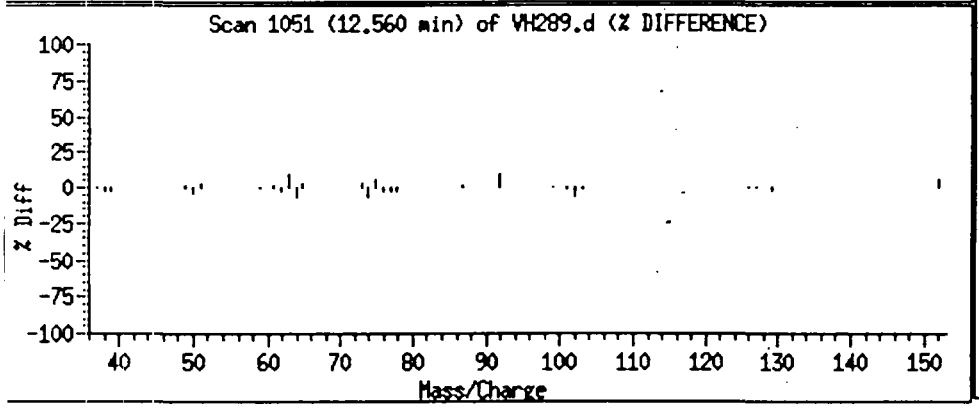












Data File: /chem/V.i/06-09-98.b/VH289.d

Date : 09-JUN-1998 15:29

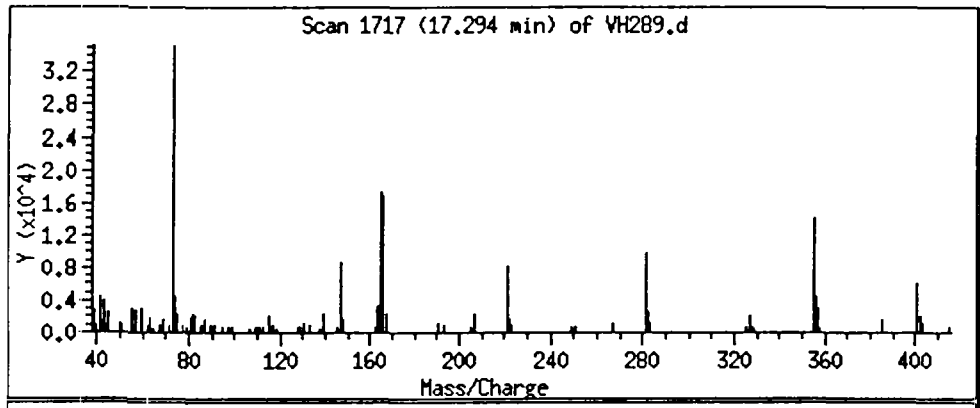
Instrument : V.i

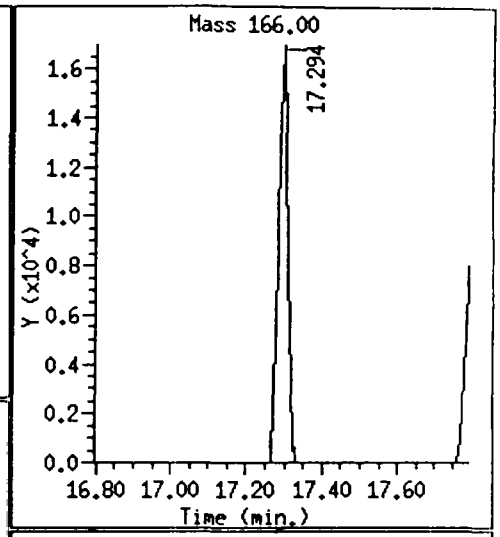
Sample ID : SITE#4 5-28-98 8HR

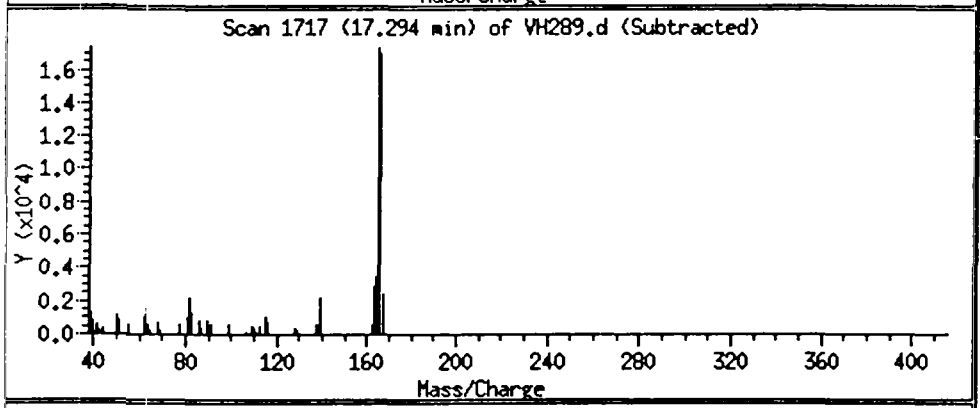
Column phase : DB-5 Column diameter : 0.32

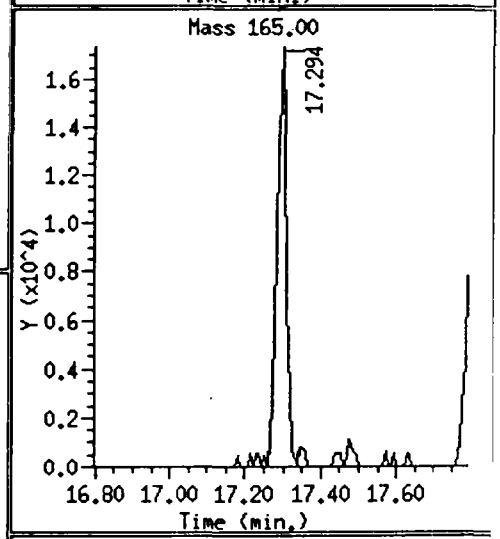
Volume Injected (uL) : 1.0

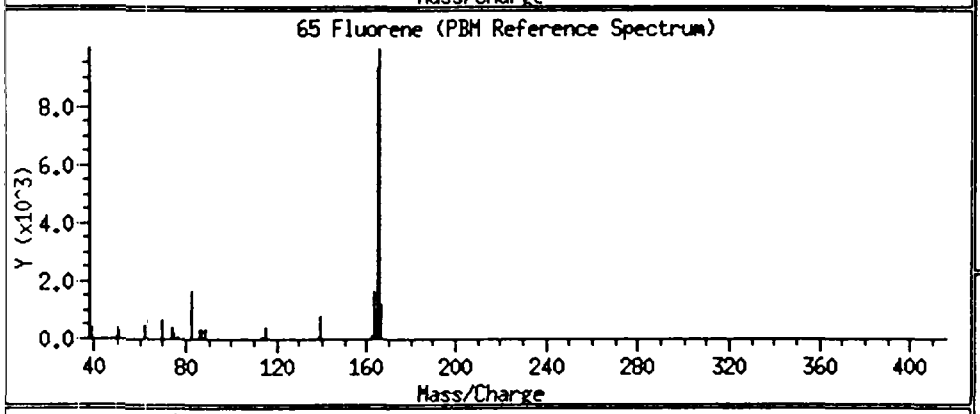
65 Fluorene

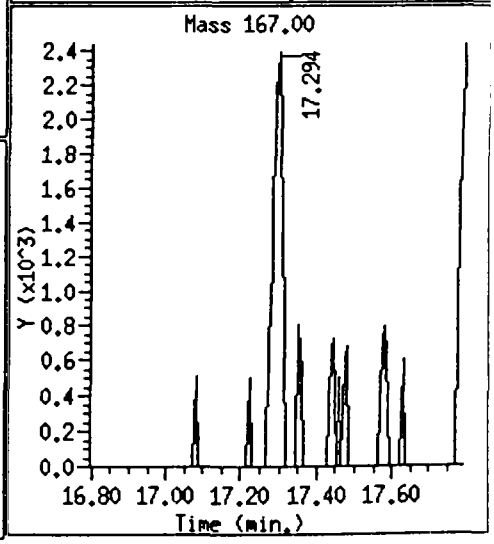


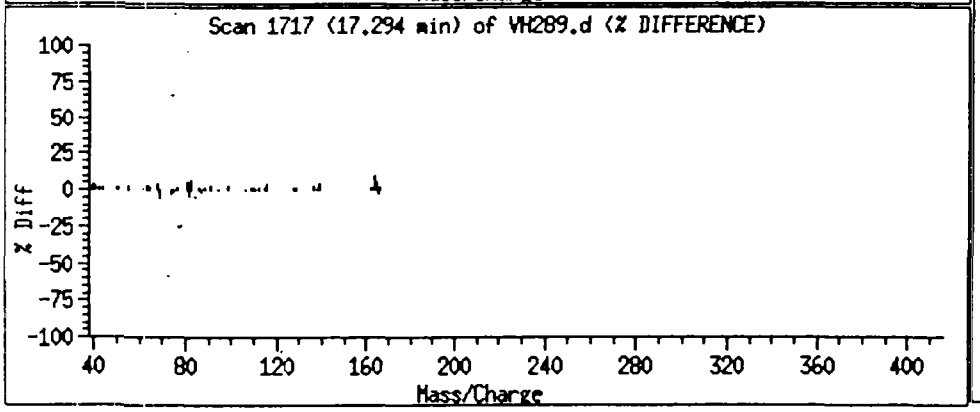












Data File: /chem/V.i/06-09-98.b/VH289.d

Date : 09-JUN-1998 15:29

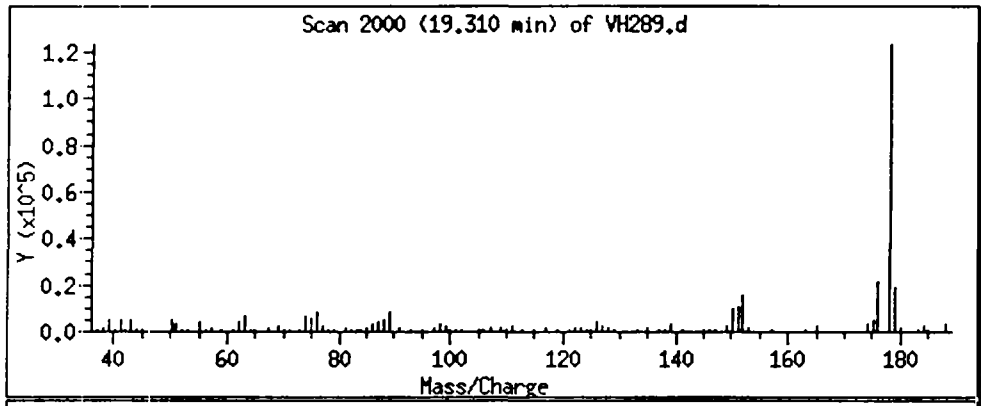
Instrument : V.i

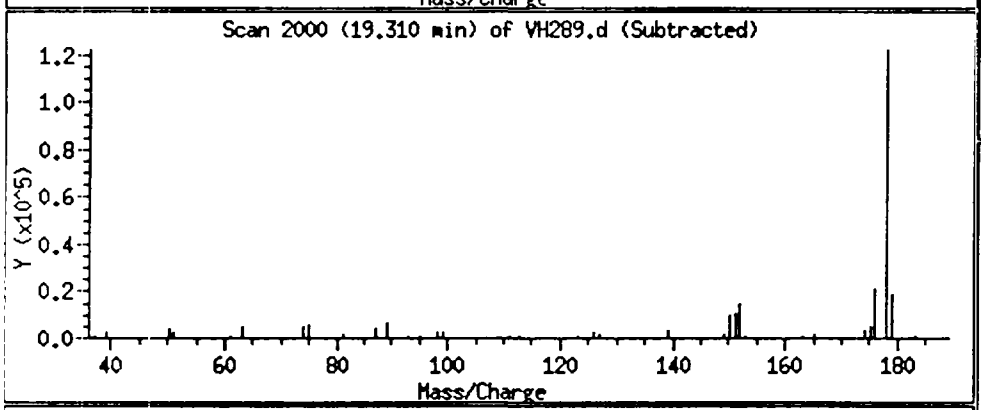
Sample ID : SITE#4 5-28-98 8HR

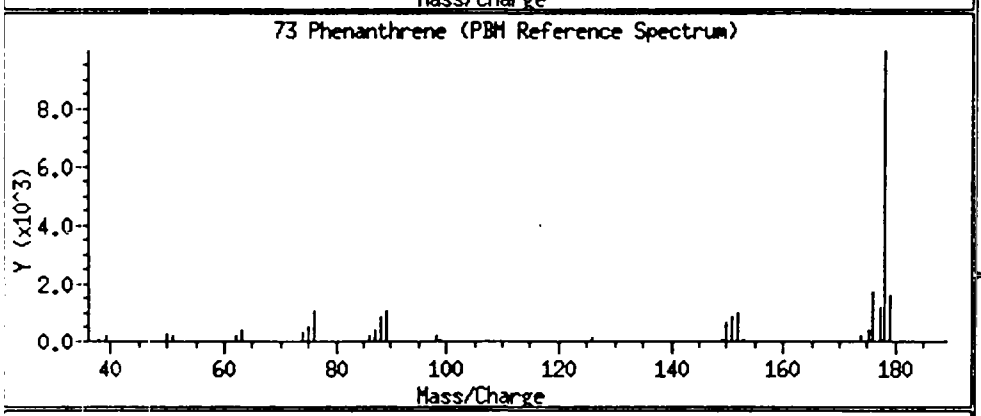
Column phase : DB-5 Column diameter : 0.32

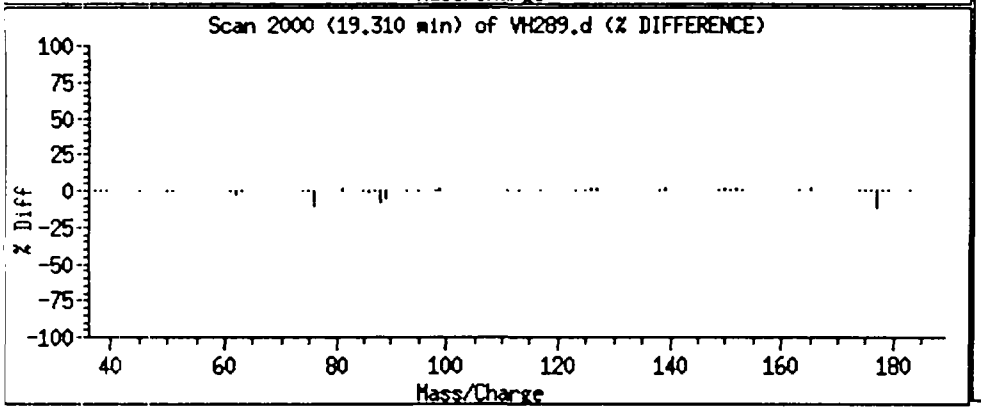
Volume Injected (uL) : 1.0

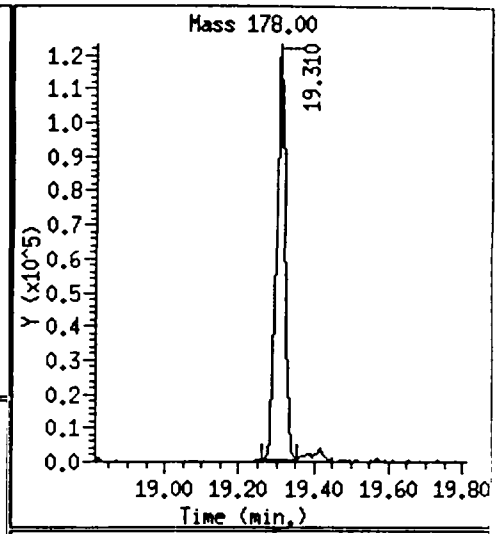
73 Phenanthrene

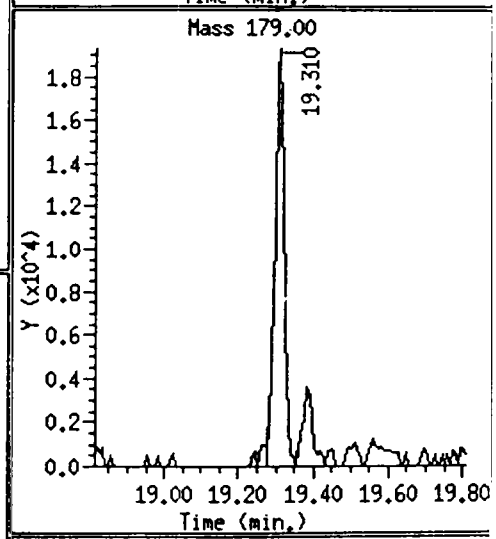


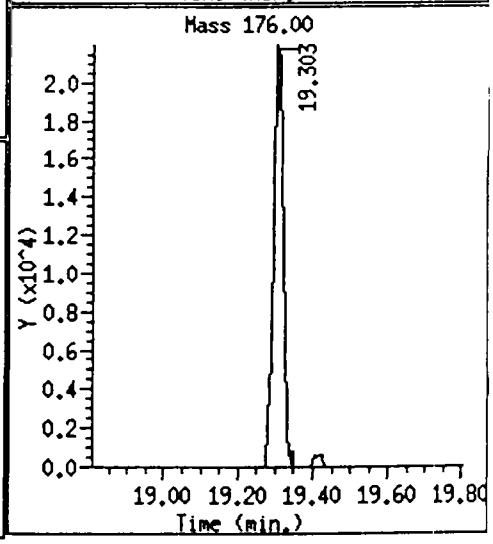












Data File: /chem/V.i/06-09-98.b/VH289.d

Date : 09-JUN-1998 15:29

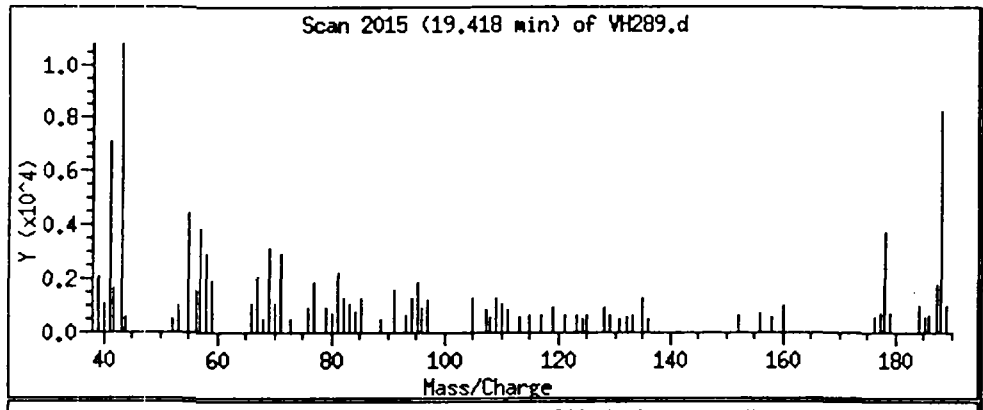
Instrument : V.i

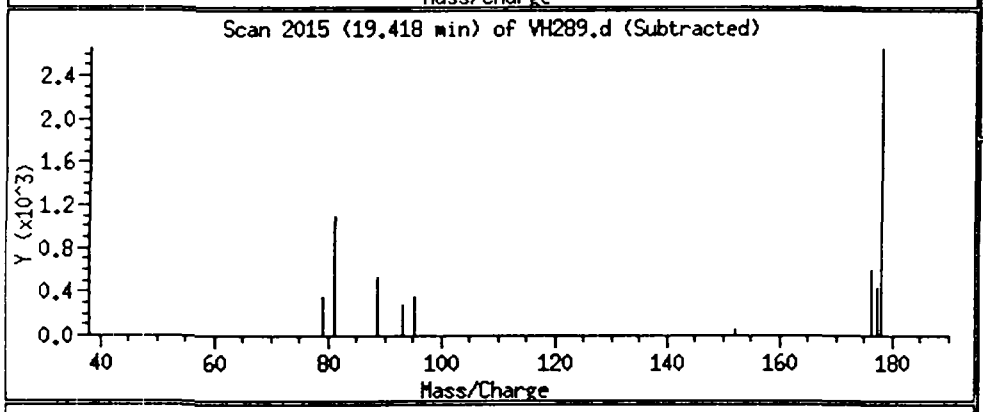
Sample ID : SITE#4 5-28-98 8HR

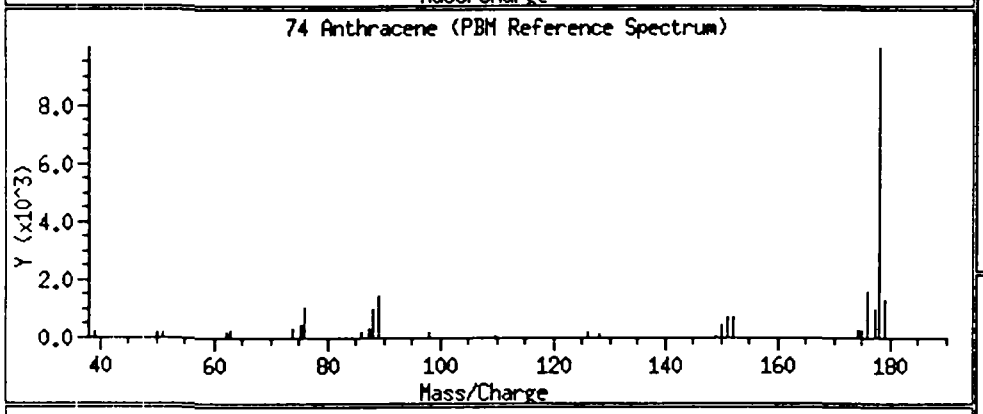
Column phase : DB-5 Column diameter : 0.32

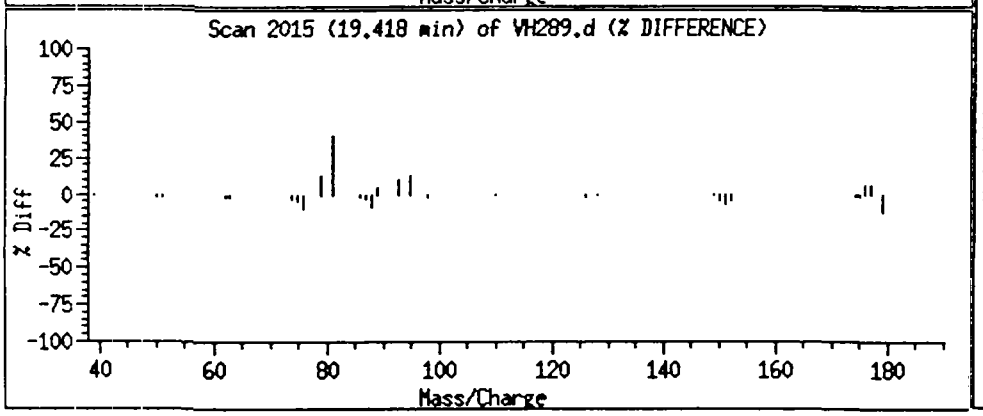
Volume Injected (uL) : 1.0

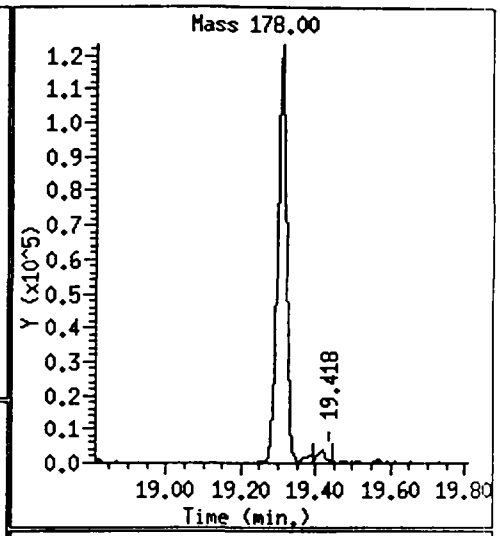
74 Anthracene

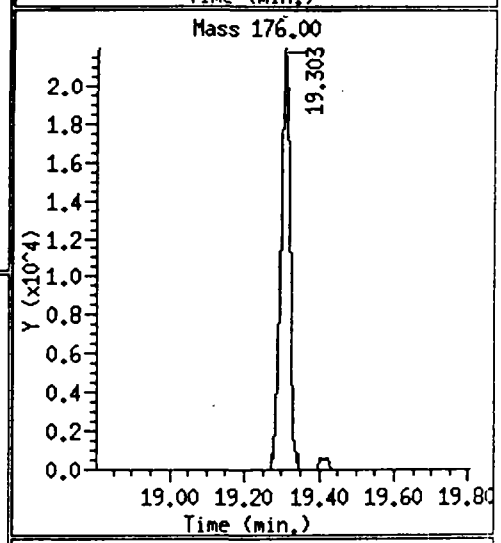


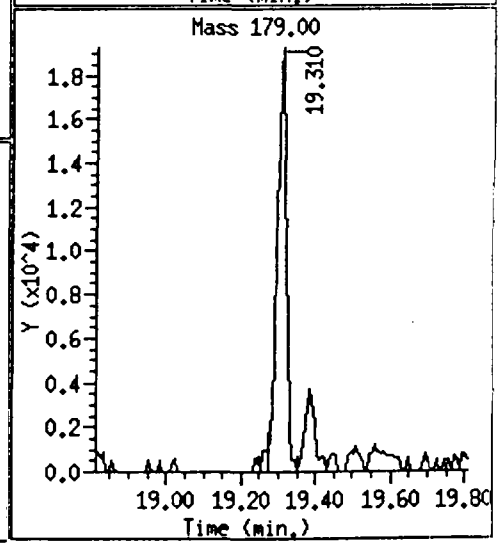












Data File: /chem/V.i/06-09-98.b/VH289.d

Date : 09-JUN-1998 15:29

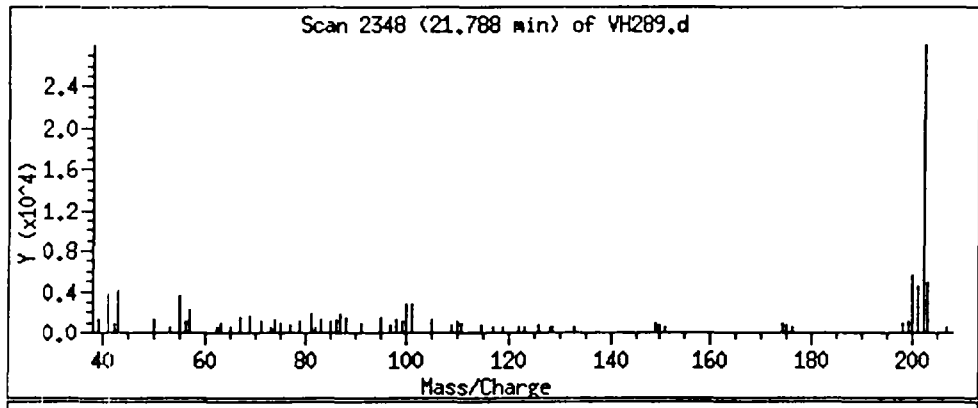
Instrument : V.i

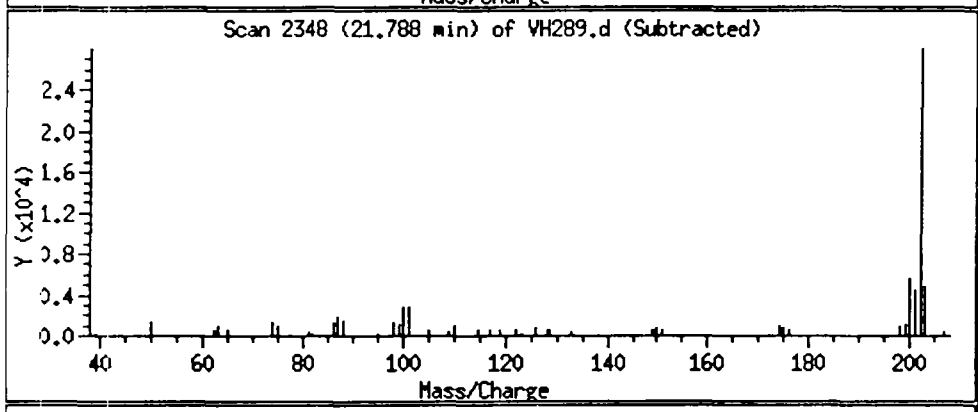
Sample ID : SITE#4 5-28-98 8HR

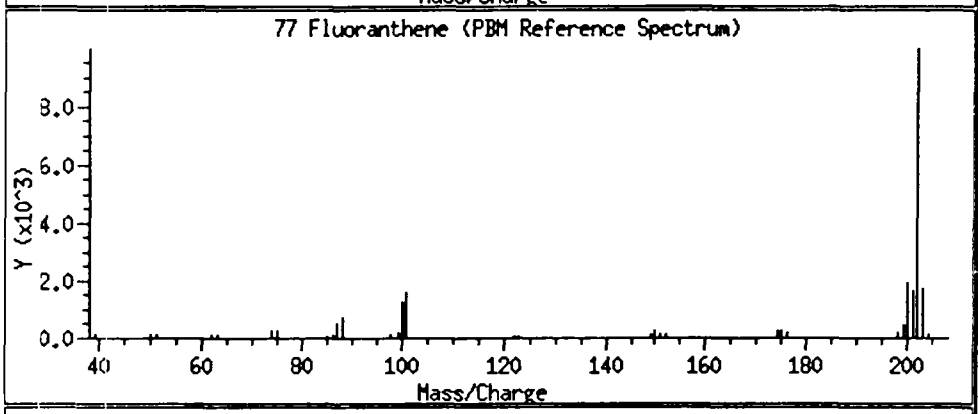
Column phase : DB-5 Column diameter : 0.32

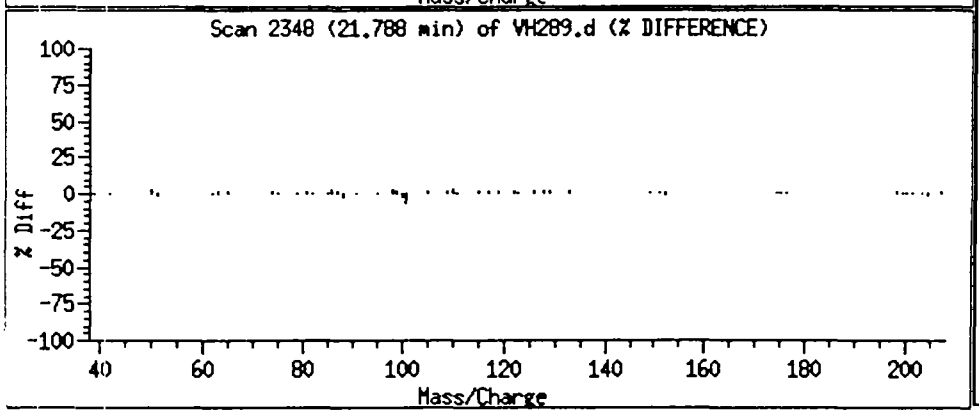
Volume Injected (uL) : 1.0

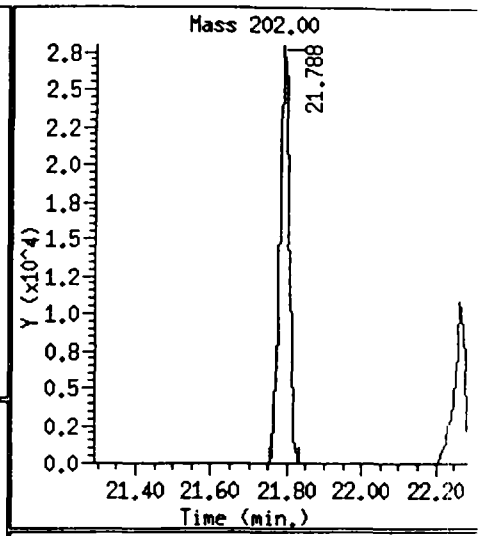
77 Fluoranthene

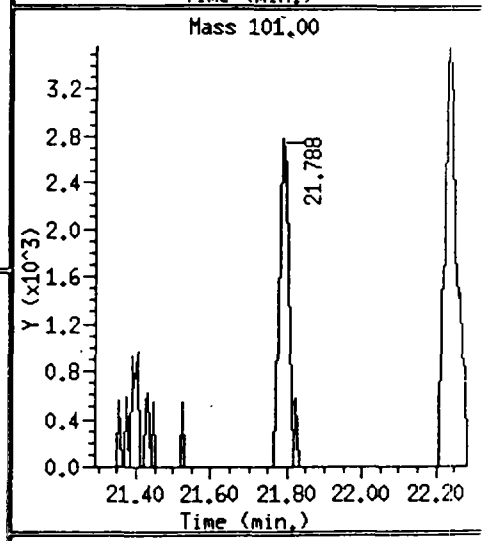


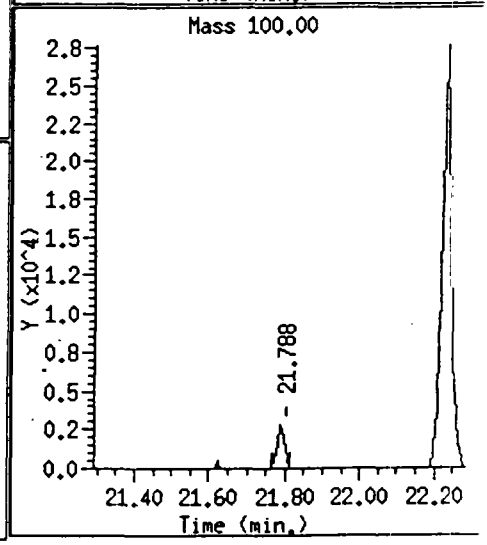












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH288

Method 8270A PUF
Sample ID: SITE#4 5-29-98 24hour

Client Project: Hull & Assoc
TLI ID: 209-67-20,24

Date Received: 06/02/98
Date Extracted: 06/04/98
Date Analyzed : 06/09/98

Response File: VH284

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	14.80	B	12.56		10
Acenaphthene-d_{10}		IS 3	16.16		
Fluorene	5.75	J	17.30		10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene	21.28	B	19.31		10
Anthracene	0.71	J	19.42		10
Fluoranthene	3.36	J	21.79		10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.72	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	91.69	19.38	4	92
Pyrene-d_{10}	122.04	22.24	5	122
Terphenyl-d_{14}	107.07	22.60	5	107

Reviewed by PAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

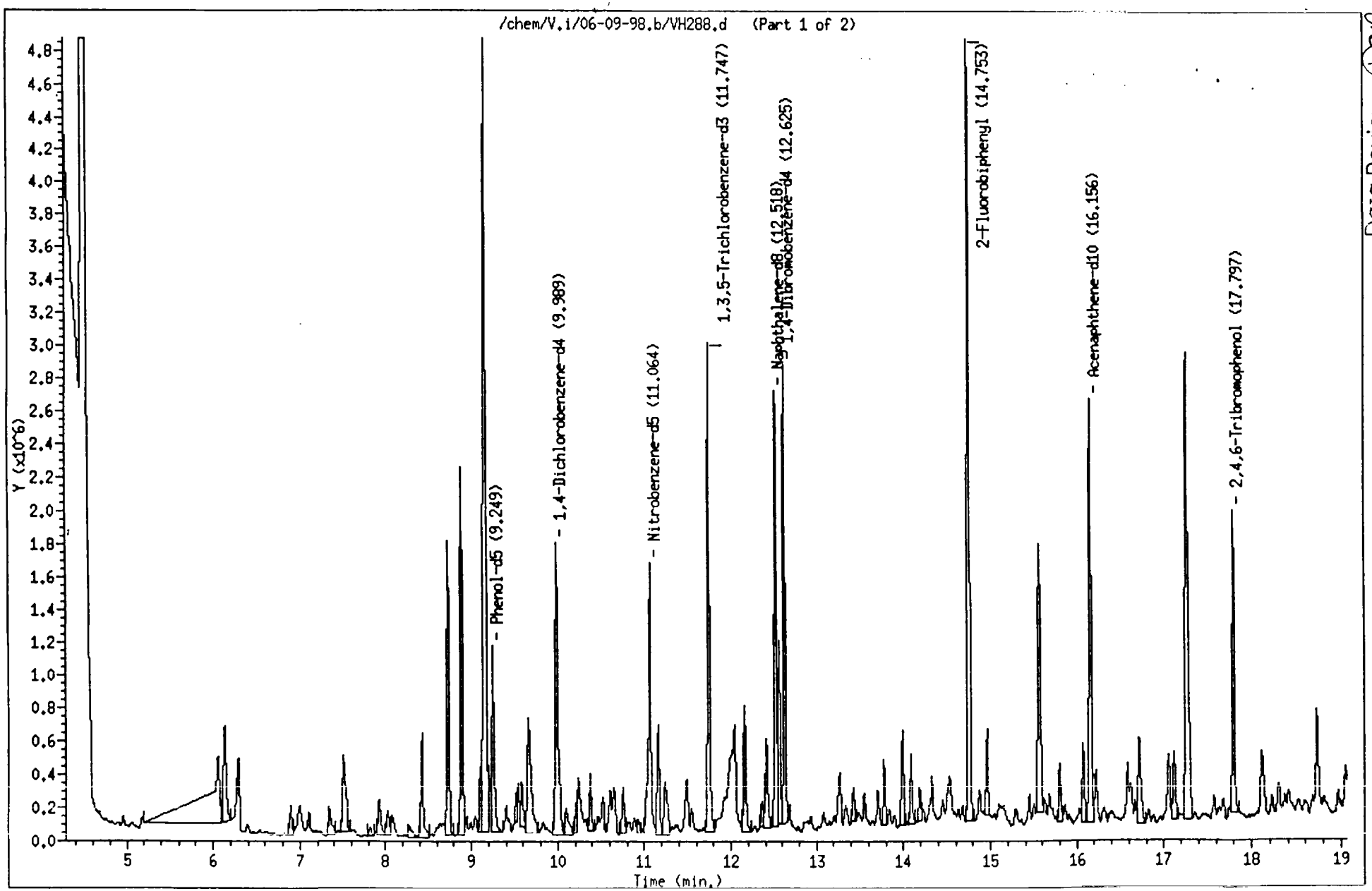
IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Data File: /chem/V.i/06-09-98.b/VH288.d
Date : 09-JUN-1998 14:42
Instrument : V.i
Sample ID : 24 HOUR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

158

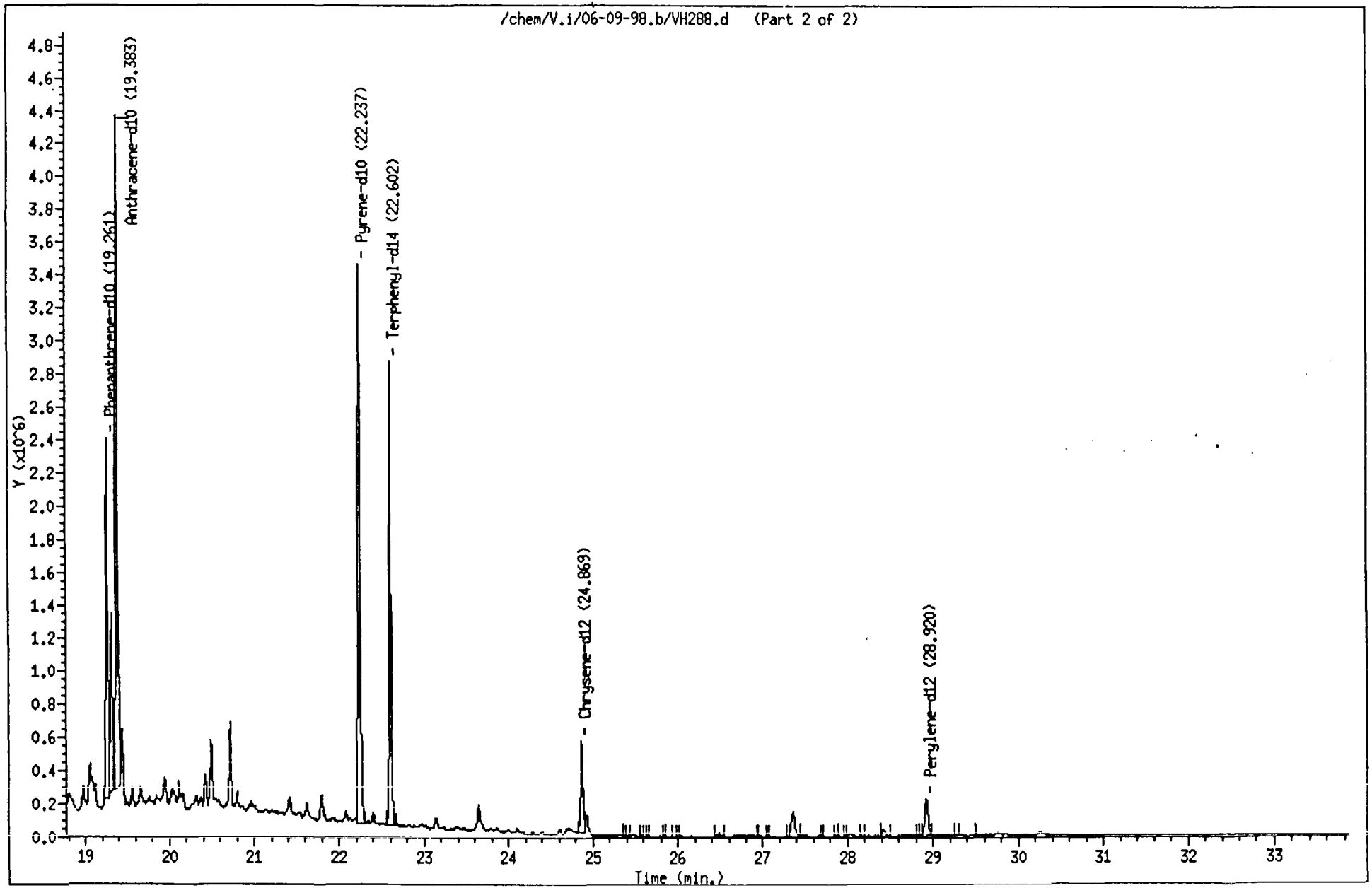


Data Review: [illegible]
Date: 6/12/98

Data File: /chem/V.i/06-09-98.b/VH288.d
Date : 09-JUN-1998 14:42
Instrument : V.i
Sample ID : 24 HOUR
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories
of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-09-98.b/VH288.d
Lab. Id. : 45869
Inj Date : 09-JUN-1998 14:42 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : 24 HOUR
Misc Info : 209-67-20,24
Comment :
Method : /chem/V.i/06-09-98.b/cclist.m
Meth Date : 09-Jun-1998 12:27 target
Cal Date : 09-JUN-1998 10:31 Cal File: VH284.d
Als bottle: 4
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

		Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1	1,4-Dichlorobenzene-d4	152.00	9.989(1.000)	433955	40.00	
*	2	Naphthalene-d8	136.00	12.518(1.000)	1575239	40.00	
*	3	Acenaphthene-d10	164.00	16.156(1.000)	827765	40.00	
*	4	Phenanthrene-d10	188.00	19.261(1.000)	1354072	40.00	
*	5	Chrysene-d12	240.00	24.869(1.000)	329149	40.00	
*	6	Perylene-d12	264.00	28.920(1.000)	234295	40.00	
$	7	2-Fluorophenol	Compound Not Detected.				
$	8	Phenol-d5	99.00	9.249(0.926)	427528	31.95	31.95
$	9	2-Chlorophenol-d4	Compound Not Detected.				
$	10	1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11	Nitrobenzene-d5	82.00	11.064(0.884)	746807	56.35	56.35
$	12	1,3,5-Trichlorobenzene-d3	185.00	11.747(0.938)	845692	59.25	59.25
$	13	1,4-Dibromobenzene-d4	240.00	12.625(1.264)	703409	64.55	64.55
$	14	2-Fluorobiphenyl	172.00	14.753(0.913)	2062908	87.58	87.58
$	15	2,4,6-Tribromophenol	329.80	17.797(1.102)	270512	70.46	70.46
$	16	Anthracene-d10	188.00	19.383(1.006)	2551382	91.66	91.66
$	17	Pyrene-d10	212.00	22.237(0.894)	2065647	122.03	122.03(R)
$	18	Terphenyl-d14	244.00	22.602(0.909)	1100409	107.05	107.05
	42	Naphthalene	128.00	12.561(1.003)	577492	14.80	14.80
	46	2-Methylnaphthalene	142.00	13.988(1.117)	201928	8.43	8.43(a)
	50	2-Chloronaphthalene	Compound Not Detected.				
	56	Acenaphthylene	152.00	~~16.134(0.999)~~	~~7203~~	~~0.21~~	~~0.21(a)~~ NA PaB
	58	Acenaphthene	154.00	16.220(1.004)	76530	3.81	3.81(a)
	65	Fluorene	166.00	17.295(1.071)	161998	5.75	5.75(a)
	73	Phenanthrene	178.00	19.311(1.003)	647694	21.28	21.28
	74	Anthracene	178.00	R.42 ~~19.311(1.003)~~	~~647697~~ 26407	~~22.68~~	~~22.68~~ NA PaB
	77	Fluoranthene	202.00	21.794(1.132)	100763	3.36	3.36(a)
	78	Pyrene	202.00	~~21.794(0.876)~~	~~74984~~	~~3.39~~	~~3.39(a)~~ NA PaB
	83	Benzo(a)anthracene	Compound Not Detected.				
	84	Chrysene	Compound Not Detected.				
	86	Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PaB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).
R - Spike/Surrogate failed recovery limits.

Data File: /chem/V.i/06-09-98.b/VH288.d

Date : 09-JUN-1998 14:42

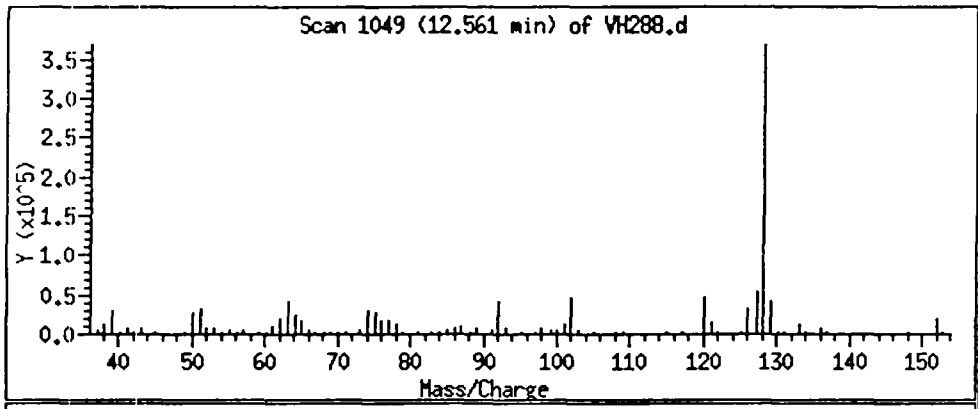
Instrument : V.i

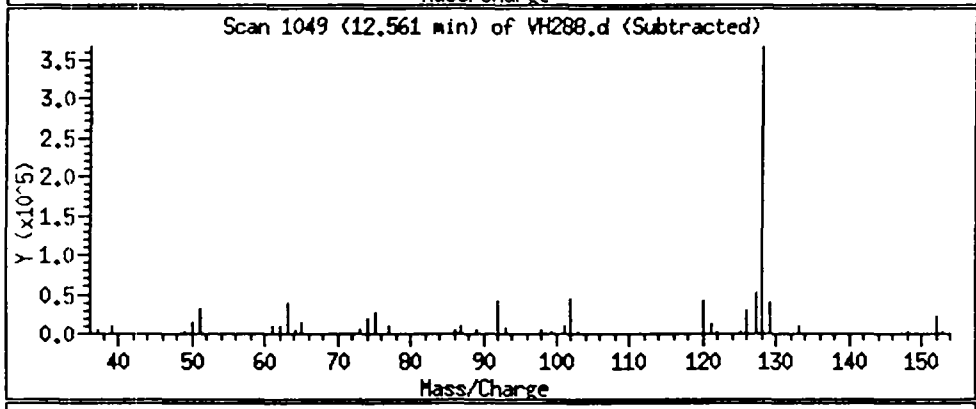
Sample ID : 24 HOUR

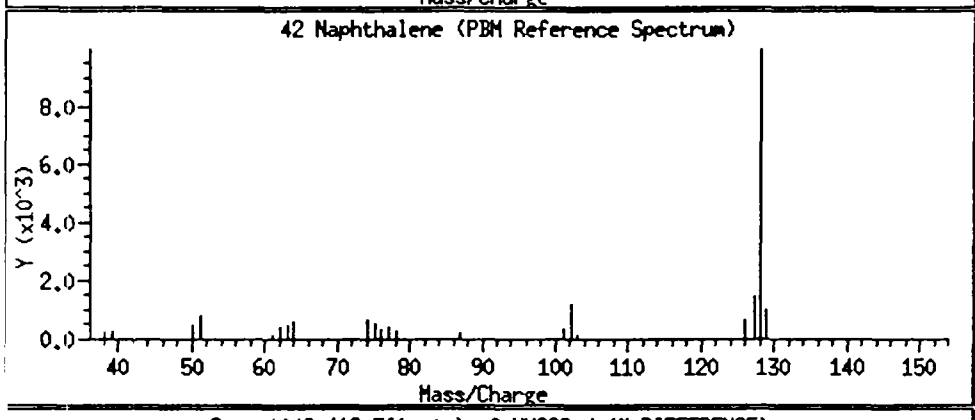
Column phase : DB-5 Column diameter : 0.32

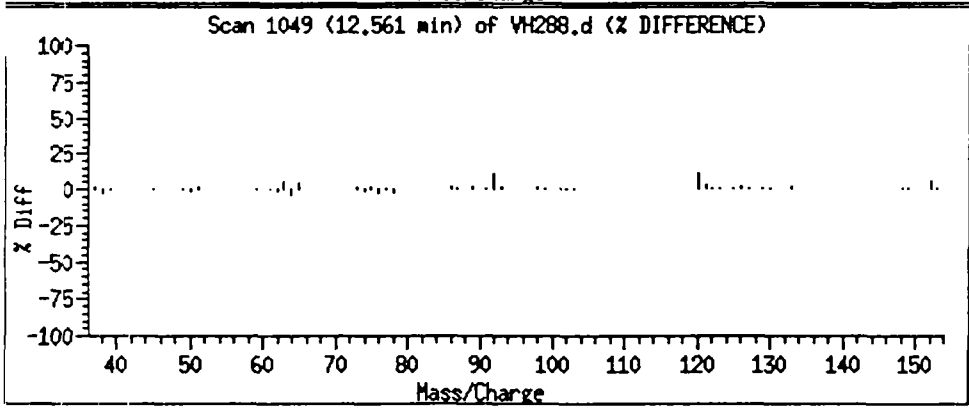
Volume Injected (uL) : 1.0

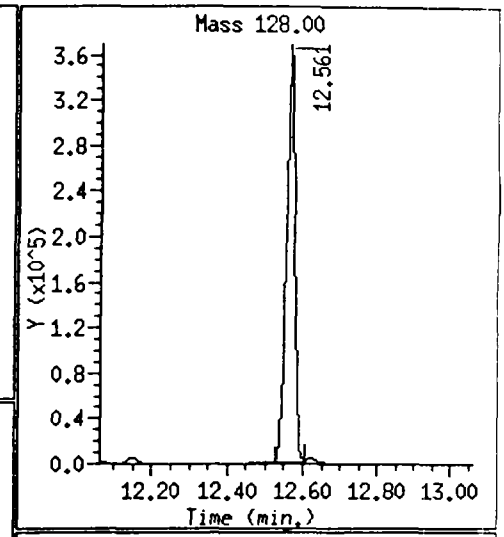
42 Naphthalene

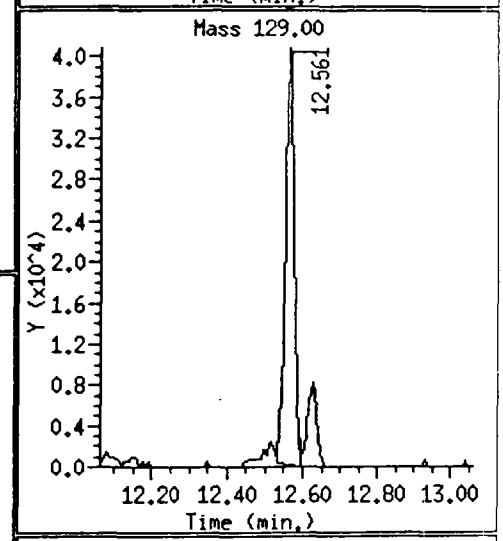


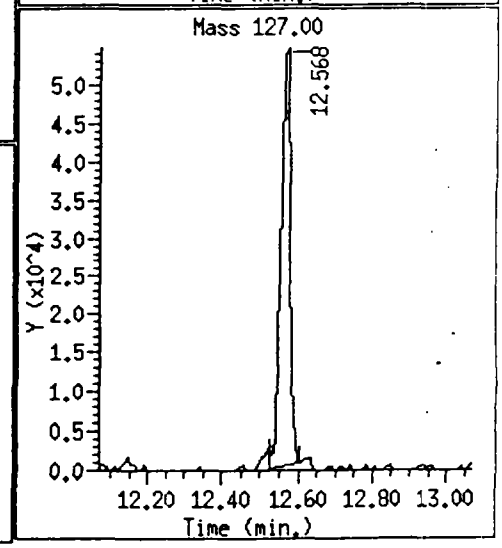












Data File: /chem/V.i/06-09-98.b/VH288.d

Date : 09-JUN-1998 14:42

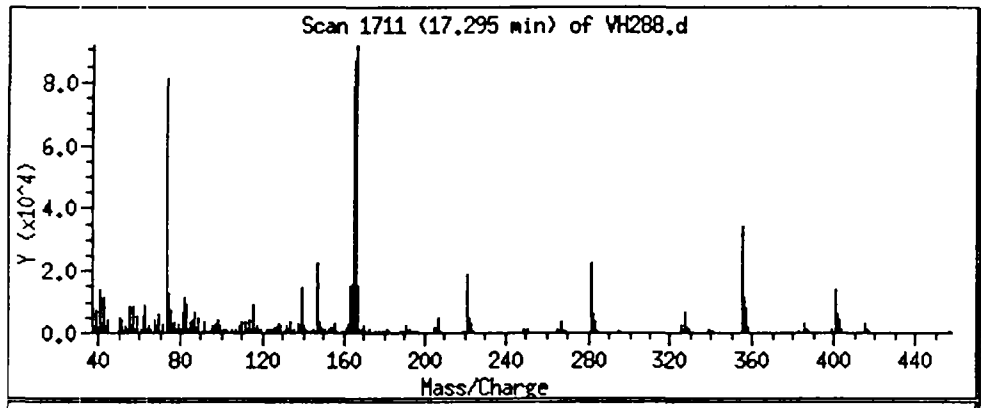
Instrument : V.i

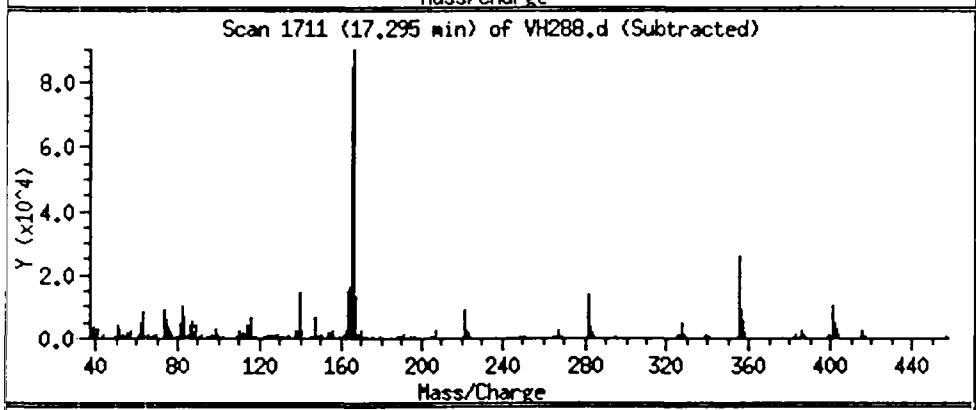
Sample ID : 24 HOUR

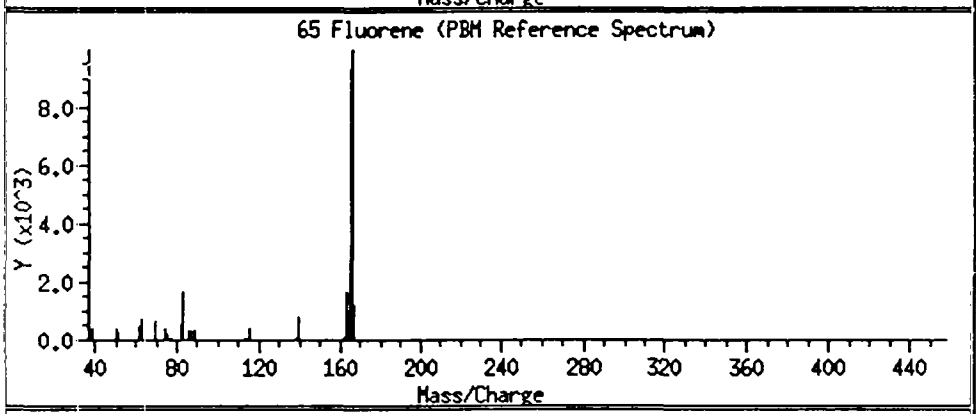
Column phase : DB-5 Column diameter : 0.32

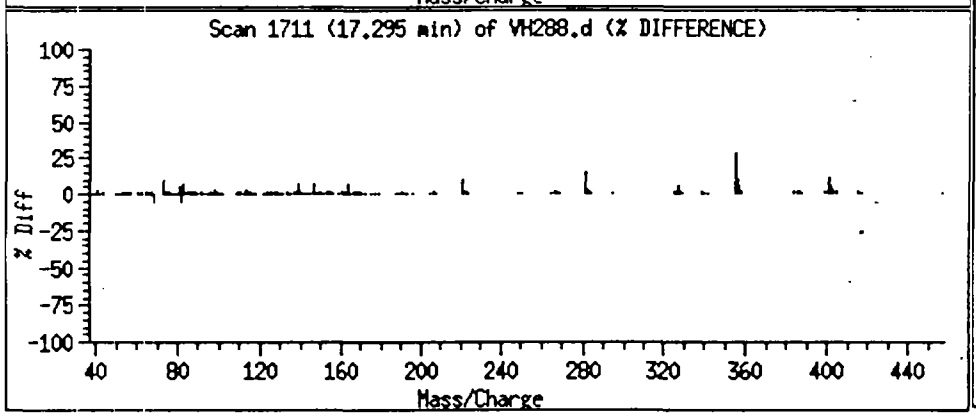
Volume Injected (uL) : 1.0

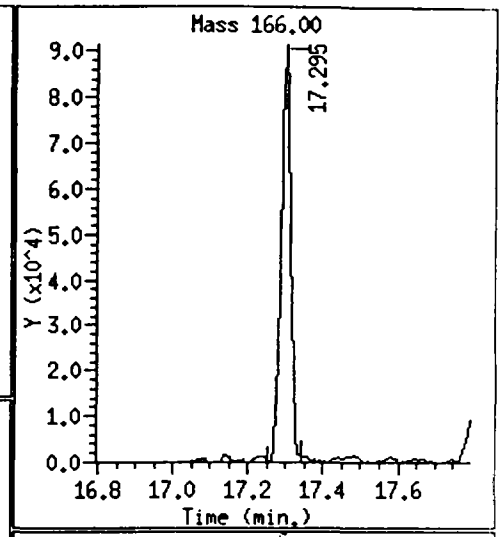
65 Fluorene

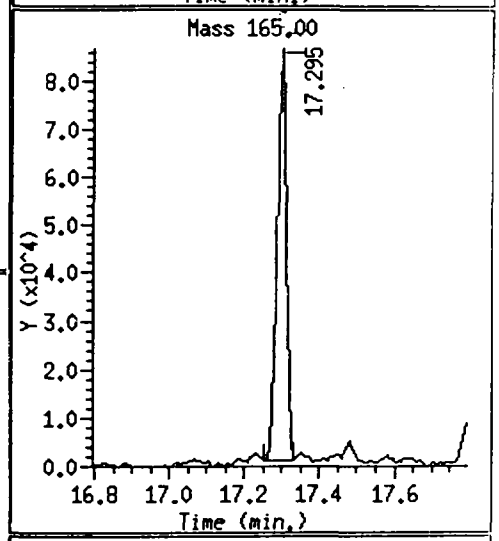


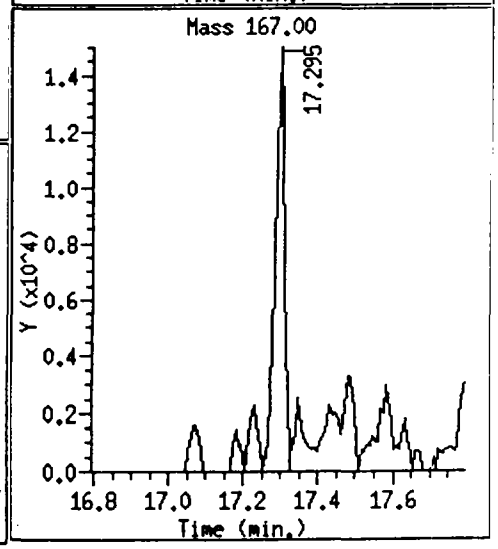












Data File: /chem/V.i/06-09-98.b/VH288.d

Date : 09-JUN-1998 14:42

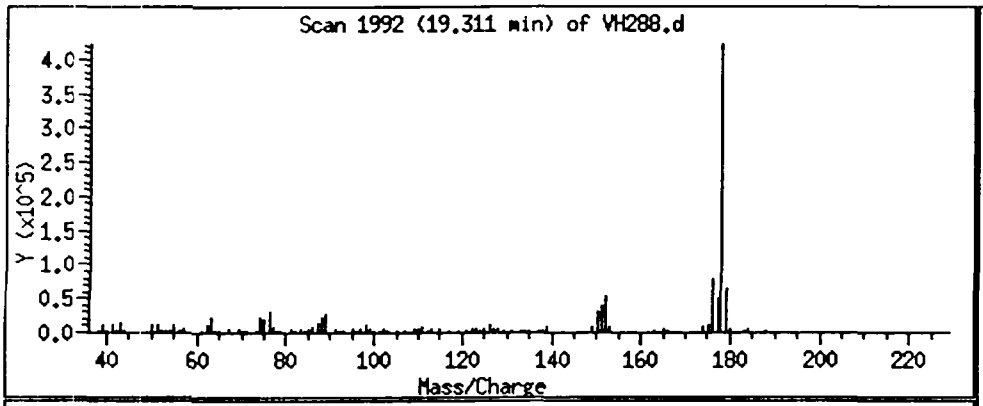
Instrument : V.i

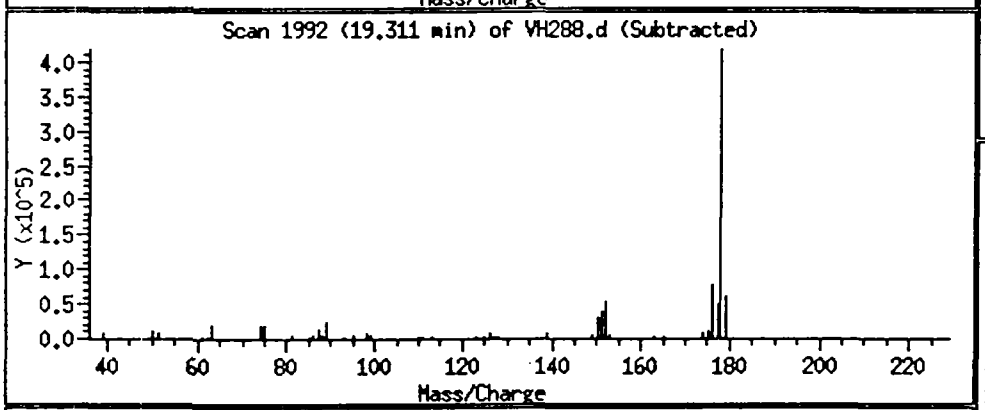
Sample ID : 24 HOUR

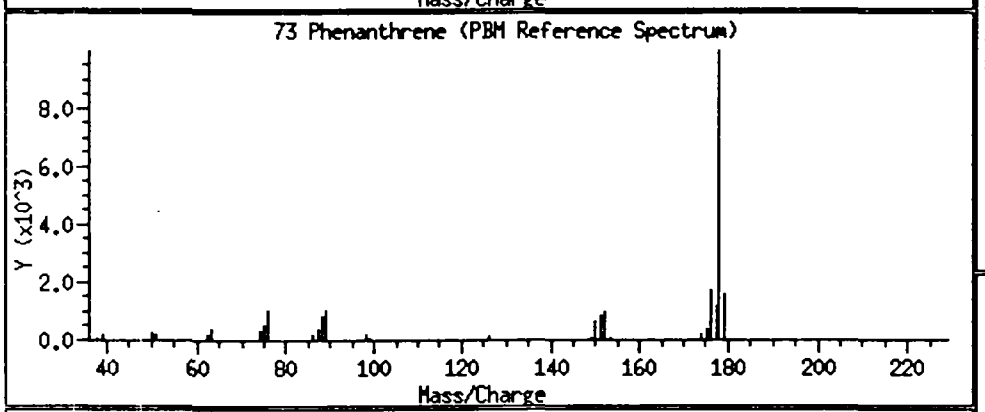
Column phase : DB-5 Column diameter : 0.32

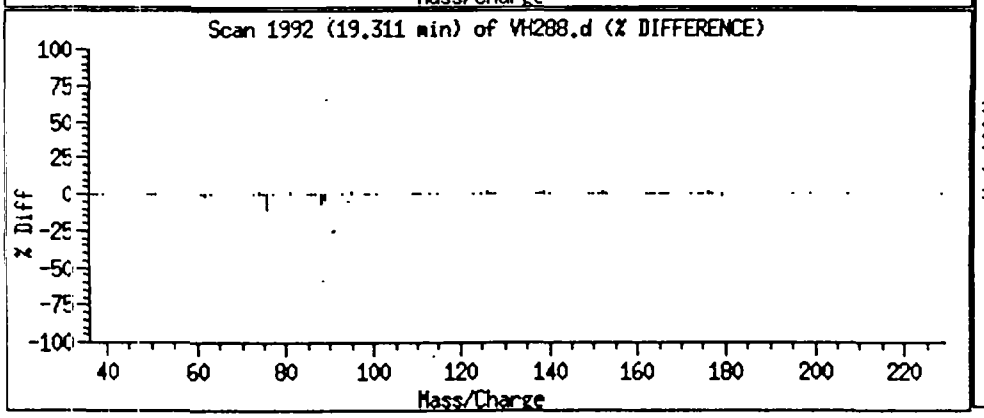
Volume Injected (uL) : 1.0

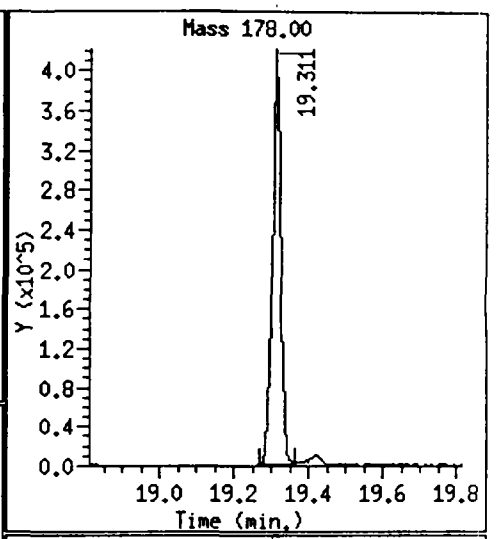
73 Phenanthrene

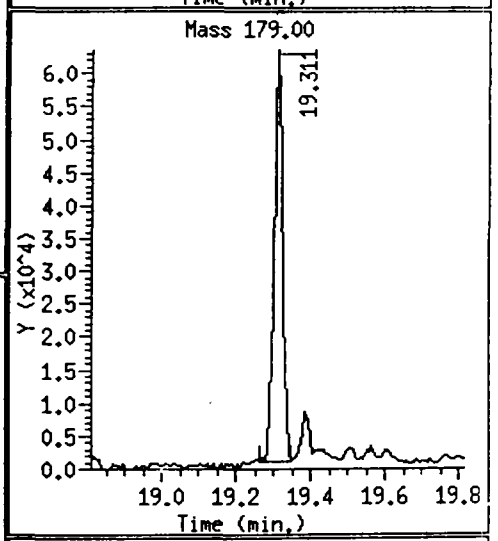


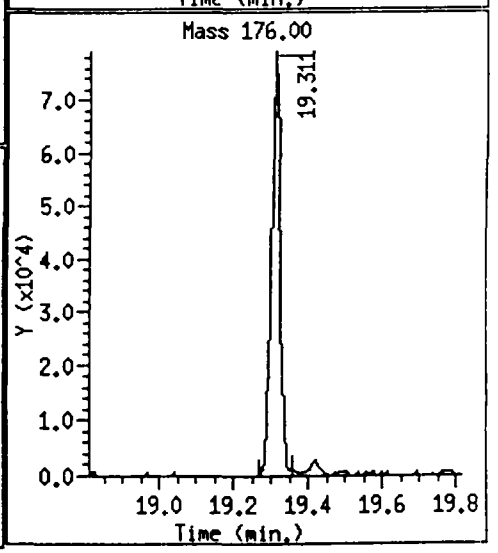












Data File: /chem/V.i/06-09-98.b/VH288.d

Date : 09-JUN-1998 14:42

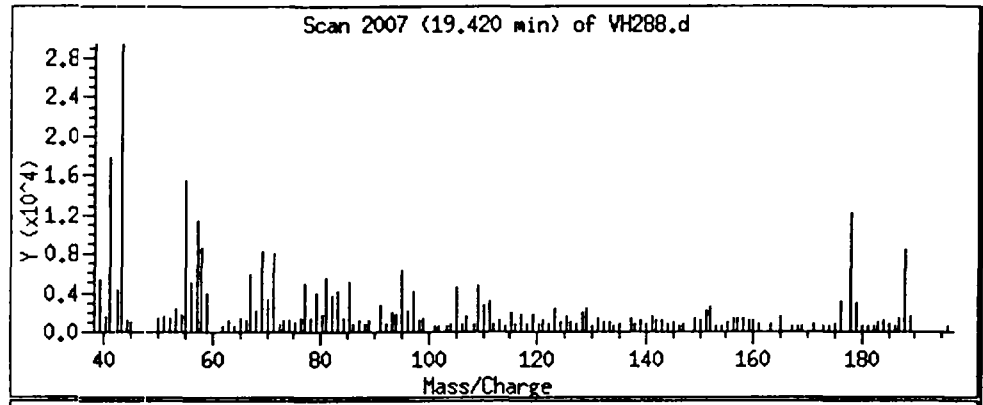
Instrument : V.i

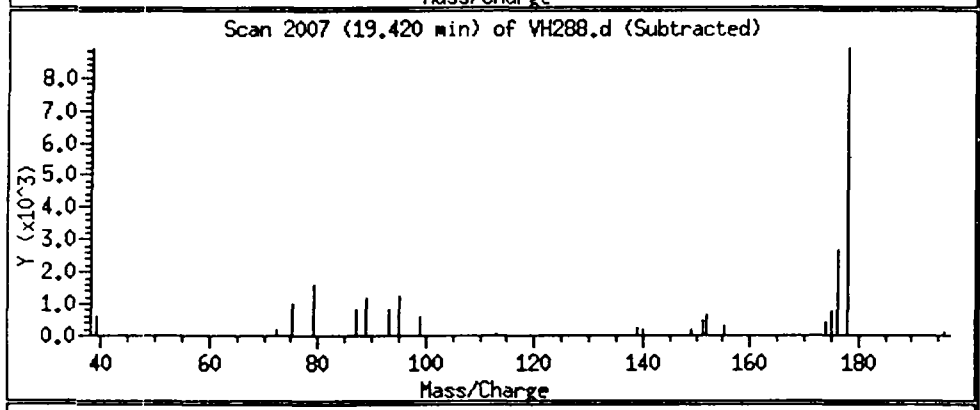
Sample ID : 24 HOUR

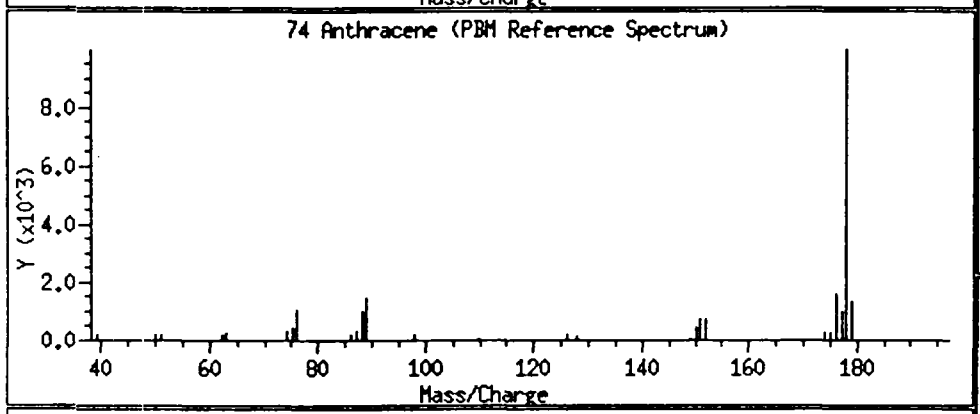
Column phase : DB-5 Column diameter : 0.32

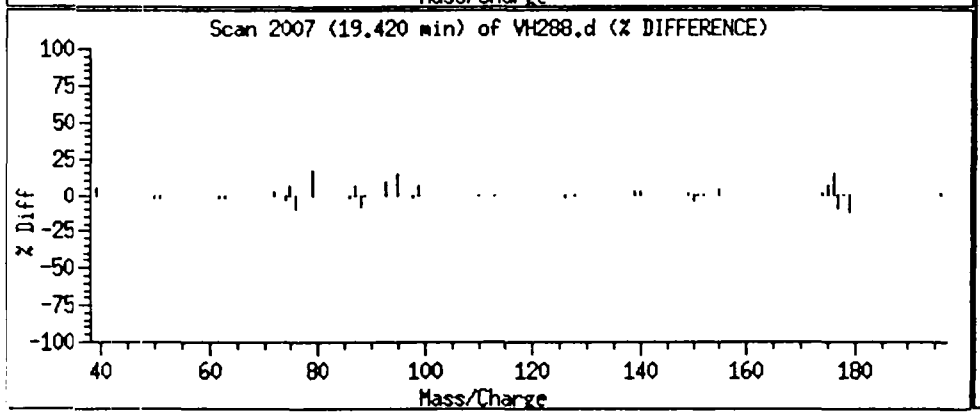
Volume Injected (uL) : 1.0

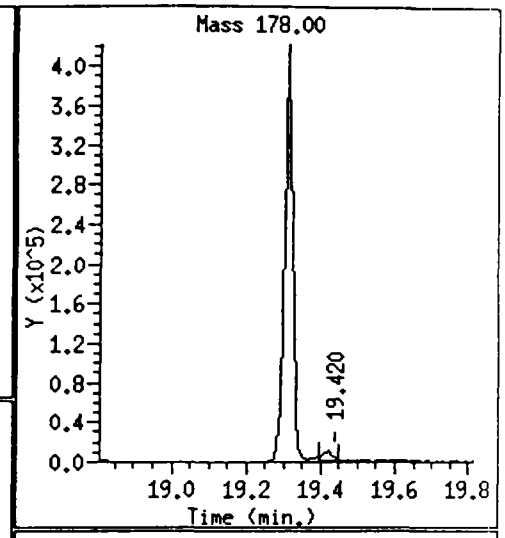
74 Anthracene

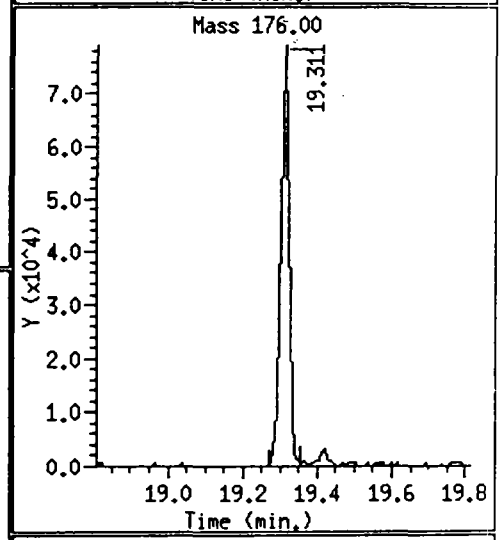


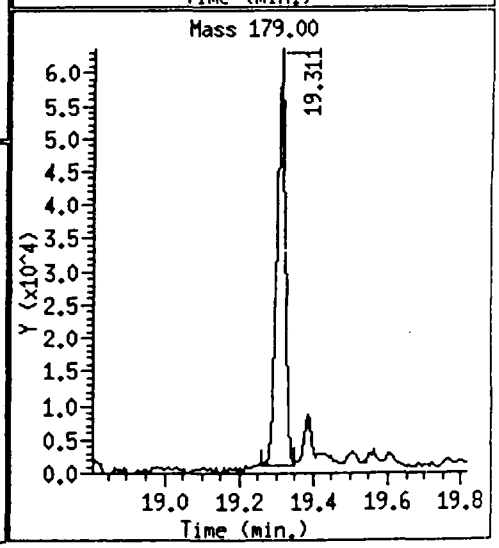












Data File: /chem/V.i/06-09-98.b/VH288.d

Date : 09-JUN-1998 14:42

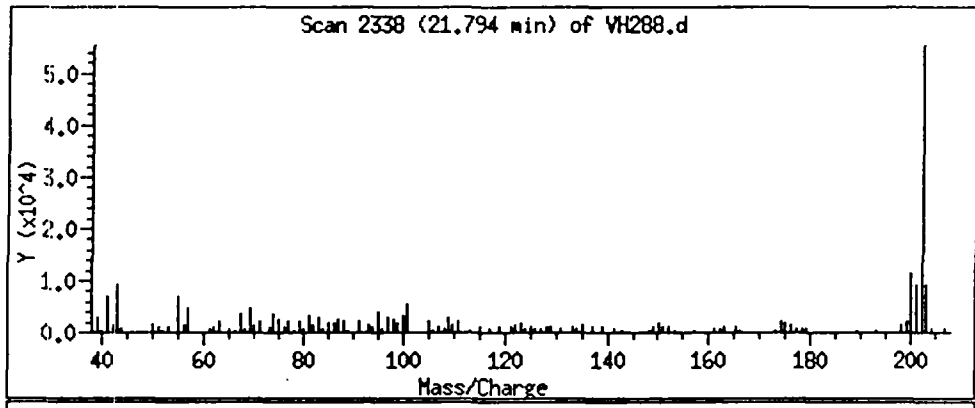
Instrument : V.i

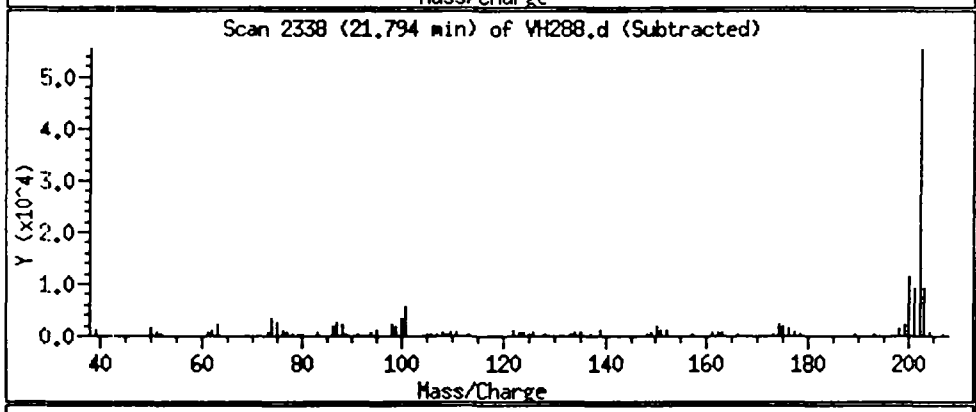
Sample ID : 24 HOUR

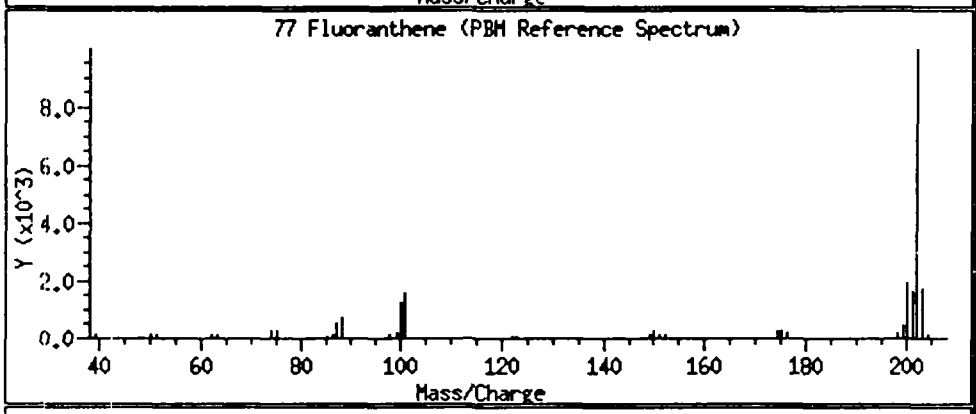
Column phase : DB-5 Column diameter : 0.32

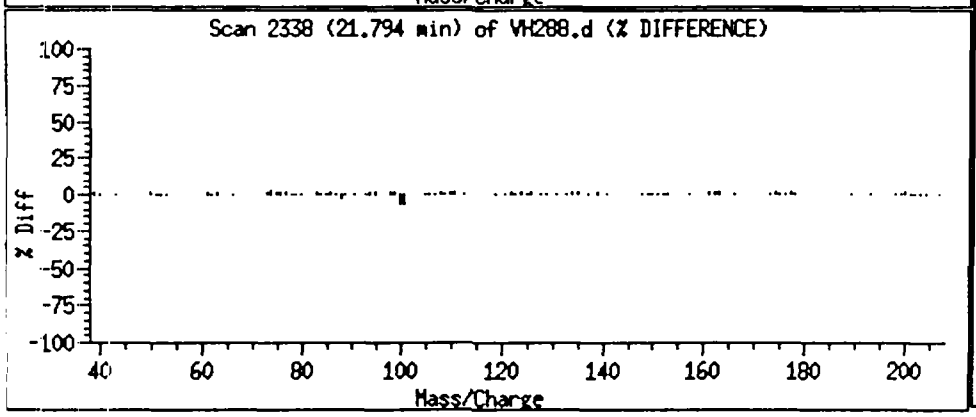
Volume Injected (uL) : 1.0

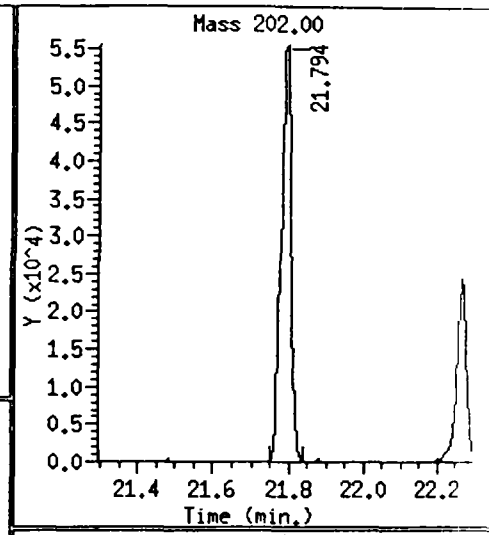
77 Fluoranthene

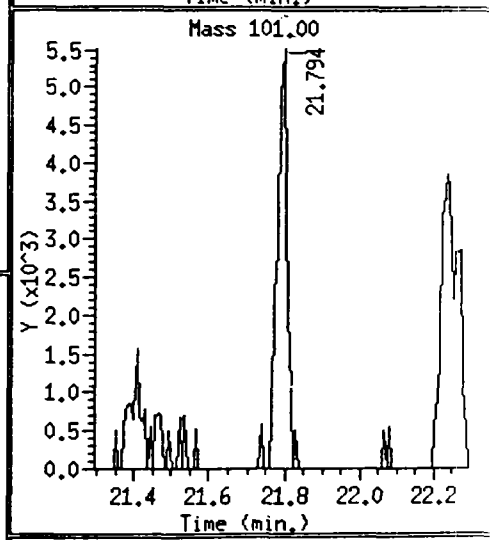


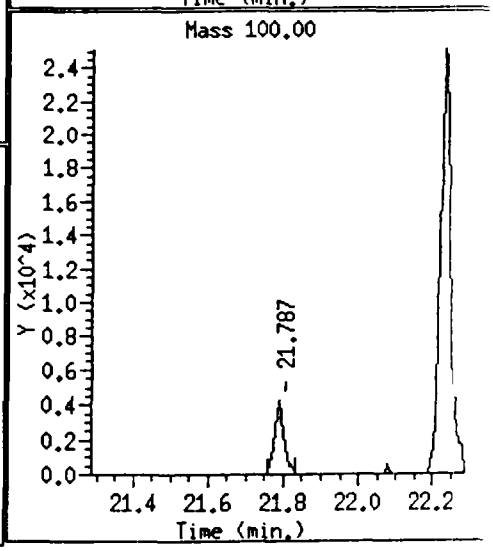












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH272

Method 8270A PUF
Sample ID: PUF FILTER BLANK

Client Project: Hull & Assoc
TLI ID: 209-67-25

Date Received: 06/02/98
Date Extracted: 06/04/98
Date Analyzed : 06/08/98

Response File: VH267

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	0.16	BJ	12.56		10
Acenaphthene-d_{10}		IS 3	16.16		
Fluorene		U		0.31	10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene		U		0.27	10
Anthracene		U		0.29	10
Fluoranthene		U		0.26	10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.26	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	84.91	19.38	4	85
Pyrene-d_{10}	118.58	22.23	5	119

Reviewed by PAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

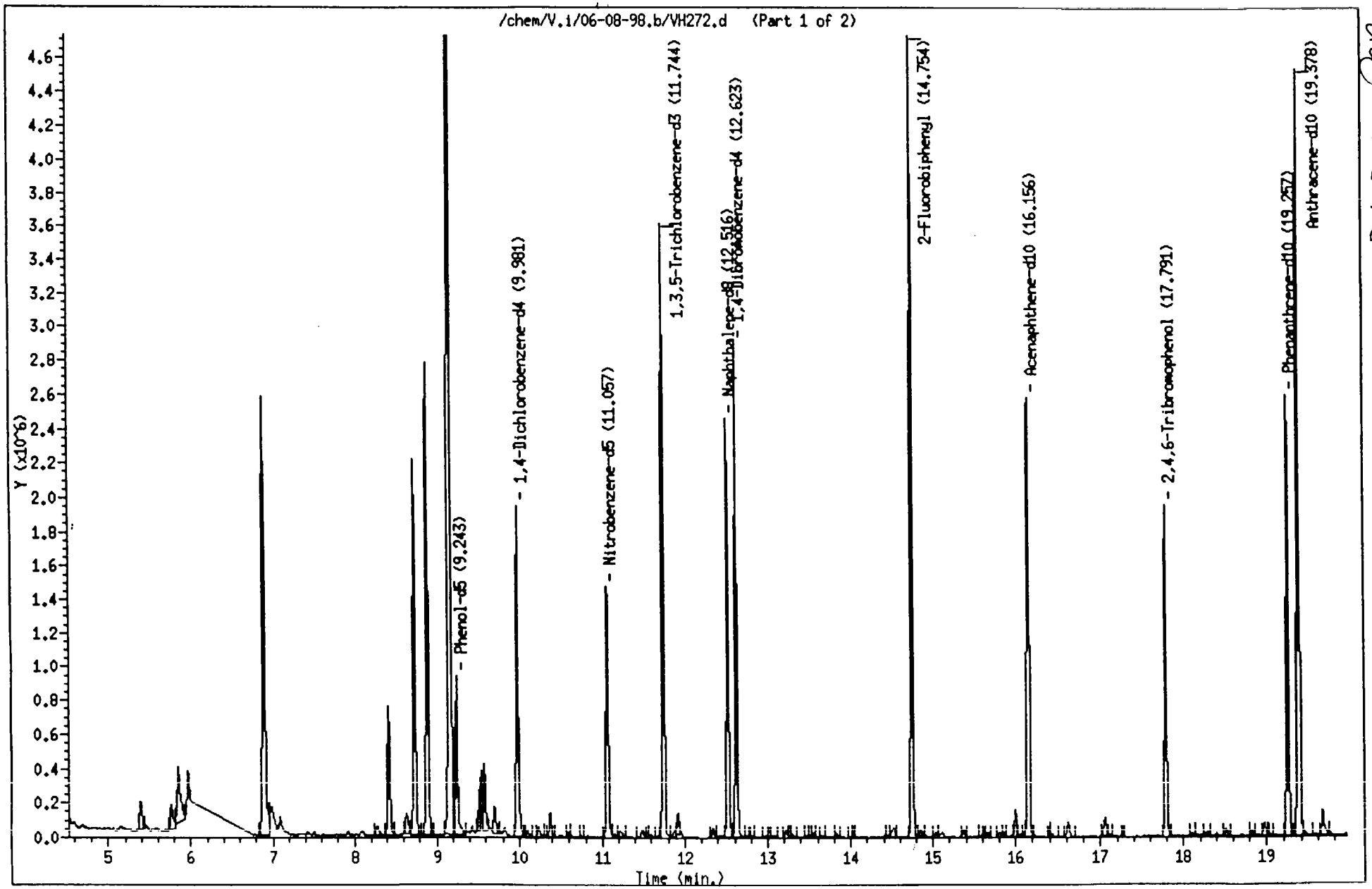
IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Data File: /chem/V.i/06-08-98.b/VH272.d
Date : 08-JUN-1998 18:15
Instrument : V.i
Sample ID : PUF FILTER BLANK
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32

168

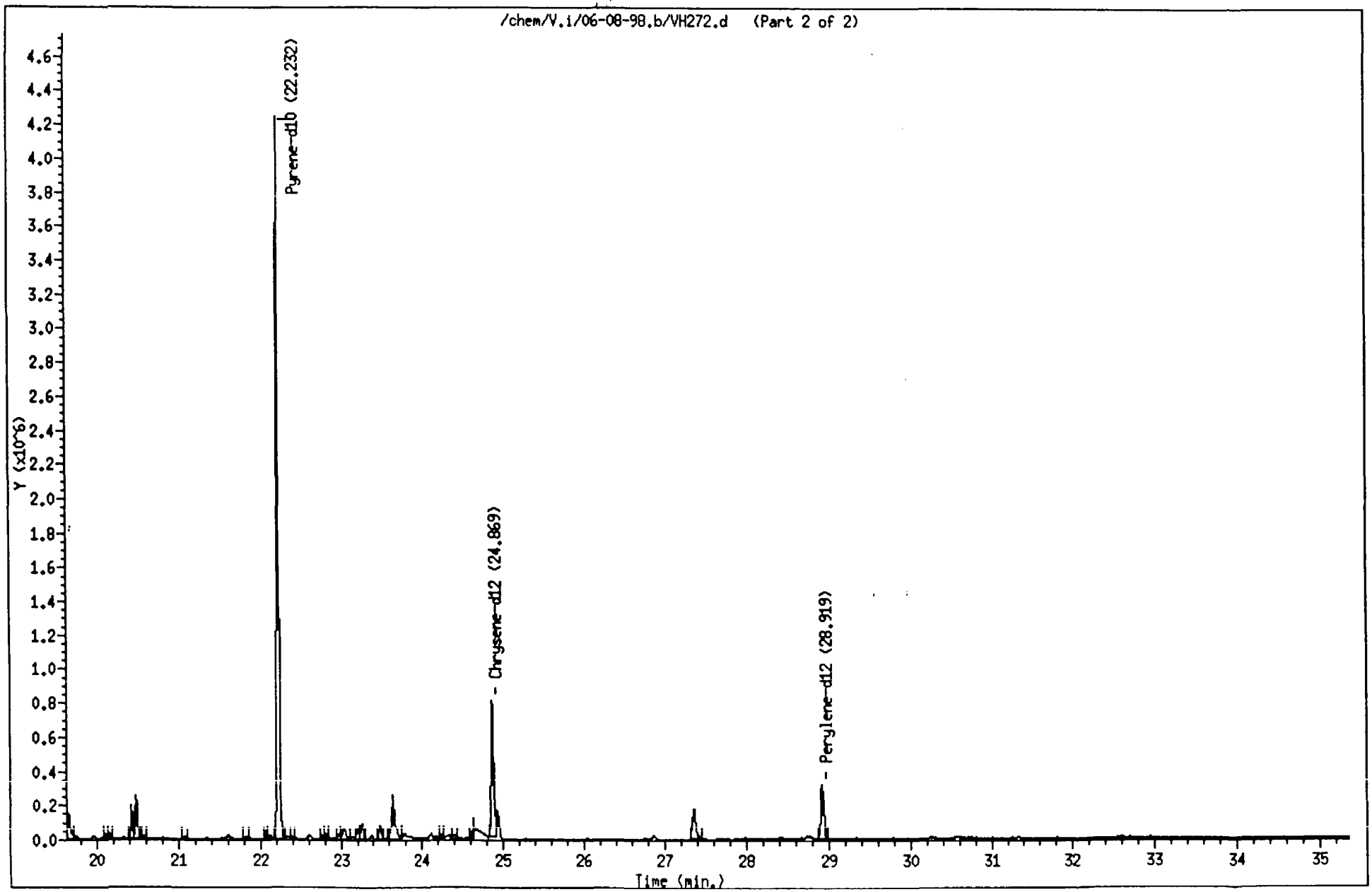


Data Review: PAB
Date: 6/12/98

Data File: /chem/V.i/06-08-98.b/VH272.d
Date : 08-JUN-1998 18:15
Instrument : V.i
Sample ID : PUF FILTER BLANK
Column phase : DB-5
Volume Injected (uL) : 1.0

Triangle Laboratories of RTP, Inc.

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH272.d
Lab. Id. : 405869 DD 6/9/98
Inj Date : 08-JUN-1998 18:15 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : PUF FILTER BLANK
Misc Info : 209-67-25
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 5
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.981(1.000)	490393	40.00	
*	2 Naphthalene-d8	136.00	12.516(1.000)	1808022	40.00	
*	3 Acenaphthene-d10	164.00	16.156(1.000)	1010170	40.00	
*	4 Phenanthrene-d10	188.00	19.257(1.000)	1638973	40.00	
*	5 Chrysene-d12	240.00	24.869(1.000)	508175	40.00	
*	6 Perylene-d12	264.00	28.919(1.000)	321675	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.243(0.926)	404956	27.35	27.35
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.057(0.883)	706047	45.97	45.97
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.744(0.938)	1040390	66.66	66.66
$	13 1,4-Dibromobenzene-d4	240.00	12.623(1.265)	852146	77.17	77.17
$	14 2-Fluorobiphenyl	172.00	14.754(0.913)	2153954	74.28	74.28
$	15 2,4,6-Tribromophenol	329.80	17.791(1.101)	325467	56.11	56.11
$	16 Anthracene-d10	188.00	19.378(1.006)	2860011	84.92	84.92
$	17 Pyrene-d10	212.00	22.232(0.894)	2753840	118.56	118.56
$	18 Terphenyl-d14	Compound Not Detected.				
	42 Naphthalene	~~Compound Not Detected.~~	12.56	6858	(M) PaB	
	46 2-Methylnaphthalene	142.00	13.982(1.117)	3796	0.14	0.14(a)
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	Compound Not Detected.				
	65 Fluorene	Compound Not Detected.				
	73 Phenanthrene	Compound Not Detected.				
	74 Anthracene	Compound Not Detected.				
	77 Fluoranthene	Compound Not Detected.				
	78 Pyrene	Compound Not Detected.				
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PaB
Date: 6/12/98 PaB 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).

Data File: /chem/V.i/06-08-98.b/VH272.d

Date : 08-JUN-1998 18:15

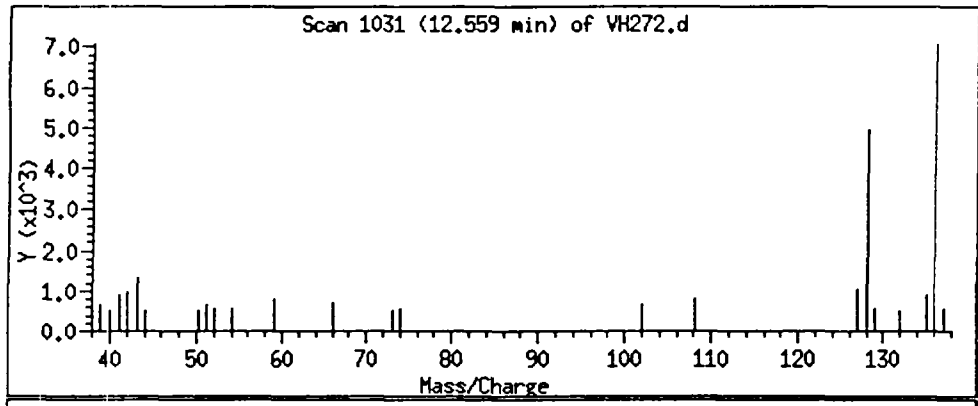
Instrument : V.i

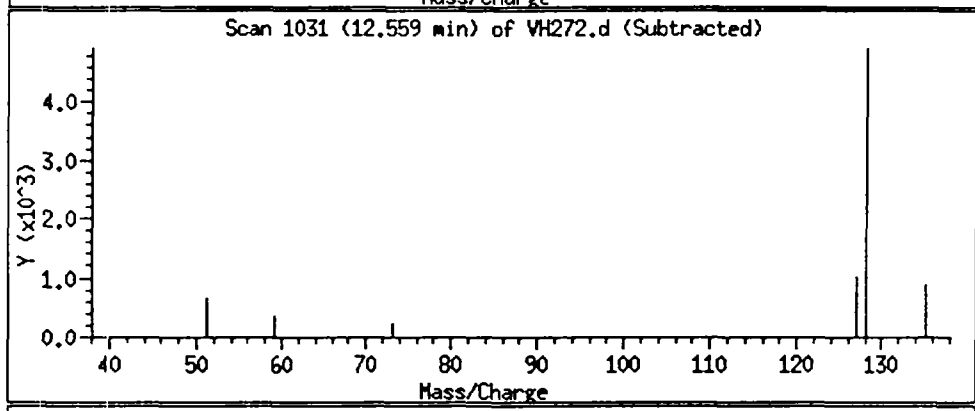
Sample ID : PUF FILTER BLANK

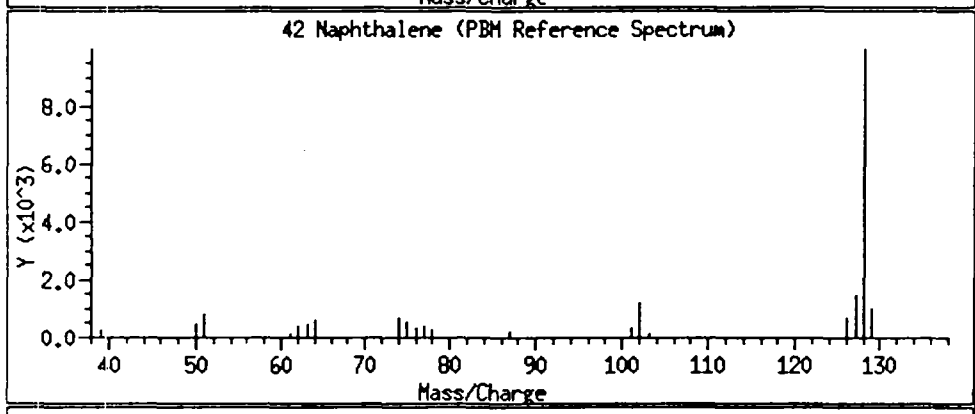
Column phase : DB-5 Column diameter : 0.32

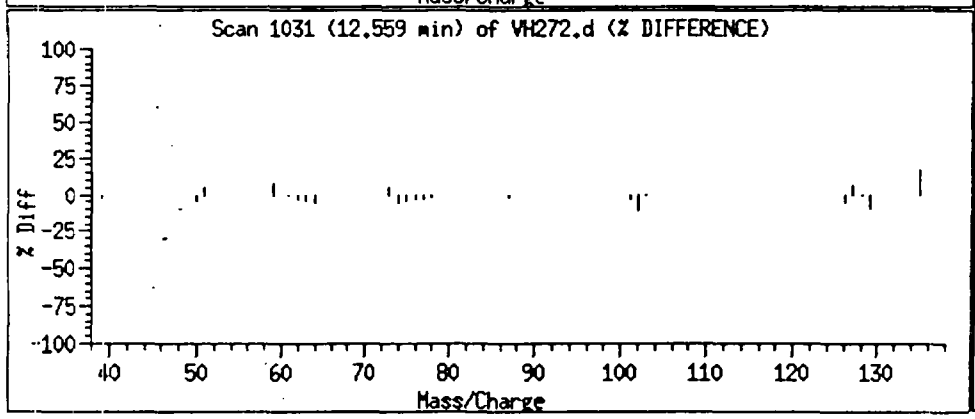
Volume Injected (uL) : 1.0

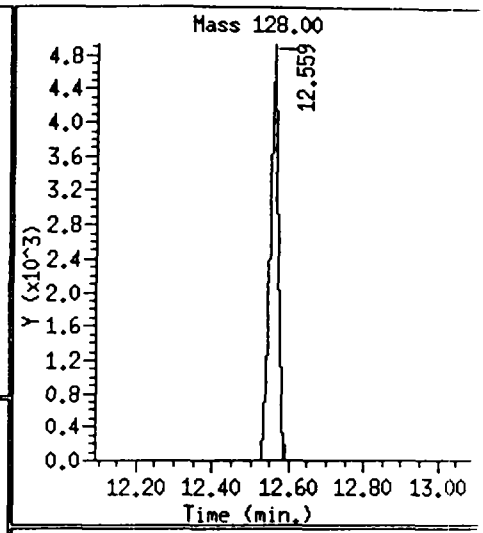
42 Naphthalene

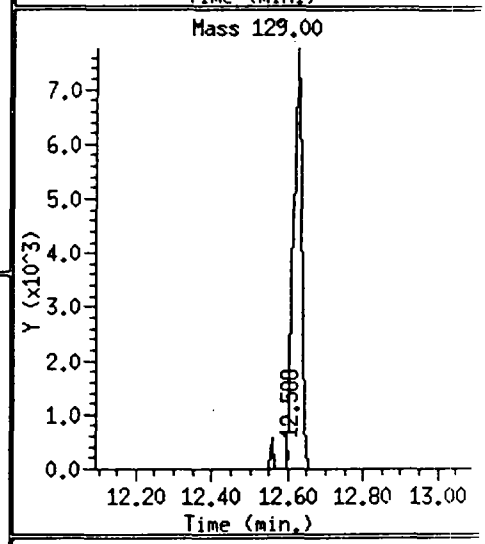


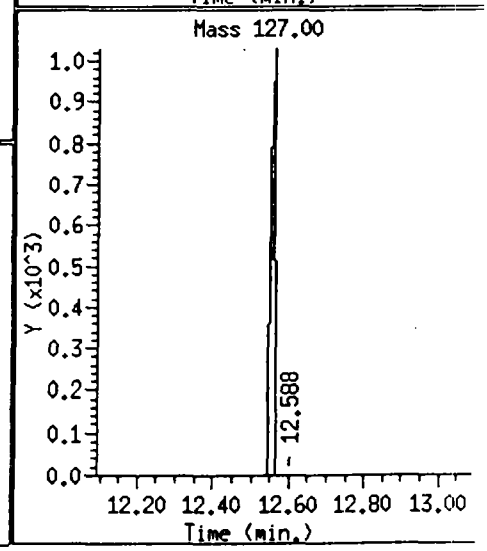












Derenzo and Associates, Inc.

Project Number: 45869
Sample File: VH271

Method 8270A PUF
Sample ID: SBLK060498

Client Project: Hull & Assoc
TLI ID: SBLK060498

Date Received: / /
Date Extracted: 06/04/98
Date Analyzed : 06/08/98

Response File: VH267

Dilution Factor: 1.00

Analyte	Amount ug	FLAG	RT	Det. Limit ug	Quan. Limit ug
Naphthalene-d_8		IS 2	12.52		
Naphthalene	0.70	J	12.56		10
Acenaphthene-d_{10}		IS 3	16.15		
Fluorene		U		0.32	10
Phenanthrene-d_{10}		IS 4	19.26		
Phenanthrene	0.15	J	19.30		10
Anthracene		U		0.29	10
Fluoranthene		U		0.27	10
Chrysene-d_{12}		IS 5	24.87		
Perylene-d_{12}		IS 6	28.92		
Benzo(a)pyrene		U		1.22	10

Surrogate Summary	Amount (ug)	RT	IS Ref	%REC
Anthracene-d_{10}	101.25	19.38	4	101
Pyrene-d_{10}	141.10	22.24	5	141
Terphenyl-d_{14}	129.75	22.60	5	130

Reviewed by PAB Date 6/12/98

NA- Not Applicable; Det. Limit: Detection Limit; Quan. Limit: Quantitation Limit

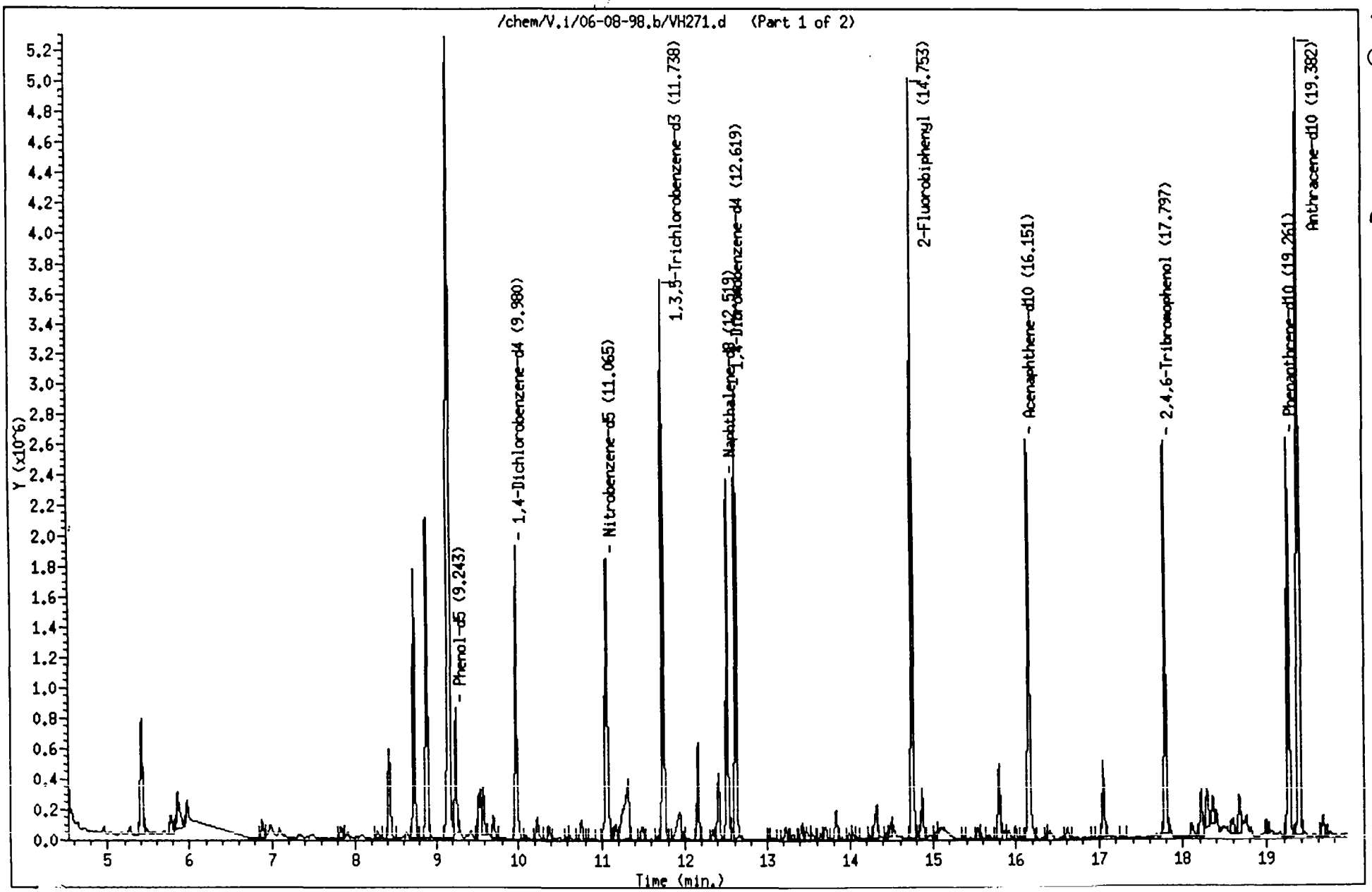
IS: Internal Standard; U: Undetected; B: Present In Blank; J: Estimated- Below Quantitation Limit; E: Estimated- Above Calibration Range

Triangle Laboratories, Inc.
801 Capitola Drive • Durham, North Carolina 27713
Phone: (919) 544-5729 • Fax: (919) 544-5491

Savar v3.7
Printed: 15:07 06/12/1998

Data File: /chem/V.i/06-08-98.b/VH271.d
Date : 08-JUN-1998 17:25
Instrument : V.i
Sample ID : SBLK 060498
Column phase : DB-5
Volume Injected (uL) : 1.0

Column diameter : 0.32

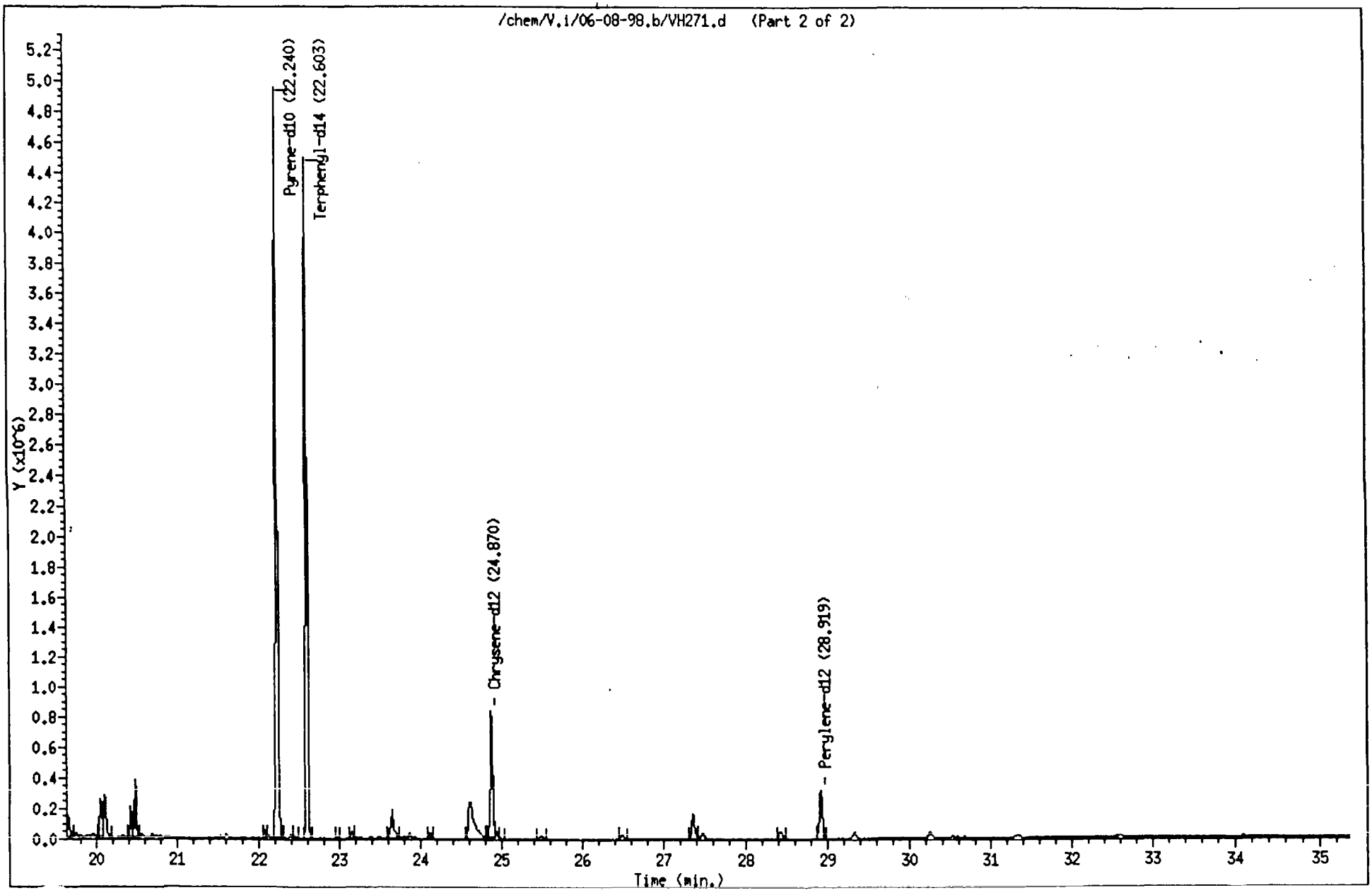


174

Data Review: PAB
Date: 6/12/98

Data File: /chem/V.i/06-08-98.b/VH271.d
Date : 08-JUN-1998 17:25
Instrument : V.i
Sample ID : SBLK 060498
Column phase : DB-5
Volume Injected (uL) : 1.0

Column diameter : 0.32



TRIANGLE LABORATORIES, INC.

SEMI-VOLATILE QUANTITATION REPORT

Data file : /chem/V.i/06-08-98.b/VH271.d
Lab. Id. : 45869 DD 6/9/98
Inj Date : 08-JUN-1998 17:25 Autotune Date: 05-Jun-98 13:48:1
Operator : DD Inst ID: V.i
Smp Info : SBLK 060498
Misc Info :
Comment :
Method : /chem/V.i/06-08-98.b/cclist.m
Meth Date : 08-Jun-1998 15:01 target
Cal Date : 08-JUN-1998 14:17 Cal File: VH267.d
Als bottle: 4
Dil Factor: 1.000 Target Version: Target 2.20
Integrator: HP RTE Compound Sublist: pah.sub
Sample Type: AIR

	Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
*	1 1,4-Dichlorobenzene-d4	152.00	9.980(1.000)	483775	40.00	
*	2 Naphthalene-d8	136.00	12.519(1.000)	1718900	40.00	
*	3 Acenaphthene-d10	164.00	16.151(1.000)	987543	40.00	
*	4 Phenanthrene-d10	188.00	19.261(1.000)	1589576	40.00	
*	5 Chrysene-d12	240.00	24.870(1.000)	517450	40.00	
*	6 Perylene-d12	264.00	28.919(1.000)	331574	40.00	
$	7 2-Fluorophenol	Compound Not Detected.				
$	8 Phenol-d5	99.00	9.243(0.926)	375399	25.70	25.70
$	9 2-Chlorophenol-d4	Compound Not Detected.				
$	10 1,2-Dichlorobenzene-d4	Compound Not Detected.				
$	11 Nitrobenzene-d5	82.00	11.065(0.884)	908591	62.23	62.23
$	12 1,3,5-Trichlorobenzene-d3	185.00	11.738(0.938)	1068189	71.99	71.99
$	13 1,4-Dibromobenzene-d4	240.00	12.626(1.265)	858232	78.78	78.78
$	14 2-Fluorobiphenyl	172.00	14.753(0.913)	2284600	80.59	80.59
$	15 2,4,6-Tribromophenol	329.80	17.797(1.102)	462062	81.48	81.48
$	16 Anthracene-d10	188.00	19.382(1.006)	3307351	101.25	101.25
$	17 Pyrene-d10	212.00	22.240(0.894)	3336733	141.08	141.08(R)
$	18 Terphenyl-d14	244.00	22.603(0.909)	1925204	129.78	129.78
	42 Naphthalene	128.00	12.562(1.003)	29090	0.70	0.70(a)
	46 2-Methylnaphthalene	142.00	13.980(1.117)	8078	0.32	0.32(a)
	50 2-Chloronaphthalene	Compound Not Detected.				
	56 Acenaphthylene	Compound Not Detected.				
	58 Acenaphthene	~~154.00~~	~~16.216(1.004)~~	~~870~~	~~0.04~~	~~0.04(a)~~ TP PAB
	65 Fluorene	Compound Not Detected.				
	73 Phenanthrene	178.00	19.304(1.002)	5482	0.15	0.15(a)
	74 Anthracene	~~178.00~~	~~19.304(1.002)~~	~~5482~~	~~0.16~~	~~0.16(a)~~ TP PAB
	77 Fluoranthene	Compound Not Detected.				
	78 Pyrene	Compound Not Detected.				
	83 Benzo(a)anthracene	Compound Not Detected.				
	84 Chrysene	Compound Not Detected.				
	86 Benzo(b)fluoranthene	Compound Not Detected.				

Data Review: PAB
Date: 6/12/98

Compounds	QUANT SIG MASS	RT (REL RT)	RESPONSE	CONCENTRATIONS ON-COLUMN (ng)	FINAL (ug)
87 Benzo(k)fluoranthene	Compound Not Detected.				
88 Benzo(e)pyrene	Compound Not Detected.				
89 Benzo(a)pyrene	Compound Not Detected.				
90 Perylene	Compound Not Detected.				
91 Indeno(1,2,3-cd)pyrene	Compound Not Detected.				
92 Dibenz(a,h)anthracene	Compound Not Detected.				
93 Benzo(g,h,i)perylene	Compound Not Detected.				

QC Flag Legend

a - Target compound detected but, quantitated amount
Below Limit Of Quantitation(BLOQ).
R - Spike/Surrogate failed recovery limits.

Data File: /chem/V.i/06-08-98.b/VH271.d

Date : 08-JUN-1998 17:25

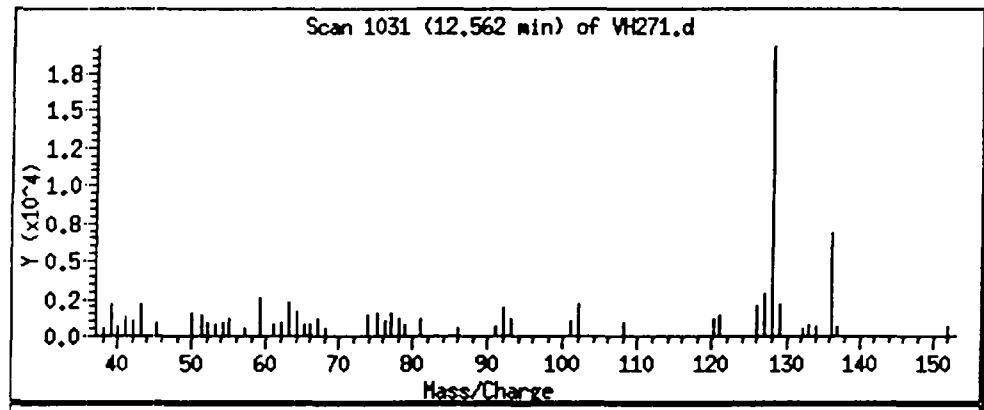
Instrument : V.i

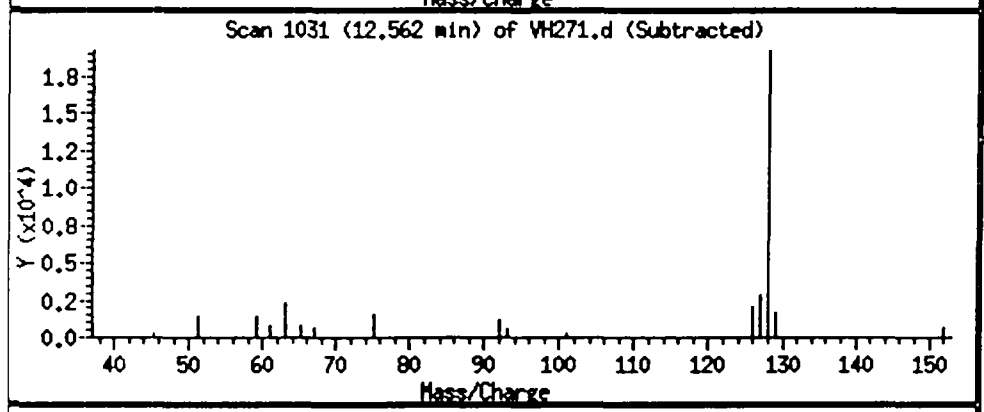
Sample ID : SBLK 060498

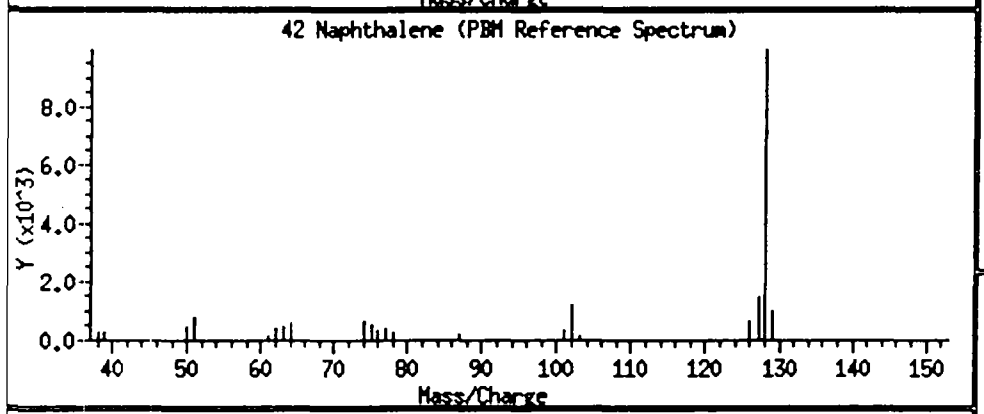
Column phase : DB-5 Column diameter : 0.32

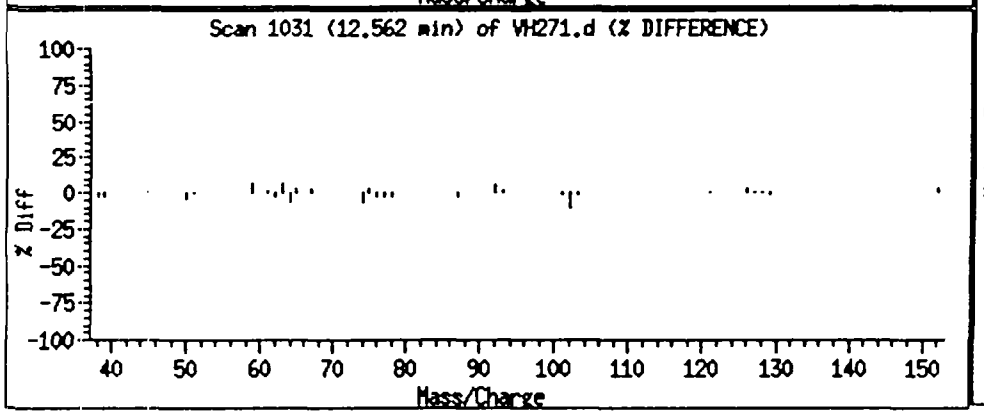
Volume Injected (uL) : 1.0

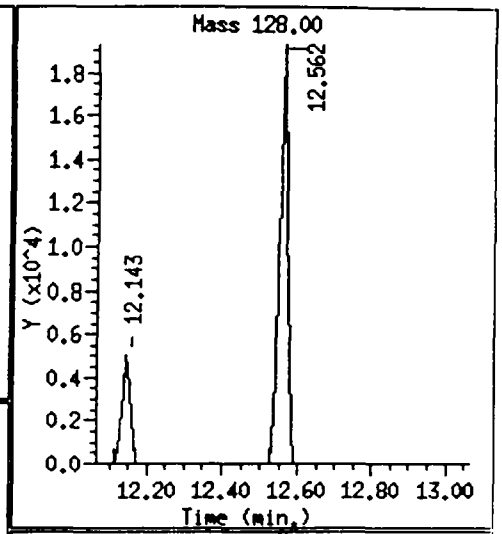
42 Naphthalene

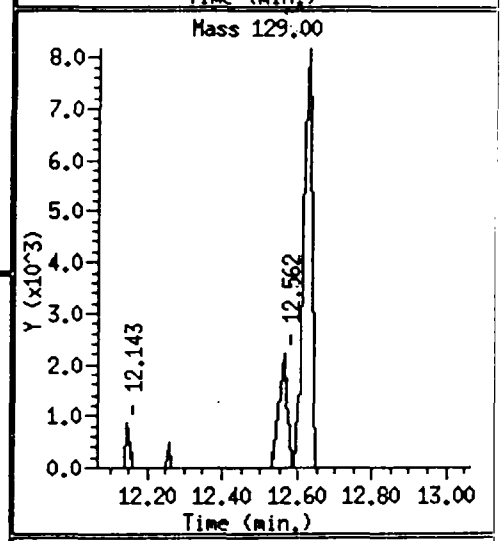


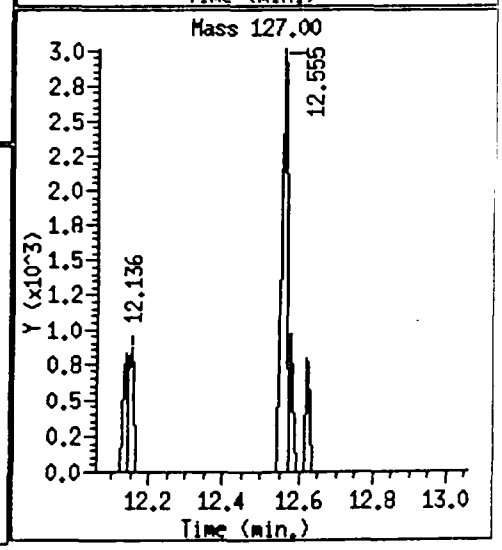












Data File: /chem/V.i/06-08-98.b/VH271.d

Date : 08-JUN-1998 17:25

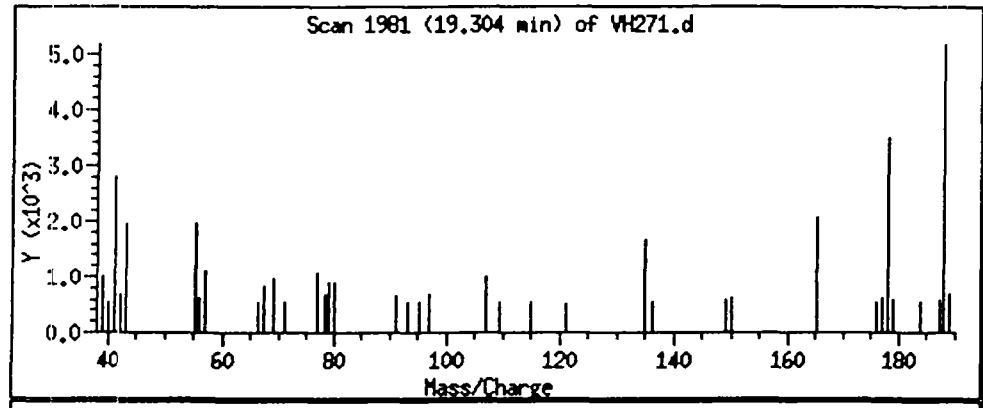
Instrument : V.i

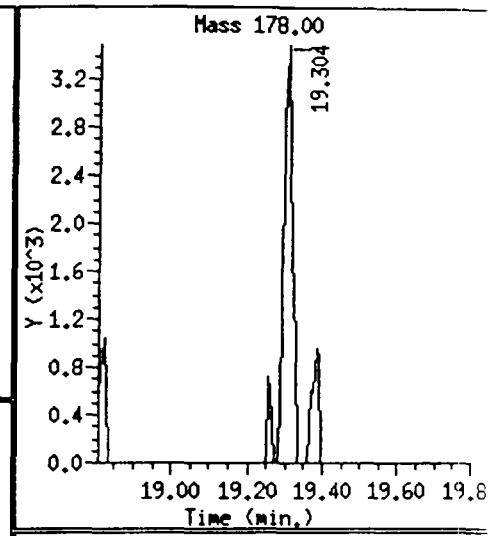
Sample ID : SBLK 060498

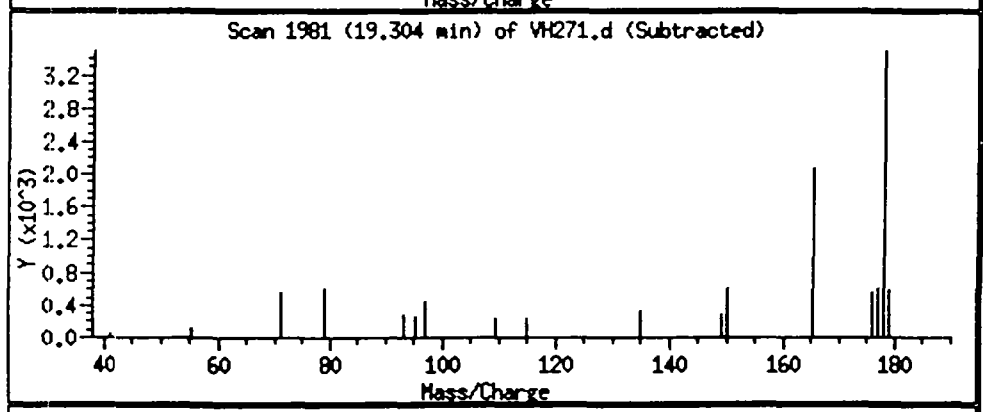
Column phase : DB-5 Column diameter : 0.32

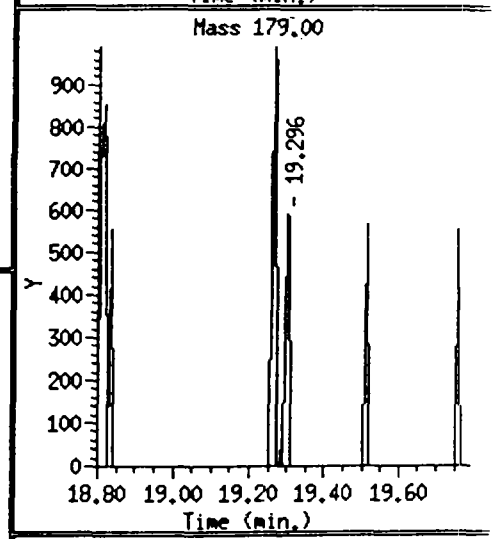
Volume Injected (uL) : 1.0

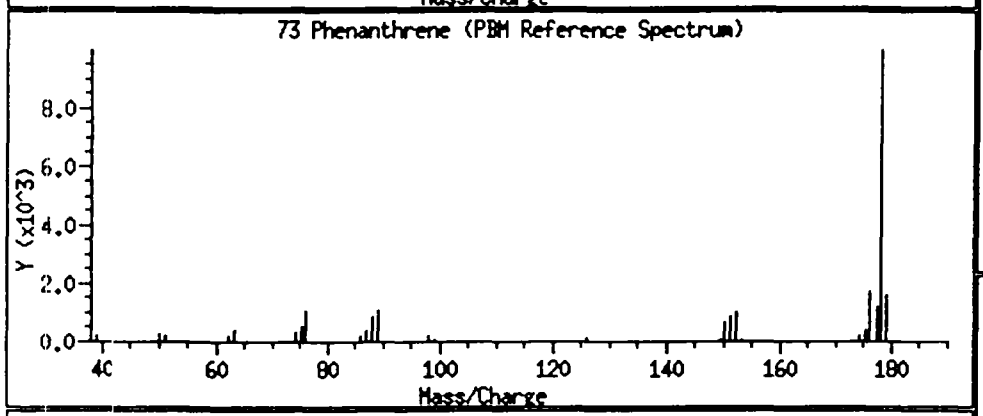
73 Phenanthrene

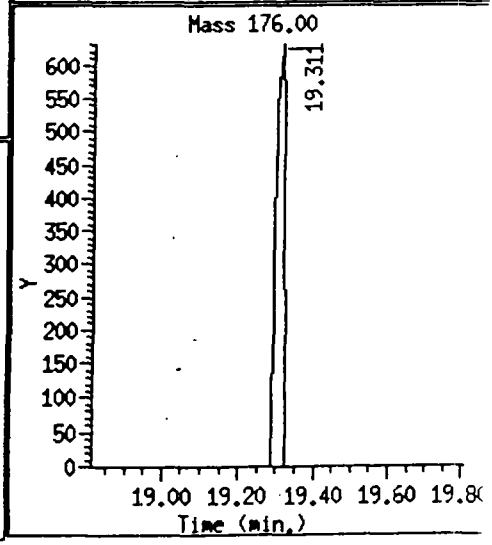


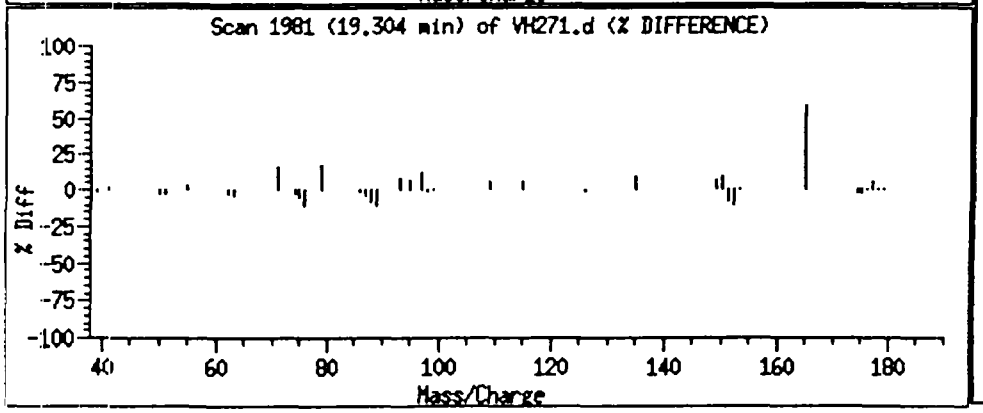












Triangle Laboratories, Inc.
Continuing Calibration Curve

CCAL File: VH267 Date of Analysis :06/08/98 Analyte List: 8270A
ICAL File: ICALV506

Analyte	Flag	RF50	RFMEAN	%D
Naphthalene-d8	I			
Naphthalene		0.962	0.989	2.7
Acenaphthene-d10	I			
Fluorene		1.275	1.308	2.5
Phenanthrene-d10	I			
Phenanthrene		0.917	0.992	7.6
Anthracene		0.855	0.949	9.9
Fluoranthene	C	0.936	1.081	13.4
Chrysene-d12	I			
Perylene-d12	I			
Benzo(a)pyrene	C	0.987	1.094	9.8

Surrogate	Flag	RF50	RFMEAN	%D
Anthracene-d10	S	0.822	0.894	8.1
Pyrene-d10	S	1.828	1.706	-7.2
Terphenyl-d14	S	1.147	1.160	1.1

Approved by: PaB Date 6/12/98

*- Fails QC Criteria for %D; << - Rf less than minimum QC RF; >>- RF greater than maximum QC RF

Triangle Laboratories, Inc.
Continuing Calibration Curve

CCAL File: VH284	Date of Analysis :06/09/98	Analyte List: 8270A
ICAL File: ICALV506		

Analyte	Flag	RF50	RFMEAN	%D
Naphthalene-d8	I			
Naphthalene		0.991	0.989	-0.2
Acenaphthene-d10	I			
Fluorene		1.361	1.308	-4.1
Phenanthrene-d10	I			
Phenanthrene		0.899	0.992	9.4
Anthracene		0.844	0.949	11.1
Fluoranthene	C	0.885	1.081	18.1
Chrysene-d12	I			
Perylene-d12	I			
Benzo(a)pyrene	C	0.991	1.094	9.4

Surrogate	Flag	RF50	RFMEAN	%D
Anthracene-d10	S	0.822	0.894	8.1
Pyrene-d10	S	2.057	1.706	-20.6
Terphenyl-d14	S	1.249	1.160	-7.7

Approved by: BaB Date 6/12/98

*- Fails QC Criteria for %D; << - Rf less than minimum QC RF; >>- RF greater than maximum QC RF

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Triangle Laboratories, Inc.
Initial Calibration Curve

ICAL File: ICALV506	Date of Analysis :05/06/98	Analyte List: 8270A
RF20 VH172	RF50 VH171	RF80 VH169
RF120 VH170	RF160 VH168	

Analyte	Flag	RF20	RF50	RF80	RF120	RF160	MEAN	%RSD
Naphthalene-d8	I							
Naphthalene		1.053	0.974	0.968	0.955	0.997	0.989	3.9
Acenaphthene-d10	I							
Fluorene		1.237	1.199	1.426	1.277	1.400	1.308	7.7
Phenanthrene-d10	I							
Phenanthrene		1.059	0.985	0.961	0.941	1.014	0.992	4.7
Anthracene		0.982	0.946	0.938	0.907	0.973	0.949	3.1
Fluoranthene	C	1.240	1.133	1.046	0.985	1.004	1.081	9.7
Chrysene-d12	I							
Perylene-d12	I							
Benzo(a)pyrene	C	1.116	1.062	1.067	1.121	1.104	1.094	2.5
Average %RSD								5.3

Surrogate	Flag	RF20	RF50	RF80	RF120	RF160	Mean	%RSD
Anthracene-d10	S	0.934	0.900	0.872	0.858	0.905	0.894	3.3
Pyrene-d10	S	1.606	1.623	1.794	1.779	1.726	1.706	5.1
Terphenyl-d14	S	1.050	1.094	1.218	1.227	1.209	1.160	7.0

Approved by: PaB Date 6/12/98

*- Fails QC Criteria for %RSD; << - RF less than minimum QC RF; >> - RF greater than maximum QC RF

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APPENDIX D

LABORATORY
STATEMENT OF QUALIFICATIONS

STATEMENT OF QUALIFICATIONS

FOR

Fibertec

ANALYTICAL LABORATORY SERVICES

INTRODUCTION

This statement of qualifications provides information on the analytical laboratory services provided by Fibertec, along with outlining many of the advantages of using Fibertec as an integral part of your environmental compliance team.

COMPANY PROFILE

Founded in 1987, Fibertec is a well respected leader in providing laboratory services to the environmental community. We offer our customers a broad range of analyses, complemented by our value added services and personal care. With nearly a decade of experience, we have learned the importance of developing a partnership which focuses on meeting our customer's regulatory requirements in a timely and cost effective manner.

BUSINESS PHILOSOPHY

Fibertec believes in purchasing state-of-the-art instrumentation, hiring the highest quality chemists and technicians, providing necessary training, then allowing freedom and flexibility for Fibertec's staff to better serve our customers.

We recognize that trust is earned and that each opportunity we have to assist our customers must be met with the utmost care and responsiveness.

ANALYTICAL SERVICES

Fibertec's reputation is built upon providing consistently high quality laboratory services in support of numerous federal, state, and local programs. A few of the major programs and services are listed below.

Underground Storage Tank (UST)

- Volatiles (BTEX/MTBE)
- Semivolatiles (PNA's)
- Total Petroleum Hydrocarbons (GRO & DRO)
- Metals

National Pollution Discharge Elimination System (NPDES)

- Volatiles
- Semivolatiles
- Pesticides
- Metals
- Wet Chemistry Parameters

Hazardous Waste Characterization

- Ignitability
- Corrosivity
- Reactivity
- TCLP Analyses

PCB Analysis

Other Services

- Developing Quality Assurance Project Plans
- Expert Testimony / Litigation Support
- Analytical Method Development
- Field Services / Sampling Support

CUSTOMER SERVICES

In addition to the many analytical services we provide, Fibertec differentiates itself based on its customer services. Fibertec's overriding goal is to provide excellent customer service and uncompromised data quality. Service excellence is maintained through flexibility and the customer focus necessary to continually improve our understanding of our customers' business and specific needs. We then identify and improve ways to service these needs. The following are just a few of the customer services we have provided:

- electronic deliverables formatted and ready to be used by customer database systems
- laboratory completed forms for compliance monitoring
- customized reporting formats
- sampling bottles (with preservatives, if required)
- sample pick-up
- laboratory sponsored training seminars
- fax/phone results
- RUSH analytical services
- sampling services
- literature searches
- QA/QC packages

We at Fibertec realize that our customers are the reason we are in business, and this concept of customer focus is what drives our business. For this reason, we are constantly searching for new and innovative ways to better service our customers, and we welcome your suggestions.

FACILITIES AND EQUIPMENT

Fibertec's modern laboratory is located in a 6000 square foot building in Holt, Michigan on the south side of Lansing. The laboratory occupies approximately half of the building. The remainder is used for staff offices, conference room, and reception area.

The lab was designed to (1) minimize potential contamination with ventilation and control technology, while (2) maximizing analyst comfort and productivity. The laboratory is designed with individual departments to streamline and enhance work flow. The result is increased productivity which allows us to maintain or even reduce both turnaround time and our already competitive fees, while maintaining the superior quality you've grown to expect from Fibertec.

Fibertec utilizes the most technologically advanced analytical instrumentation available. From the state-of-the-art ICP-mass spectrometer, gas chromatographs, and GC/MS's to a comprehensive wet chemistry department, Fibertec uses the latest technology to better service our customers.

Fibertec'S analytical laboratory instrumentation list is provided in Appendix A.

ORGANIZATIONAL STRUCTURE

The laboratory is designed to incorporate the advantages of instrumentation redundancy with the service and project ownership advantages of a local niche laboratory. Fibertec provides the best of both worlds with a local commitment to excellent customer service and innovative solutions for our customers.

PERSONNEL QUALIFICATIONS

Fibertec believes people are its greatest asset. We hire only the most qualified individuals, and provide specialized training, as necessary, in analytical methodology, advanced techniques, quality assurance, and customer service. Our professionals also attend conferences and training seminars to keep current on the latest regulations and methodologies. The result is improved service for our customers.

QUALITY ASSURANCE/QUALITY CONTROL PROGRAM

Fibertec Analytical Services incorporates a quality system approach to ensure results are accurate and of known and consistently high quality. Using this approach, Fibertec focuses its attention on proactively identifying potential problems and correcting them before significant problems arise. We do this by statistically monitoring key quality indicators which include:

- spike recoveries
- duplicate sample analyses
- blanks (lab, field, & trip)
- surrogate recoveries
- performance evaluation samples
- laboratory control samples
- instrument calibration
- second source standards analysis

Fibertec's QA/QC procedures are designed to meet or exceed applicable regulatory requirements and are based on guidelines established in the following programs:

- USEPA Test Methods for Evaluating Hazardous Waste, SW846
- USEPA Drinking Water
- USEPA Wastewater
- USEPA FIFRA & TSCA
- USEPA Superfund CLP Statement of Work
- US Army, USATHAMA QA Program

PROFICIENCY TESTING PROGRAMS

Performance evaluation (PE) programs are an important element in our continuing effort to monitor and improve Fibertec result quality through external QA programs. We participate in the following PE programs:

Agency	Program	Frequency
USEPA	Water Supply	semi-annual
USEPA	Water Pollution	semi-annual
USEPA	Discharge Monitoring Report (DMR)	annual

INSURANCE

Fibertec recognizes the risks associated with environmental regulations. We therefore maintain insurance which meets or exceeds industry standards to minimize risk for our customers.

The insurance carried by Fibertec is provided in appendix B.

WHY CHOOSE FIBERTEC ?

- accuracy
- speed
- dependability
- flexibility
- personal care
- uncompromized quality

Fibertec's personnel include regulatory, consulting, and laboratory backgrounds. We understand many of your needs. That's why we pride ourselves on our ability to service, service, service customers. We promise this effort because we want your business and we want your business to be repeat business. Fibertec is staffed with highly skilled professionals with the vision of providing the best customer service in the industry. We believe bigger is not always better and have structured ourselves to be big enough to serve, yet small enough to care. On behalf of all the dedicated professionals at Fibertec Analytical, we welcome the opportunity to meet and exceed your expectations.

Fiberfec
Environmental
Services

Analytical Laboratory Equipment List

PROBE SAMPLING
MOBILE LABORATORIES

ANALYTICAL LABORATORY
ASBESTOS SERVICES

VOLATILE ORGANICS

2 - Hewlett Packard 5971 GC/MSD with Tekmar 16 & 32 position autosamplers
1 - Hewlett Packard 5890 GC/PID with Dynatech 30 position autosampler

SEMI-VOLATILE ORGANICS

2 - Hewlett Packard 5971 GC/MSD with 100 position autosamplers
1 - Hewlett Packard 5890 GC/FID/ECD with 100 position autosampler
1 - Hewlett Packard 1050 High Pressure Liquid Chromatography (HPLC)
1 - Gel Permeation Chromatography
1 - Zymark Turbovap sample concentrator

INORGANICS

1 - Perkin Elmer Elan 500 Inductively Coupled Plasma/MS w/144 position autosampler
1 - Perkin Elmer Inductively Coupled Plasma with 106 position autosampler
1 - Shimadzu AA/Graphite Furnace with 50 position autosampler
2 - Microwave Digestors with digestion vessels

MISCELLANEOUS EQUIPMENT

TCLP/SPLP extraction equipment with ZHE extractors, 10 position tumbler
Multiple fume hoods, balances, pH meters, and computers

Call (517) 699-0345 for pricing or scheduling information.

Appendix A

2280 Aurelius Road Holt, Michigan 48842 Telephone: (517) 699-0345 Facsimile: (517) 699-0388
29300 Wall Street Wixom, Michigan 48393 Telephone: (810) 348-5598 Facsimile: (810) 348-4896

ACORD. CERTIFICATE OF INSURANCE

DATE (MM/DD/YY): 7/30/96

PRODUCER

Kelter-Thorner Inc.
210 South Woodward - Suite 200
P.O. Box 1300
Birmingham, MI 48012-1300
810-540-3131

THIS CERTIFICATE IS ISSUED AS A MATTER OF INFORMATION ONLY AND CONFERS NO RIGHTS UPON THE CERTIFICATE HOLDER. THIS CERTIFICATE DOES NOT AMEND, EXTEND OR ALTER THE COVERAGE AFFORDED BY THE POLICIES BELOW.

COMPANIES AFFORDING COVERAGE

COMPANY A FIDELITY & GUARANTY INS. CO.

COMPANY B GENERAL STAR INDEMNITY CO.

COMPANY C

COMPANY D

INSURED

Fibertec, Inc.
2280 Aurelius Road
Holt, MI 48842

COVERAGES

THIS IS TO CERTIFY THAT THE POLICIES OF INSURANCE LISTED BELOW HAVE BEEN ISSUED TO THE INSURED NAMED ABOVE FOR THE POLICY PERIOD INDICATED. NOTWITHSTANDING ANY REQUIREMENT, TERM OR CONDITION OF ANY CONTRACT OR OTHER DOCUMENT WITH RESPECT TO WHICH THIS CERTIFICATE MAY BE ISSUED OR MAY PERTAIN. THE INSURANCE AFFORDED BY THE POLICIES DESCRIBED HEREIN IS SUBJECT TO ALL THE TERMS, EXCLUSIONS AND CONDITIONS OF SUCH POLICIES. LIMITS SHOWN MAY HAVE BEEN REDUCED BY PAID CLAIMS.

CO LTR	TYPE OF INSURANCE	POLICY NUMBER	POLICY EFFECTIVE DATE (MM/DD/YY)	POLICY EXPIRATION DATE (MM/DD/YY)	LIMITS	
A	GENERAL LIABILITY [X] COMMERCIAL GENERAL LIABILITY [ ] CLAIMS MADE [X] OCCUR [ ] OWNER'S & CONTRACTOR'S PROT	1MP301289384	7/30/96	7/30/97	GENERAL AGGREGATE	$ 2000000
					PRODUCTS-COMP/OP AGG	$ 2000000
					PERSONAL & ADV INJURY	$ 1000000
					EACH OCCURRENCE	$ 1000000
					FIRE DAMAGE (Any one fire)	$ 50000
					MED EXP (Any one person)	$ 5000
A	AUTOMOBILE LIABILITY [X] ANY AUTO [ ] ALL OWNED AUTOS [ ] SCHEDULED AUTOS [ ] HIRED AUTOS [ ] NON-OWNED AUTOS	1MP301289384	7/30/96	7/30/97	COMBINED SINGLE LIMIT	$ 1000000
					BODILY INJURY (Per person)	$
					BODILY INJURY (Per accident)	$
					PROPERTY DAMAGE	$
	GARAGE LIABILITY [ ] ANY AUTO				AUTO ONLY - EA ACCIDENT	$
					OTHER THAN AUTO ONLY:	
					EACH ACCIDENT	$
					AGGREGATE	$
A	EXCESS LIABILITY [X] UMBRELLA FORM [ ] OTHER THAN UMBRELLA FORM	1MP301289384	7/30/96	7/30/97	EACH OCCURRENCE	$ 1000000
					AGGREGATE	$ 1000000
						$ 10000
A	WORKERS COMPENSATION AND EMPLOYERS' LIABILITY THE PROPRIETOR/ PARTNERS/EXECUTIVE OFFICERS ARE [ ] INCL [ ] EXCL	2108388966	7/30/96	7/30/97	STATUTORY LIMITS	
					EACH ACCIDENT	$ 500000
					DISEASE - POLICY LIMIT	$ 500000
					DISEASE - EACH EMPLOYEE	$ 500000
B	OTHER ENVIRONMENTAL CONSULTANT'S PROFESSIONAL LIAB	1YG230350B NO POLLUTION EXCL.	2/15/96	2/15/97	$1,000,000AGGREGATE 1,000,000 PER CLAIM CLAIMS MADE FORM	

DESCRIPTION OF OPERATIONS/LOCATIONS/VEHICLES/SPECIAL ITEMS

CERTIFICATE HOLDER

CANCELLATION

SHOULD ANY OF THE ABOVE DESCRIBED POLICIES BE CANCELLED BEFORE THE EXPIRATION DATE THEREOF, THE ISSUING COMPANY WILL ENDEAVOR TO MAIL 10 DAYS WRITTEN NOTICE TO THE CERTIFICATE HOLDER NAMED TO THE LEFT, BUT FAILURE TO MAIL SUCH NOTICE SHALL IMPOSE NO OBLIGATION OR LIABILITY OF ANY KIND UPON THE COMPANY, ITS AGENTS OR REPRESENTATIVES.

AUTHORIZED REPRESENTATIVE

Marilyn Clarence

002298000

ACORD 25-S (3/93)

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APPENDIX B

STATEMENT OF QUALIFICATIONS

1998

Triangle Laboratories, Inc.
Laboratory Services

801 Capitola Drive
Durham, NC 27713
919-544-5729 (phone)

PO Box 13485
Research Triangle Park, NC 27709
919-544-5491 (fax)

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Mission Statement

Triangle Laboratories is in the business of applying scientific knowledge and measurement to the solution of health, environmental and other issues confronting society.

Beliefs

We believe that we must excel in relationships with our customers, our employees, and our investors while establishing leadership in our technology and operations management.

We believe that in all things and at all times our behavior must follow the highest ethical standards. This includes commitments made to customers, suppliers, employees, investors, and to one another.

We believe that to our customers, we must be the laboratory of choice. Our marketing program will always honestly inform. We will set the quality and timeliness standard in our markets. We will structure our company so that we have the flexibility and versatility required to be responsive to customers' needs. We will work until the customer is satisfied.

We believe that for our employees, we must be the employer of choice. Through the application of high ethical standards, maintenance of efficient operations and a respect for diversity, we will provide a work environment that enriches and builds people while giving them an opportunity to excel and enjoy the dignity, pride, and material rewards of being part of a winning team.

We believe that for our investors, we must commit to the development of long term value in their investment. This will be accomplished by taking those risks that have an appropriate probability of reward, controlling expenses to maintain high profitability, and aggressively seeking opportunities to achieve growth through expansion of existing business and developing new business opportunities.

We commit ourselves to conducting research and development so that we are always a leader in technology, to apply the knowledge gained to maintain efficient operations and to service our customers needs in a timely manner while providing a reasonable profit for our investors.

1. INTRODUCTION

Triangle Laboratories, Inc. (Triangle Labs) has a history of excellence in applying scientific knowledge and measurement technology to the solution of health and environmental issues. Since beginning operations in 1984 as a privately held corporation, Triangle Labs has set the standard for, and remains committed to, delivering high quality, legally-defensible analytical data. Building on our expertise in the trace analysis of organic compounds, specifically dioxins and furans, Triangle Labs has maintained a pattern of steady growth by: building relationships with strategic partners, continuing our commitment to research and development, and centralizing production in order to maximize the utilization of human resources, scientific instrumentation, and financial capital.

- ***Independent Ownership***

  Unlike many companies in the analytical chemistry services marketplace, which are held by larger companies that control their destiny, Triangle Labs remains an independent, self-directed corporation. More than 50 percent of our equity holders are employees of the corporation and as such, are actively committed to a long range vision of growth and stability.

- ***Special Expertise***

  Triangle Labs has assembled an international staff of unparalleled expertise in analytical sciences. Our experts offer a diversity of analytical skills, decades of experience in large volume analyses with a wide variety of enviro- and biomatrices and new method development skills. Our highly trained chemists are dedicated to finding solutions to our customers' complex analytical problems. Many of our experts have worked in the analytical sciences field for over 20 years and are called upon to teach at local universities, appear as expert witnesses, and/or visit our customers' sites as analytical consultants. Throughout our history, we have earned a reputation as a reliable out-sourcing center to other commercial, research, and government laboratories. Triangle Labs' highly trained staff has special strengths in high resolution mass spectrometry. We operate one of the largest high resolution mass

- ***Special Expertise*** *(cont.)*

spectrometry laboratories in the world.

Triangle Labs also provides the analytical expertise required by the pharmaceutical industry in developing new methods for the registration of new pharmaceutical products. By combining the environmental and pharmaceutical lines of business into a single production facility, we are able to recognize economies of scale and incorporate efficiencies into our production, management, and information systems. Our total network of instrumentation is maintained in an environment that contains reliable security systems, a quality control system throughout the process, and control of standards and reference materials. These systems are routinely inspected and calibrated by experienced operators and factory trained, in-house engineers.

- ***Commitment to Quality***

Triangle Labs is committed to providing the highest quality analytical data and strictly adheres to Quality Assurance/Quality Control (QA/QC) procedures. The Quality Assurance Department performs continuous monitoring of the quality systems to provide feedback to the highest level of management. Our laboratories operate in accordance with standard operating procedures (SOPs) that are designed to follow Good Laboratory Practice (GLP) standards wherever possible. Specific QA procedures govern all activities from sample receipt through data packaging and disposition. These procedures, available for client review, ensure the correct identification, integrity, and analytical results of any samples received by our laboratory. Moreover, the procedures ensure that the analytical results obtained from samples received by Triangle Labs are admissible in a court of law. Requests for a copy of the latest QA Manual can be made through Client Services at (919) 544-5729 ext. 260.

- ***Looking Ahead***

Triangle Labs continues to apply our knowledge and expertise in analytical chemistry to other business opportunities, both nationally and internationally, in order to meet the future needs of the industries we serve. We are expanding our pharmaceutical services to provide companies assistance in bringing new drugs to market. Through careful long-range planning, we are continuing to build and expand relationships with partners throughout the world, testing new protocols, and designing the next generation of methods and products in our Development Laboratories.

While we continue to invest in technology to meet service demands, we are committed to maintaining the human touch that is essential to long term relationships. By adhering to the fundamentals and focusing on what we do well, Triangle Labs will continue to add value for our customers. We are committed to being the best at providing analytical services for our clients, and our strong position provides a tremendous advantage as we face the future.

2. SCIENTIFIC EXPERTISE - OUR SENIOR STAFF

Triangle Labs operates one of the largest high resolution mass spectrometry laboratories in the world. Our mass spectrometry experts have decades of experience in large volume analyses of environmental and pharmaceutical samples. They have also authored hundreds of publications in the field of mass spectrometry and have contributed to thousands of publications on analytical chemistry methods and techniques. More detailed information on our expertise in environmental and pharmaceutical analyses is provided in Section 3. A listing of Triangle Labs' scientific instrumentation is provided in Section 4.

Our Senior Technical Management team has a rich history of research, teaching, management and business experience. As we move into our second decade of providing high quality service, these individuals are focused on continuing our corporate mission and maintaining the standard of excellence that they helped establish.

Ronald Hass, Ph.D. - President & CEO

Trained as an Analytical Chemist specializing in mass spectrometry, Dr. Hass received a B.A. in Chemistry from Appalachian State University in 1967 and a Ph.D. in Analytical Chemistry from the University of North Carolina at Chapel Hill. He has more than 17 years practical experience in the analysis of complex environmental samples, including 10 years as Mass Spectrometry Group Leader at the National Institute of Environmental Health Sciences. Dr. Hass has authored over 100 articles on mass spectrometry. He has given more than 30 seminars as an invited lecturer and has addressed colleagues at several international conferences. In 1996, Dr. Hass was appointed interim voting board member to the International Association of Environmental Testing Laboratories (IAETL). As a member of the Triangle Labs' five member start-up team, Dr. Hass has been President and Chairman of the Board of Directors of the company since its inception in 1984.

Donald Harvan - Vice President

Earning a B.S. in Chemistry from Michigan State University and an M.A. in Chemistry from Duke University, Mr. Harvan's background includes extensive applications of mass spectrometry. He was a chemist for 16 years with the National Institute of Environmental Health Sciences, prior to joining Dr. Hass as a co-founder of Triangle Labs. Mr. Harvan has authored over 50 articles on mass spectrometry and has successfully developed new methods of high-resolution GC/MS for the analysis of environmental and pharmaceutical samples. Mr. Harvan has held several key positions with Triangle Labs, including Vice-President of Operations and Quality Assurance Manager, and has also served on the Board of Directors of Triangle Labs since 1987. He is currently responsible for business development.

Phillip William Albro, Ph.D. - Laboratory Technical Director

Dr. Albro's training includes a B.A. in Chemistry from the University of Rochester and a Ph.D. in Biochemistry from St. Louis University. He has over 26 years professional experience with the National Institute of Environmental Health Sciences at the Research Triangle Park campus. Dr. Albro has extensive experience in analytical and preparative methods for unstable chemicals, analytical method development, bioanalysis and environmental sample analysis. He also has extensive experience designing methods for unusual and complex biological matrices. Evidence of this experience is his authorship in over 100 publications in the area of chlorinated organics. Since joining Triangle Labs in 1994, Dr. Albro has played an integral role in preparing the Research Triangle Park facility to handle the broad range of analyses required by our customers.

Mohammad Hamzavi, Ph.D. - Senior Development Chemist	Dr. Hamzavi received his Ph.D. in analytical chemistry from Duke University, NC in 1987. Dr. Hamzavi has over 12 years of experience in the bioanalytical industry. He most recently managed the development of testing protocol for over 60 drugs in biological fluids using: GC, HPLC, Immunoassays, TLC and GC/MS instrumentation. With Triangle Labs, he is responsible for method development and validation for individual bioanalytical projects and the production of validated methods.
Michael Chu - Senior Scientist	Mr. Chu has the equivalent of an M.S. in chemistry from the Slough College of Advanced Technology in England. Prior to joining Triangle Labs in 1986, he was responsible for GC/MS analysis and the proper functioning of the Adrenal Research Laboratory at Veterans Administration Hospitals and later the high resolution lab with the Neoplastics Diseases Division of The Mount Sinai Medical Center. As a Senior Scientist, Mr. Chu is responsible for technical oversight and guidance in the areas of method development and refinement. An expert in both high and low resolution mass spectrometric analyses, Mr. Chu is an invaluable technical resource for the instrumentation sections, as well as, the wet chemistry laboratories.

3. ANALYTICAL SERVICES

Triangle Labs is one of the leading analytical chemistry laboratories in the United States serving the environmental and pharmaceutical markets. The company has experienced steady growth over the last decade based primarily on the strength of our expertise in the detection and identification of hazardous compounds in environmental samples. Since its formation in 1984, Triangle Labs has developed a strong specialty for trace analysis by high resolution mass spectrometry and broadened our product lines to include dioxins, semi-volatile and volatile organics, pesticides, PCBs, inorganics and metals as required by environmental regulations.

A summary of analytical methods for environmental analysis is provided in Table 1. Many of our environmental samples are derived from stack sampling and feed analysis in incineration testing, ambient air testing, hazardous waste site cleanup and regulatory monitoring from manufacturing sites.

Triangle Labs serves the research pharmaceutical industry by providing analytical results for drugs of interest from a variety of biomatrices. The data are typically used for pharmacokinetic Phase I through Phase IV studies for reporting to the Food and Drug Administration (FDA). The first project of this type was initiated in 1991 and continues to the present. The Division uses GC/MS and LC/MS/MS methods, coupled with advanced sample preparation techniques to provide rapid, rugged, state-of-the-art assays. We are experienced using high resolution mass spectrometers and alternative ionization methods to achieve lower detection limits. Our expertise also includes assays for chiral compounds, using both GC and LC separations.

Good Laboratory Practice (GLP) compliance, extremely low detection limits, and customized client reporting are the hallmarks of Triangle Labs' pharmaceutical services. Through our project scientists, continuous contact with the client is maintained throughout studies. Some example assays for pharmaceutical analysis are provided in Table 2.

Project Scientists

Michael Allen
Mr. Allen received a B.S. in Biology from East Carolina University before joining Triangle Labs in 1991. Mr. Allen is experienced in analyzing samples using both high and low resolution gas chromatographs/mass spectrometers (GC/MS). Mr. Allen is currently working on method development and validation of bioanalytical sample assays. His duties include maintaining and operating the high pressure liquid chromatograph/MS/MS (HPLC/MS/MS), managing bioanalytical projects, and providing technical assistance to clients.

Carl Anderson
Mr. Anderson received a B.S. in Chemistry from NC Wesleyan College before joining Triangle Labs in 1990. Mr. Anderson is experienced in wet laboratory extraction and clean-up procedures and in the analysis of samples using GC/ MS. As a Project Scientist, his primary responsibilities are to manage air emissions and pharmaceutical projects.

Amy Boehm
Ms. Boehm joined Triangle Labs in 1991 as a Data Review Officer. As a project scientist, Ms. Boehm provides quick responses to technical issues of source emissions and pulp/paper companies. Ms. Boehm earned her BA degree in 1990 from University of North Carolina in Chapel Hill.

Shelia Lee-Lewis
Ms. Lee-Lewis started her career with Triangle Labs in 1990 as a Bench Chemist I. She was promoted to Project Scientist in 1997. In this position, Ms. Lee-Lewis serves as the primary technical interface on projects from environmental consulting and engineering firms and pulp/paper companies. She graduated from the University of North Carolina in Chapel Hill in 1989 with a BA degree in Biology.

Mary McDonald
Ms. McDonald's experience in handling client projects began in 1989 as Project Monitor. She was promoted to Project Manager in 1990 and Project Initiation Chemist in 1993. In 1995, Ms. McDonald was named to her current position of Project Scientist. She specializes in projects from the areas of remediation, risk assessment and base cleanups. Ms. McDonald received a B.S. in Biology from the University of North Carolina in Chapel Hill.

Rose West

Ms. West joined Triangle Laboratories of Houston in 1990 as a Sample Preparation Chemist and Sample Custodian. Since then, she has worked in various aspects of customer service and project management, including the Data Review area before becoming a Project Scientist in 1997. Ms. West received her BA degree, *cum laude*, from Wells College in 1989, where she majored in Chemistry with a Math minor.

Table 1. Summary of Analytical Methods for Environmental Analysis

Analyte	Method	Technique	Matrix	Year started
Dioxins/Furans	8290	HRGC/HRMS	soil, water, waste, other	1987
	1613A/B		water, soil	1990
	M23		air	1987
	551		water	1990
	TO 9		ambient air	1989
	8280/8280A	LRMS	soil, water, waste, other	1991
	613		water	1990
	DFLM01.1		soil, water, waste	1990
	8/88 SOW		soil, water, wipe	1993
	8270	HRGC/LRMS	soil, water, waste, other	1985
	TO 13		ambient air	1989
	MM 680		air, water, soil, other	1991
	MM 680	HRGC/LRMS		1986
	8080	GC/ECD	air, soil, water, waste, other	1986
	TO 4 / TO 10		ambient air	1989
Semivolatiles	8270	HRGC/LRMS	air, soil, water, waste, other	1985
Volatiles	8240	HRGC/LRMS	soil, water, waste, other	1985
	8260			1994
	5040 (VOST)		air effluents	1985
	TO 1		ambient air effluents	1985
Metals	6000-7000 series	ICP/GFAA/CVAA	air, water, soils, other	1986
Pesticides	8081	GC/ECD	soil, water, waste, other	1986
AOX/ TOX	1650A/B	Microcoulometry	water	1989
	9020			1989
	DIN 28409		pulp, solids, paper	1989
	DIN 38414/1650A			1989
Hexavalent	0013	Ion Chromatography	air, water	1993
Chromium	218.6			1993
HCl/cations/	300.0, 206		water	1992
NOx	206, 7D		air	1996
	26/26A			1992
	9056/9057			1992
Particulates	5, 202, 201A	Gravimetric	air	1995

Note: Methods used for analysis are from EPA's Office of Solid Waste, Office of Water, Office of Air Quality and Emissions Branch. Some of the methods used are developed internally since there are no existing promulgated methods. This is not a comprehensive list of all methods performed. Please call if you have a specific request.

Table 2. Bioanalyses for the Pharmaceutical Market

Compounds:	Method development and validation for client specific **proprietary compounds.** Some example generics include: **5-Fluorouracil, Chiral chlorpheniramine, Fentanyl, Lidocaine, Naltrexone, Thymidine, Uracil.**
Matrices:	**urine, blood, plasma, serum, tissue**
Assay ranges:	**Typical LLOQ ranges from 0.02 ng/ml to 0.1 ng/ml with ULOQs ranging from 10 to 20,000 ng/ml.**
Analytical techniques:	**LC/MS/SIM** **(liquid chromatography/MS/selected ion monitoring)** **LC/MS/MS/MRM** **(liquid chromatography/MS/MS/multiple reaction monitoring)** **GC/MS/SIM** **(gas chromatography/MS/selected ion monitoring)** **GC/HRMS/SIM** **(gas chromatography/high resolution MS/selected ion monitoring)** **GC/HRMS/NICI/SIM** **(gas chromatography/high resolution MS/negative ion chemical ionization/selected ion monitoring)**
Therapeutic Areas:	**Analgesic** pharmaceuticals Phases I-IV (32,000+ samples) **Oncological** pharmaceuticals Phases II-III (9,000+ samples) **Urological** pharmaceuticals Phases I-III (9,000+ samples) **Cardiovascular** pharmaceuticals Phases I-III (6,000+ samples) **Mental Illness** pharmaceuticals Phases II-III (6,000+ samples)
Capacity:	**40,000+** bioanalyses capability on an annual basis. **Redundant instrumentation:** 3 LC/MS/MS 7 GC/HRMS 9 quadrupole GC/MS
Experienced Staff:	Triangle Laboratories, Inc. is a **science based** company with more than **fifty years of combined research and development experience at the NIH.** More than **100 years of combined analytical chemistry experience** in the senior staff. More than **300 scientific publicatons and presentations** from the senior staff.

4. SCIENTIFIC INSTRUMENTATION

Triangle Labs' instrumentation laboratory contains one of the highest concentrations of research grade high resolution mass spectrometry instrumentation in the United States. Available scientific instrumentation is summarized in Table 3.

* **High Resolution Mass Spectrometry Laboratory**	Triangle Labs maintains seven VG Analytical high resolution gas chromatograph/ high resolution mass spectrometers that provide state-of-the-art research and analytical capabilities. These high resolution GC/ MS systems permit mass measurements with a precision of 5 parts per million (ppm) in selected ion recording mode. Sub-picogram analytical sensitivities are routinely available.
* **HPLC/MS/MS Laboratory**	Triangle Labs utilizes Quattro-II triple quadrupole mass spectrometers interfaced to a Hewlett Packard LC for LC/MS/MS analytical methods. The instruments are equipped with atmospheric pressure chemical ionization (APCI) and electrospray ionization (ESI) sources that provide the ability to analyze non-volatile compounds. The instrumentation is used for method development and assays for the pharmaceutical industry, and has been used to develop a new analytical method for explosives.
* **HPLC Laboratory**	A Waters LC Module 1 Plus high performance liquid chromatograph (HPLC) is used to perform the analyses for explosives by Method 8330. It also has applications in the field of bioanalysis.
* **Low Resolution Mass Spectrometry Laboratory**	The Low Resolution Mass Spectrometry Laboratory includes 9 quadrupole GC/ MS instruments. Three instruments are configured for volatile organic analyses, using a Tekmar LSC 2000 Purge and Trap. One instrument is dedicated to low resolution dioxin and furan analyses (Methods 613, 8280 and DFLM01.1). Five instruments are configured for

* **Low Resolution Mass Spectrometry Laboratory** (con't)	semivolatile and pharmaceutical analytical applications.
* **Gas Chromatography Laboratory**	The Gas Chromatography Laboratory contains four Hewlett Packard 5890A gas chromatographs (GC). Three of the GCs are fitted with dual capillary columns and electron capture detectors (ECD). The remaining GC also has dual capillary columns, as well as, an ECD and flame ionization (FID) detector. All of these GCs take advantage of Hewlett Packard 7673A robotic autosamplers. Two Maxima Chromatography data systems and two Hewlett Packard Chem Station data systems handle the data acquisition for all of the GCs while data processing is performed with software programs developed at Triangle Labs.
* **Inorganic Laboratory**	Due to the special procedures required for metals analyses the Inorganic Laboratory has its own sample preparation and glass washing areas. Hotplate and microwave digestions are also performed in this area. The primary inductively coupled plasma emission spectrophotometer (ICP) is a simultaneous Trace ICP from Thermo-Jarrell-Ash. The Trace ICP has instrument detection limits that are comparable to those that can be achieved by using graphite furnace atomic absorption (GFAA) and allows for the analysis of multiple metals in a single injection. A Perkin-Elmer Plasma 1000 sequential ICP is also available for sample analyses. Two atomic absorption spectrophotometers (AA) are available for metals analyses. Both systems are made by Perkin-Elmer and employ Zeeman background correction which is preferred by the EPA. Mercury analyses are performed by cold vapor AA using a Leeman Labs mercury preparation system and analyzer.

* **AOX/TOX and Ion Chromatography Laboratory**	Adsorbable Organic Halides (AOX) or Total Organic Halides (TOX) analyses use carbon adsorption of organic halides followed by combustion and microcoulometric titration to measure the organic halide content in water, soil and pulp matrices. A Xertex-Dohrmann model DX-20A instrument is used to perform the analyses following established protocols. Ion chromatography analyses use different ion exchange columns to isolate anions and cations that are detected by an electrical conductivity cell. A Dionex DX-300 system is used for these analyses. Hexavalent chromium analyses use a post column reaction techniques followed by detection by a UV/VIS spectrophotometer while common anion and cation analyses are performed using conductivity detection.
* **Gravimetric Laboratory**	The Gravimetric Laboratory contains three analytical balances, 12 desiccators, a five foot fume hood, oven and water bath used for sample concentrations. The laboratory is also isolated to keep it free from dust contamination, air drafts and is temperature and humidity regulated. Samples are processed following Methods 5, 5A-F, 17, 201A, 202 and others. Laboratory data are stored by and processed through the lab information system. Filters are weighed, tared, and labeled in this lab prior to being sent to the field site for sample collection.

* **Extraction Laboratory Equipment**	In excess of 10,000 square feet of laboratory space are used for sample preparation. Separate sample preparation locations are used for the handling of environmental samples to prevent contamination from high level to low level samples. Separate rooms are utilized for bioanalytical sample preparation. There is a separate laboratory area for Soxhlet extractions and concentrations of sample extracts. There is a capacity to perform over 100 Soxhlet extractions at one time in this area. Similarly, there is a separate area where column chromatography (mainly for the dioxin and furan analyses) are performed.
* **Sampling Kit and Media Preparation**	Isolated areas within the laboratory are used for the preparation of sampling kits and air sampling media. These locations separate the glassware and media from solvents and field samples. Sampling kits typically consist of amber glass bottles and jars, coolers, ice packs, labels, and chain-of-custody forms. All glassware meet EPA Level III criteria and is certified by the suppliers. Preservation chemicals are available as necessary. Air sampling media, including XAD, PUFs, VOST tubes, and filters are prepared and sent directly to the sampling location. The XAD traps and glass PUF sleeves are typically owned by the client and are maintained in inventory at the lab. The QC-certified VOST tubes are purchased in bulk from Supelco, Inc. All other media and materials are supplied by Triangle Labs and are subjected to QC testing as appropriate.

* **Management Information for the Lab Environment System (MILES)**

Triangle Labs maintains an internally developed lab information system, MILES, that meets the guidelines of the Good Automated Laboratory Practices (GALP) required by the EPA. The current system is utilized for all facets of laboratory information including quotations, sample receipt, project management, intra-laboratory communication, scheduling, tracking and data reporting. System security is ensured by passwords that are utilized to prevent access to the data files by unauthorized users. Software and hardware access checkpoints for levels of accessibility to the data are provided throughout the MILES programs to ensure only authorized personnel have the ability to write data files. In addition, the laboratory areas containing data terminals and software system access are secured by cypher locks. The combinations are changed on a monthly basis or as needed. The access codes are given only to active employees who require access to the laboratory areas.

Table 3. Summary of Scientific Instrumentation

Equipment Type	Model Name	Applicable Methods
High Resolution GC/MS	VG Analytical Autospec	
High Resolution GC/MS	VG Analytical 70 S	1613A, 8290, 23, 551
High Resolution GC/MS	VG Analytical 70 S	
High Resolution GC/MS	VG Analytical 70 SE	Unchlorinated Dioxins/Furans
High Resolution GC/MS	VG Analytical 70 SE	Pharmaceutical/Bioanalysis
High Resolution GC/MS	VG Analytical 70 VS	
High Resolution GC/MS	VG Analytical 70 EQ	DFLMO1.1, 8280, 613
Low Resolution GC/MS	Hewlett Packard MSD 5971A	
Low Resolution GC/MS	Hewlett Packard MSD 5971A	
Low Resolution GC/MS	Hewlett Packard MSD 5971A	8270, 680, TO-13
Low Resolution GC/MS	Hewlett Packard MSD 5971A	Pharmaceutical/Bioanalysis
Low Resolution GC/MS	Hewlett Packard MSD 5971A	
Low Resolution GC/MS	VG Analytical 12-250	DFLMO1.1, 8280, 613
Low Resolution GC/MS	VG Analytical TRIO-1	
Low Resolution GC/MS	VG Analytical TRIO-1	8240, 8260, 5040/5041, TO-1
Low Resolution GC/MS	VG Analytical TRIO-1	
Gas Chromatograph	Hewlett Packard 5890 dual column ECD	
Gas Chromatograph	Hewlett Packard 5890 dual column ECD	8080, 8081
Gas Chromatograph	Hewlett Packard 5890 ECD/FID	Used for sample screening and special project
Gas Chromatograph	Hewlett Packard 5890 ECD/FID	Total chromatographable organics (TCO)

Equipment Type	Model Name	Applicable Methods
HPLC/MS/MS	VG Quattro - II	Pharmaceutical/Bioanalysis
HPLC/MS/MS	VG Quattro - II	Pharmaceutical/Bioanalysis
HPLC/MS/MS	VG Quattro - II	Pharmaceutical/Bioanalysis
Inductively Coupled Plasma Spectrometer	Thermo Jarrell Ash TRACE ICP	6010A/B
Inductively Coupled Plasma Spectrometer	Perkin-Elmer P1000	6010A/B
Atomic Absorption Spectrometer	Perkin-Elmer 5100Z	
Atomic Absorption Spectrometer	Perkin-Elmer 4100Z	7000A Series
Mercury Prep System	Leeman Labs AP200	
Mercury Analyzer	Leeman Labs PS200	7470/ 7471
Ion Chromatograph	Dionex DX-300	300.0, 9056/ 9057, 26/ 26A, 7A/ 7D, 0013, 218.6
Organic Halide (AOX/TOX) Analyzer	Dohrmann DX-20A	1650A, 38409, 38414, PTS RH 11/ 90, 9020, 450.1, APHA 5320B, ISO 9562
High Performance Liquid Chromatograph (HPLC)	Waters LC Module 1 Plus	
Microwave Digestion	CEM MDS-2000	metals digestions
Accelerated Solvent Extraction	Dionex ASE 200	Solvent extractions for solids & tissue
Speed-vac	Savant ASE 2000	Concentration of dioxin and furan sample extracts

5. International Operations

Triangle Laboratories, an independent, employee-owned contract laboratory founded in 1984. has grown into a multi-site, international organization with high technology products and services to fill a broad range of scientific needs. Triangle conducts government and commercial investigations for assessment, research, and remediation. Industrial testing is conducted for all areas of manufacturing.

Brief History

Since 1991, Triangle Laboratories has built many alliances throughout the world as a direct result of its reputation for expertise in trace analysis for organic compounds, specifically polychlorinated dibenzo-p-dioxins and polychlorinated-dibenzofurans.

Africa

The Technology Development Unit of the **Atomic Energy Corporation of South Africa** and Triangle Labs entered into an exclusive agreement in 1994 for the establishment of technologies for dioxin analysis in South Africa. Prelindaba Toxilabs and Triangle Labs provide analyses which support new product contract research, risk assessment projects, and toxicology studies.

Asia-Pacific Rim

In 1991, Triangle Labs and **Mitsubishi Petrochemical** of Tokyo signed a Memorandum of Understanding which gave Triangle representation in Japan. That partnership, now under the Mitsubishi Chemical name, offers environmental measurements, new chemical investigations (including pharmaceuticals), and high resolution mass spectrometry analyses to serve industrial and governmental clients.

In 1996, Triangle Labs and **Young-In Scientific Company**, headquartered in Seoul, signed a Representative Agreement which gives Triangle Labs exclusive representation in the South Korean environmental market. Young-In is one of the largest distributors of scientific instruments in Korea, and with 20 years of experience, has become known as a problem solver and technical adviser with additional lines of business serving the analytical, training, and research and development needs of their customers.

GM Laboratories Pty Ltd., of Sydney Australia, offers a full range of environmental sampling and measurement services through its affiliation with Triangle Labs. Special services include water and waste testing, plus waterway and harbor characterization.

Western Europe

Triangle Laboratories U.K., Ltd. operates one of the most advanced chemical testing laboratories in Europe, featuring ultra trace analysis by high resolution mass spectrometry with gas and liquid chromatography for product characterization, new drug trials, research and development, or environmental testing. Triangle Laboratories U.K., Ltd. offers special services in Europe that include:

- GC and LC Column Development
- Method Development for Chemical Analysis
- Source Sampling Kits and Sample Handling
- Trace Dioxin, Furan, PCB Analysis in Air, Biota, Water and Waste Samples
- Voluntary Standard Consultation (BS 7750, EMAS, ISO 14000)
- Training Program with University Offering Credits Toward Professional Development Award

Triangle Laboratories U.K., Ltd. continues to work with regulators in the United Kingdom to assist them in evaluating the dioxin and furan data from stack emissions. Triangle Laboratories U.K., Ltd. provides guidance concerning data quality similar to that used in U.S. EPA methods and are working in conjunction with the regulators to identify dioxin and furan emission patterns by source type.

Mexico, Central America and the Caribbean

PROA, a Mexican conglomerate, and Triangle Labs maintain an extensive sampling network with full service environmental analysis in Mexico. Backed by rigorous quality control procedures and high resolution measurements, **Laboratorio de Ecologia Industrial** conducts major industrial and government projects. Triangle Labs was contracted in early 1996 by a Mexican governmental agency to conduct the first ever compliance trial burns for that country. Method 23 and HCl samples from two hazardous waste incinerators were collected by Laboratorio de Ecologia Industrial, with Triangle Labs performing the sample recovery on-site and the analysis at its central production facility in Research Triangle Park, North Carolina.

South America

RIOCELL, a leading Brazilian wood products producer located in the state of Rio Grande do Sul, and Triangle Labs provide specialized environmental services for the Brazilian pulp and paper industry with regards to project planning, sampling, analysis, data review and evaluation services.

In 1997, **Estudos E Avaliacões Ambientais Ltda.** (ECONSULT) and Triangle Labs formalized their relationship to provide specialized environmental services in Brazil for the air industry. Specialized services also include due diligence testing, environmental background tests, waterways and harbor characterization, and wastewater testing.

6. PROJECT EXPERIENCE

Triangle Labs provides analytical and technical support to a variety of customers. Our laboratory analyzes thousands of samples each month, and the following information reflects the general categories of sample analysis.

- ***Air Quality Product Line***

Triangle performs analyses from many types of sampling trains, including Method 23 (M23); Modified Method 5 (MM5); Volatile Organic Sampling Train (VOST) and Multi-metals Train (MMT). Air quality samples may also come from impingers and filters used for the collection of particulates and HCl/ Cl_2.

We have analyzed thousands of samples in support of incinerator trial burns under RCRA guidelines to demonstrate Boiler and Industrial Furnace (BIF) compliance. These projects have included analyses for dioxins/ furans, semivolatiles, volatiles, metals and HCl, ammonia and cations.

In addition to trial burns, air samples collected for engineering or emission inventory purposes are also received for analysis. The emission inventory projects usually involve evaluation of non-standard analytes. Method modification of existing analytical methods or new method development is a capability that Triangle Labs provides for our clients.

- ***Water Quality Product Line***

Our Water Quality product line includes analyses of samples from surface water, waste water and drinking water. Also included in this category are sediments, sludges and fish that are monitored as required by the National Pollutant Discharge Elimination Systems (NPDES) permit program. Monthly and quarterly analyses are performed for

- ***Water Quality Product Line*** *(cont.)*

effluents for dioxins and furans using Method 551 and 1613A for compliance with permits issued under regulations from the Clean Water Act. For these

types of projects, sampling kits are available to send to the collection sites in order to expedite and simplify the sample collection process for the client.

We are certified in 29 states to perform analyses for 2,3,7,8-TCDD by Method 1613A in drinking water. In addition to drinking water, we also perform analyses for 2,3,7,8-TCDD in bottled water as required by the International Bottled Water Association.

Routine analyses are performed for AOX/TOX as part of ongoing compliance requirements for the pulp and paper industry. We have participated in method studies conducted by the EPA and the National Council of Paper Industry for Air and Stream Improvement (NCASI).

- ***International Project Experience***

Triangle routinely analyzes samples from Europe, Southeast Asia, Australia, South Africa, Mexico and South America. Through collaboration with our international sales partners, we are able to efficiently manage complex sampling and analysis projects, ship samples to our laboratory and provide the analytical results in a timely fashion. Triangle Labs has analyzed sediment samples from the Homebush Bay site on the Parramatta River in Australia. This site had the reputation of being one of Australia's most contaminated sites, but has now been chosen for the 2000 Olympics under the theme of "the Green Games." Triangle Labs continues to analyze samples as the sites around the Bay are remediated.

- ***International Project Experience (cont)***

Triangle Laboratories U.K., Ltd., provides project management and technical support for United States sampling firms collecting stack samples from various European locations. Triangle Laboratories U.K., Ltd., provides needed reagents to the site, coordinate the sample collection and analysis and

provides client-specific formatted data reports.

- ***Risk Assessment***

Risk Assessment projects conducted to determine health or ecological risk factors are becoming more common as government agencies and private clients use these studies as a means to reduce unnecessary environmental cleanups. Normally, these types of projects require analyses from non-standard matrices (tissue, milk, etc.) and lower than normal analyte detection limits.

Our experience in working with non-standard matrices and our ability to achieve low limits of detection using high resolution mass spectrometry, allows us to help solve many of the difficult technical issues that can be associated with risk assessment projects.

We have years of experience in analyzing biomatrices, such as fish, mussels, birds, rodents, jack rabbits, mollusks, crayfish, insects, vegetation, milk products, blood, serum, adipose and various other tissues. Methods have been developed for a variety of analytes in these matrices, including, dioxins, drugs of interest, semivolatile and volatile compounds, metals, and explosive compounds.

- ***Department of Defense Cleanup Activities***

Triangle Labs has been awarded numerous analytical service contracts by large Department of Defense contractors. We have analyzed hundreds of samples from international defense cleanup sites where oversite was conducted by the Air Force Center for Environmental Excellence, the U.S. Army Corps of Engineers and the Naval Facilities Engineering Command.

- ***Bioanalysis for Pharmaceutical Product Line***

Triangle Labs has provided analysis of biological samples for drugs and their metabolites for customers in the pharmaceutical industry since 1991. We have developed and validated methods using a diverse combination of low and high resolution GC/MS, LC/MS, and LC/MS/MS instrumentation.

For example, an assay that had been developed by a client using low resolution MS methods, was adapted by Triangle Labs to high resolution MS technology. This resulted in a gain in sensitivity, specificity and ruggedness for the assays. More than 10,000 patient samples have been analyzed by this method, without method failure. Similar assays have been developed using low resolution GC/MS, negative ion chemical ionization high resolution GC/MS, and LC/MS/MS methods with atmospheric pressure chemical ionization and electrospray ionization techniques. The laboratory has many years experience with handling a large number of difficult biomatrices. Some of the more routine sample cleanup techniques include: protein precipitation with liquid-liquid extraction, solid phase extractions, and dual switching valves for on-line sample cleanup at the LC/MS. The laboratory has also been involved with the development of chiral assays, using both GC/MS and LC/MS techniques.

APPENDIX A
CERTIFICATIONS AND REGISTRATIONS

ENVIRONMENTAL:

American Association for Laboratory Accreditation. Accredidation pending. Certificate Number 0226-01. Accreditation for technical competence in Environmental Testing. (Including Waste Water, Sol/Haz Waste, Pulp/Paper, and Air Matrices) Parameters are AOX/TOX, and Dioxin/Furan. Method 1613 for Drinking Water. **Pending New Certificate.**

State of Alabama - Department of Environmental Management. Expires December 31, 1998. Laboratory I.D. # 40950. Dioxin in drinking water.

State of Alaska - Department of Environmental Conservation. Expires December 21, 1998. Certificate number OS-006-98. Dioxin in drinking water.

State of Arizona - Department of Health Services. Expires May 26, 1998. Certificate #AZ0423. Drinking Water for Dioxin, Dioxin in WW and S/H Waste. **Pending New Certificate.**

State of Arkansas - Department of Pollution Control and Ecology. Expires February 19, 1999. Pulp/paper, soil, water, and Hazardous Waste for Dioxin/Furan; AOX/TOX, Volatiles, Semi-volatiles, and Metals.

State of California - Department of Health Services. Expires August 31, 1999. Certificate #1922. Selected Metals in Waste Water; Volatiles, Semi-volatiles, and Dioxin/furan in WW and Sol/Haz Waste. Dioxin in drinking water. **Pending New Certificate.**

State of Connecticut - Department of Health Services. Expires September 30, 1999. Registration # PH-0117. Dioxin in drinking water.

Delaware Health and Social Services. Expires December 31, 1998. Certificate #NC 140. Dioxin in drinking water.

Florida Department of Health and Rehabilitative Services. Expires June 30, 1998. Dioxin in DW. Drinking Water ID HRS# 87424. Metals, Extractable Organics (GC/MS), Pesticides/PCB's (GC) and Volatiles (GC/MS) in Environmental Samples. Environmental water ID HRS# E87411.

Hawaii Department of Health. Expires March 1, 1999. Dioxin in drinking water. "Accepted" status for regulatory purposes.

Idaho Department of Health and Welfare. Expires December 31, 1998. Dioxin in drinking water.

State of Kansas - Department of Health and Environment. Expires January 31, 1999. Method 1613 for drinking water. ID #'s - Drinking water and/or pollution control - E-10215. Solid or Hazardous Waste - E-101209.

Commonwealth of Kentucky, Department for Environmental Protection. Expires December 31, 1998. ID#90060. Dioxin in drinking water.

Maryland Department of Health and Mental Hygiene. Expires September 30, 1998. Certification #235 Drinking water by Method 1613A.

State of Michigan - Department of Public Health. Expires June 30, 1998. Drinking water by Method 1613.

Mississippi State Department of Health. No expiration date. Dioxin in drinking water.

Montana Department of Health and Environmental Services. Expires December 31, 1998. Dioxin in drinking water.

State of New Jersey - Department of Environmental Protection and Energy. Expires June 30, 1998. ID #67851. BNAs and Volatiles. Dioxin in drinking water.

State of New Mexico - Environment Department. Still certified, awaiting information from A2LA Dioxin in drinking water. **Pending New Certificate.**

New York State Department of Health. Expires June 30, 1998. ID #11026. Environmental Analyses of non-potable Water, Solid and Hazardous Waste. Method 1613 in DW **Pending New Certificate.**

State of North Carolina - Department of Environment Health and Natural Resources Expires August 31, 1998. Certificate # 37751. Dioxin in drinking water.

State of North Carolina - Department of Environment, Health, and Natural Resources, Division of Environmental Management. Expires December 31, 2000. Certificate # 485. Metals, pesticides & PCBs, semi-volatiles and volatiles; TCLP.

North Dakota State Department of Health and Consolidated Laboratories. Expires December 31, 1998. Certificate # R-076. Effective October 4, 1993. Dioxin in drinking water.

Oklahoma Department of Environmental Quality. Expires August 31, 1998. Laboratory #9612. Dioxin by 1613A, 8290 and 8280.

State of South Carolina - Department of Health and Environmental Control. Expires June 30, 1998. Certificate number #99040001 (drinking water). Expires August 31, 1999. Certificate number #99040002 (other parameters). Dioxin/Furans, BNA, Volatiles, and PCBs/pesticides under Clean Water Act, 2,3,7,8-TCDD for Drinking Water, and Organic extractables for Solid and Hazardous Waste.

State of Tennessee - Department of Environment and Conservation. Expires February 5, 1999. ID #02992. Method 1613 Drinking water only.

U.S. Department of Agriculture Soil Permit. Expires September 30, 2001. Permit No. S-3790. Under the authority of the Federal Plant Pest Act, permission is granted to receive foreign soil samples for use in laboratory analysis.

U.S. Army Corps of Engineers. Expires October 19, 1999. Validated to perform analyses for the Fort Belvoir, VA (Contract Number DACA31-97-D-0029), Vint Hill Farms Station, Vint Hill, VA (Contract Number DACA31-95-D-0083), and Selma Pressure Treating Superfund Site, Selma, CA (Contract number DACW45-94-D-0054).

U.S. EPA Region V. Expires November 14, 1999. Dioxin in drinking water.

U.S. EPA Region VIII for the State of Wyoming. Expires November 12, 1998. Dioxin in drinking water.

State of Utah - Department of Health. Expired May 30,1998. Certificate Number E-166. Certification for the following parameters: Semi-Volatiles and Volatiles under RCRA; Volatiles under Clean Water Act; Dioxin/furans by Method 8280; Drinking water for Dioxin by Method 1613; Metals including Mercury and Microwave Digestion. **Pending New Certificate.**

Commonwealth of Virginia - Department of General Services, Division of Consolidated Laboratory Services. Expires June 30, 1998. ID # 00341. Dioxin in drinking water.

State of Washington - Department of Ecology. Expires September 11, 1998. Lab Accreditation Number C067. Scope of Accreditation applies to water analyses for

Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans, BNA Extr (Semivolatile) Organics and Purgeable (Volatile) Organics.

State of Washington - Department of Health. Expires April 30, 1999. Dioxin in drinking water. Lab I.D. 129.

State of West Virginia - Department of Health. Expires December 31, 1998. Certificate No. 9923(C). Dioxin in drinking water.

State of Wisconsin - Department of Natural Resources. Expires August 31, 1998. Laboratory ID Number 999869530. Certification for the following categories of Organics: Purgeable, Base/Neutral, Acid, PCBs, and Dioxin. Expires November 14, 1999. Laboratory ID 999869530. Dioxin in drinking water.

PHARMACEUTICAL:

Drug Enforcement Agency (DEA). Expires November 30, 1998. Registration number RT01195835. Controlled substance registration for schedules 1,2,3,3N,4,5.

N.C. Department of Human Resources. Expires October 31, 1998. Registration number NC-PT 0000 0031. North Carolina controlled substances registration. Application submitted for renewal.

Food & Drug Administration (FDA) Registration. Expires June 1998. ID #'s 001500 1053481. Annual registration of drug establishment. Annual registration of drug establishment.

OTHER:

Clinical Laboratory Improvement Amendments (CLIA) Registration. Expires May 30, 1999. ID # 34D0705123. Department of Health & Human Services, Health Care Financing Administration.

U.S. EPA Large Quantity Hazardous Waste Generator. No expiration date. EPA ID #NCD982156879. Permit indicates that the laboratory is a large generator of hazardous waste.

North Carolina General License for Radiation Protection. No. expiration date. License No. 032-875-OG. The general license applies only to radioactive material contained in devices which have been manufactured and labeled in accordance with specific requirements.

APPENDIX B
INTERNATIONAL CONTACTS

Africa
Prelindaba Toxilabs
PO Box 582
Pretoria 0001
Republic of South Africa
Contact: Dr. Willie van Niekerk
Phone: 27-12-316-3270
Fax: 27-12-316-2102
Email: infotox@icon.co.za

Pacific Rim
Mitsubishi Chemical Corporation
Functional Materials Company
5-2, Marunouchi 2-Chome
Chiyoda-Ku, Tokyo 100 Japan
Contact: Mr. Taketoshi Fujita
Phone: 81-3-3283-5252
Fax: 81-3-3283-5828
Email: 3503781@cc.m-kagaku.co.jp

South America
Riocell Technology Center
Rua Sao Geraldo, 1680
Caixa Postal 108
DEP 93500-000
Guaiba-Rio Grande do Sul, Brazil
Contact: Ms. Claudia Alcaraz Zini
Phone: 55-51-480-7342
Fax: 55-51-480-7121
Email: cazini@nt.riocell.com.br

Estudos E Avaliacões Ambientais Ltda.
R. Joaquim Nabuco, 1637
04621-000-Sao Paulo, Brazil
Contact: Mr. Marco A. Fabiani
Phone: 55-11-530-4421
Fax: 55-11-530-9644

Western Europe
Triangle Laboratories of the UK, Ltd
Millbrook Business Centre
Floats Road, Wythenshawe
Manchester M23 9YJ, United Kingdom
Contact: Keith Hall
Phone: 44-161-286-7884
Fax: 44-161-286-7676
Email: Keith_Hall_GC2@compuserve.com

Mexico, Central America and Caribbean
Laboratorio de Ecologia Industrial, S.A.
Alfonso Herrerà No. 75
Col San Rafaęl
06470 Mexico, D. F. , Mexico
Contact: Ing. Enrique Ordonez
Phone: 52-5-535-8930
Fax: 52-5-592-7069
Email: 74173.162@compuserve.com

Korea
Young-In Scientific Ltd.
Young-Hwa Building, 547
Sin Sa-Dong, Kang Namku
Seoul, Korea
Contact: Duck-Hee Lee
Phone: 82-2-547-7771
Fax: 82-2-547-7933
Email: younginc@chollian.dacom.co.kr

@ AIR TOXICS LTD.

LABORATORY

STATEMENT OF QUALIFICATIONS

AIR TOXICS LTD.
180 BLUE RAVINE ROAD, SUITE B
FOLSOM, CA 95630
916/985-1000

APRIL 1998

Unequaled Technical Services. The laboratory is staffed by experienced analysts who have worked with all kinds of air samples and is equipped with a variety of equipment specifically designed or modified for the analysis of air. This unique combination of experience and equipment ensures that 'special projects' become routine and data quality objectives are met. In addition, the analysts are able to modify existing methods to add analytes to virtually every standard Target Compound List (TCL). For example, ATL has validated over 80 compounds using TO-14 protocols.

ATL is one of the few commercial environmental laboratories with a group devoted to method development. This group works to refine and extend existing methodologies and develop innovative sampling and analysis procedures – procedures to simplify sampling or enhance data quality. For example, their efforts produced a CARB 430/EPA TO-5 modification for soil gas analysis, which permits determination of ppbv levels of vinyl chloride in the presence of other high level (ppmv) VOCs. They have modified the sulfur chemiluminescence detector hardware to improve its stability and sensitivity. In short, ATL is able to continually develop new methods and add new services based on client needs and expectations.

Air Toxics has unique mobile capability. Our mobile laboratory is equipped with state-of-the-art GC and GC/MS instrumentation, which is specially configured to analyze air samples. It is staffed by our most experienced analysts, who are able to both analyze samples and review data. The mobile laboratory has sufficient communications capability to send and receive data from our laboratory in Folsom, which means that raw data can be reviewed by QA personnel immediately, if necessary. Special report software has been written on a project specific basis, so that reports, in the client's format, are available at the end of the day.

Risk assessment quality data is available to our clients in hours, rather than days. Decisions can be made, at the site, based upon fully defensible data. Projects are completed on time and on budget. It is like having all the capability of our Folsom laboratory at the site.

Unsurpassed Client Support. ATL goes beyond simply providing a number; we form partnerships with our clients. Our Client Services Group is staffed with knowledgeable chemists who know the methods and are experienced in answering the types of questions that arise when dealing with air sampling projects. They work with our clients to define the project requirements, with our laboratory staff to communicate those requirements and with our QA staff to ensure that we meet the requirements.

Our objective is to provide our clients with useful data to meet their specific needs. QA results are included with the standard data package and our report formats offer virtually unlimited flexibility, from special reporting units to a variety of diskette data deliverables.

US EPA Compendium Ambient Air Methods

EPA TO-1	Volatile Organic Compounds (VOC)
EPA TO-2	Volatile Organic Compounds (VOC)
EPA TO-3	BTEX/TPH
EPA TO-4	Pesticides/PCBs
EPA TO-5	Aldehydes and Ketones
EPA TO-6	Phosgene
EPA TO-8	Phenols and Cresols
EPA TO-10	Pesticides/PCBs
EPA TO-11	Aldehydes and Ketones
EPA TO-12	Non-methane Organic Compounds
EPA TO-13	Polynuclear Aromatic Hydrocarbons (PAH), Semivolatiles (SVOC)
EPA TO-14	Volatile Organic Compounds (VOC)
EPA TO-15	Volatile Organic Compounds (VOC)

US EPA Stationary Source Methods

EPA 18	Gaseous Organic Compounds
5041 "VOST"	Volatile Organic Compounds (VOCs)
EPA 25C	Non-methane Organic Compounds
EPA 3C	Atmospheric Gases
EPA 15/16	Reduced Sulfur Compounds
EPA 5	Moisture
Modified 5	Semivolatiles, PAHs
BIF 0011	Formaldehyde, Other Carbonyl Compounds
BIF 0100	Formaldehyde, Other Carbonyl Compounds

CA Air Resources Board Stationary Source Methods

CARB 410A	Low Level Benzene
CARB 410B	High Level Benzene
CARB 422	Halogenated Solvents
CARB 430	Formaldehyde and Acetaldehyde
CARB 15	Reduced Sulfur Compounds

ASTM Methods

D-1945	Light Hydrocarbon Speciation
D-1946	Atmospheric Gases, Ethane, Ethylene
D-3416	Atmospheric Gases and Hydrocarbons
D-5504	Reduced Sulfur Compounds - extended list

Miscellaneous Methods

EPA 8270	Semivolatiles
Calderon	Landfill VOCs
NIOSH 1500	Volatile Organic Compounds
NIOSH 1501	Total Petroleum Hydrocarbons
NIOSH 1550	Total Petroleum Hydrocarbons - Diesel
NIOSH 2000	Methanol
EPA 24/24A	Inks and Coatings
ASTM 3810	Methane, Ethane, Ethylene in water (headspace)

Custom Method Development, per client request

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Capacity

ATL is positioned to successfully manage large scale, high volume projects. The laboratory exclusively performs air analysis and therefore dedicates its instrumentation to these analyses. ATL currently owns one of the largest canister and flow controller inventories in the United States. Capacity for TO-14 analysis, for example, is unmatched by any other laboratory facility.

Normal operating hours are 12 hours/day, 6 days/week. However, if necessary to meet project requirements, the laboratory can be staffed up to 24 hours/day, 7 days/week.

Selected Analytical Methods and Laboratory Capacity

Volatile Organic Compounds (VOCs)

Method	Analytical Instrumentation	MDL	Weekly Capacity
EPA TO-1	GC/MS Full Scan	10 ng	60
EPA TO-2	GC/MS Full Scan	10 ng	60
EPA TO-14	Cryofocused GC/MS - Full Scan Quadrupole - SIM Quadrupole	 0.5 ppbv 0.2 ppbv	 360 210
EPA TO-15	Cryofocused GC/MS - Full Scan Quadrupole - SIM Quadrupole	 0.5 ppbv 0.2 ppbv	 360 210
EPA 5041 "VOST"	GC/MS Full Scan	10 ng	60
CARB 422	Cryofocused GC/MS Direct Inject GC/MS	1.0 ppbv 0.5 ppmv	180 180
EPA TO-3	Cryofocused GC/FID/PID	1.0 ppbv 10 TPH	210
CARB 410A	Cryofocused GC/MS GC/PID	1.0 ppbv	210
CARB 410B	GC/PID	100 ppbv	210
EPA TO-12	Cryofocused GC/FID	1.0 ppbv	240
EPA 18	GC/FID/PID/ECD	per analyte	210
EPA 25C	Cryofocused GC/FID	10 ppmvC	48
Calderon	GC/MS	0.2-500 ppbv	200

Aldehydes and Ketones

Method	Analytical Instrumentation	MDL	Weekly Capacity
EPA TO-5	HPLC/UV	0.1 ug	300
EPA TO-11	HPLC/UV	0.1 ug	300
BIF 0011	HPLC/UV	1.0 ug	300
BIF 0100	HPLC/UV	0.1 ug	300
CARB 430	HPLC/UV	0.1 ug	300

Semi-Volatile Compounds (SVOCs)

EPA TO-13	GC/MS Full Scan, SIM or GC/FID	0.5 ug	120
EPA 8270	GC/MS Full Scan	1 ug	120
EPA TO-4	GC/ECD	< 1 ng	120
EPA TO-10	GC/ECD	< 1 ng	120
EPA TO-8	HPLC/UV	1.0 ug	360
Mod. EPA 5	GC/MS	1.0 ug	60

Atmospheric Gases/C1-C6 Hydrocarbons

ASTM D-3416	GC/TCD/FID	1-100 ppmv	150
ASTM D-1945	GC/TCD/FID	10 ppmv	60
ASTM D-1946	GC/TCD	1-100 ppmv	60
EPA 3C	GC/TCD	1-10 ppmv	150

Reduced Sulfur Compounds

EPA 15/16	GC/FPD	50 ppbv	120
CARB 15	GC/FPD	50 ppbv	120
ASTM D-5504	GC/SCD	2 ppbv	120

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Figures reflect analytical capacity available for actual client field samples – based on ATL's normal 6 days/week, 12 hours/day laboratory operating schedule. Tuning and calibration and other routine QA/QC requirements, as well as a 4.2% downtime allocation, are included in the capacity calculations. Larger project volumes can be accommodated.

Instrumentation and Equipment

ATL has outfitted the laboratory with state-of-the-art instrumentation and equipment. Much of the commercially available equipment has been modified in order to enhance performance or reliability. In addition, our expert in-house staff have designed and built numerous innovative pieces of instrumentation in order to address laboratory processing challenges or meet unique project needs:

- to overcome challenging sampling problems;
- to analyze difficult matrices;
- to increase data process/review efficiency;
- to produce more reliable results; or
- to achieve greater sensitivity.

Instrument maintenance and repair log books are maintained for each instrument. Service contracts are in place for the GC/MS systems. The laboratory is equipped with sophisticated Pentium-based workstations. Centralized data reduction is performed on a high speed Hewlett-Packard UNIX system.

In addition to the fixed laboratory instrumentation, the field analytical group has a number of portable GCs and other instrumentation designed specifically for on-site analysis.

ATL maintains an extensive inventory of sampling containers, media and equipment, including:

Sample Media	Quantity
Summa air sampling canisters	
6-Liter canister	1000
1-Liter canisters	400
Flow Controllers for air sampling canisters	300
Veriflo 24-hour flow controllers for canisters	50
Vaccuum gauges	60
Tedlar bags: 1, 3, 10 liter	In inventory
High Volume PUF/XAD air sampling traps	150
VOST tubes kept in inventory	500 pair

Instrumentation and Equipment

Description	Acquired
Hewlett-Packard 5973 GC/Mass Selective Detector	1997
Hewlett-Packard 5970 GC/Mass Selective Detector	1989
Hewlett-Packard 5971 GC/Mass Selective Detector [2]	1991/1993
Hewlett-Packard 5972 GC/Mass Selective Detector with HP 7673 Autosampler [3]	1994/1996/ 1997
Varian Ion Trap GC/MS	1994
Hewlett-Packard 5890 GC/FID/TCD Valved Special Analyzer	1990
Hewlett-Packard 5890 GC/FID/TCD Custom Oxidizer/Reducer	1996
Hewlett-Packard 5890 GC/FID/TCD	1996
Hewlett-Packard 5890 GC/FID/FPD	1990
Hewlett-Packard 5890 GC/FID/SCD	1994
Hewlett-Packard 5890 GC/FID/PID [2]	1990/1992
Hewlett-Packard 5890 GC/ECD/PID	1992
Hewlett-Packard 5890 GC/ECD/ECD [2] with HP 7673 Autosampler	1993/1995
Shimadzu 14-A GC/PID/FID	1991
Kratos Isocratic HPLC/UV-VIS with HP 1050 Autosampler	1994
Hewlett-Packard 1050 Gradient HPLC with HP 1050 Autosampler	1993
Dionex Ion Chromatograph DX-100	1991/1993/ 1995
Tekmar LSC 2000 - Purge & Trap [4]	1991/1993
Zymark TurboVap Concentrators	1993
Microsensor Technology Inc. M400 Micro GC	1994
Microsensor Technology Inc. P200H Micro GC	various
Custom Cryotrap Desorption Units for Canisters and Tedlar Bags [5]	1994
Custom Sorbent Tube Desorption Unit	1992
Custom Calibration Gas Blending Manifold	various
Custom Convectron Based Pressure/Vacuum Multi-Canister Cleaning Manifold [3]	

Sampling Media and Equipment Available from ATL In Support of Analytical Services

Collection Media/Equipment	Applicable Methods	Description
Summa Canisters (1 & 6 Liter)	TO-3, TO-12, TO-14, TO-15, ASTM 1945, 1946, 3416	Stainless steel vessel with passivated, chemically inert interior.
Silcosteel™ Canisters	ASTM 5504	Stainless steel vessel with glass-lined interior, no metal contact
Flow Controllers		Pre-configured for sampling periods from 20 minutes to 24 hours.
Vacuum gauges		For use with Summa canisters to record canister vacuum
Tedlar Bags (1, 3, 6, 10 Liter)	CARB 410A/B, 422; EPA TO-3, TO-14, TO-15; ASTM 1945, 1946, 3416, 5504	PVF (Tedlar) film bag - no metal fittings.
Tenax Tubes	TO-1	Glass cartridge packed with Tenax, stainless steel end caps, high temp. Teflon ferrules.
'VOST' Tubes	EPA 0031/5041	Analytical cartridge packed with Tenax, backup cartridge with Tenax/charcoal; stainless steel end caps, high temp. Teflon ferrules.
Carbon Molecular Sieve	TO-2	Glass cartridge packed with CMS, stainless steel end caps and ferrules.
DNPH Impinger	CARB 430, TO-5, BIF 0011	Aldehyde and ketone derivatization reagent.
DNPH Sep-Pak Cartridge	TO-11, BIF 0100	Sep-Pak cartridges coated with DNPH; each tube packaged in a glass transport tube.
PUF Cartridge	TO-4, TO-10	Clean and batch certified PUF/glass sampling cartridge.
PUF/XAD Trap	TO-13, 8270	High volume PUF/XAD "sandwich" glass sampling cartridge.

ATL can also provide a limited number of sampling pumps, lung samplers, canes and sampling trains, as well as custom fittings and sampling T's.

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Quality Assurance

ATL maintains a comprehensive Quality Assurance and Quality Control program to assess data quality, ensure analytical data is within method specified control limits, and comply with project specific QAPP requirements. Wherever possible, the quality control measures used are those specified in "Test Methods for Evaluating Solid Waste, SW-846," Third Edition, Revised September 1994. Elements of the following QA/QC protocols are also used where applicable:

- EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air (TO-1 through -14), EPA 600/4-87-006;
- EPA Compendium of Methods for the Determination of Air Pollutants in Indoor Air (IP-1A, IP-1B);
- Organic Superfund Contract Lab Program Statement of Work 10/86;
- CLP Draft Analytical Method for Determination of VOCs in Air Collected in Summa Canisters and Analyzed by GC/MS, 12/91;
- Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act 40 CFR (136) 1984;
- NIOSH Manual of Analytical Methods;
- Annual Book of ASTM Standards;
- OSHA Analytical Methods Manual;
- California Air Resources Board Stationary Source Test Manual;
- Bay Area Air Quality Management District Manual of Procedures;
- DOE HAZWRAP Quality Control Requirements; and
- NFESC Sampling and Chemical Analysis Quality Assurance Requirements.

ATL's Laboratory Quality Assurance Program Manual provides documentation of all QA/QC procedures in place, including:

- personnel responsibilities and training;
- standardization;
- validation;
- safety;
- accuracy and precision guidelines;
- minimum detection limits (MDLs);
- sample handling;
- chain of custody procedures;
- sample tracking;
- analytical procedures;
- waste storage and disposal;
- data reduction and reporting;
- quality control and corrective action procedures;
- preventive maintenance; and
- performance and system audits.

A copy of the Laboratory QAP is available upon request.

Audits and Certifications

ATL participates in EPA's WP/WS performance evaluation program. We routinely undergo on-site audits and analyze proficiency samples submitted by certifying agencies as well as independent clients. ATL currently holds the following certifications, validations and approvals:

- US Army Corps of Engineers - Missouri River Division
- California State Department of Health Services
- New York State Department of Health
- Utah State Department of Health
- Arizona State Department of Health

ATL is under contract as a quality assurance laboratory for "air" projects for the Army Corps of Engineers, New England District.

While there is no formal US Navy certification for specialty (such as air) laboratories, we have successfully completed NFESC project-specific audits conducted by Contech.

In addition, ATL is a participant in the NIOSH PAT proficiency program.

ATL was an active participant in the EPA Special Analytical Services (SAS) program. This enabled us to contract with the EPA on projects that were generally extensions of existing methodology. In 1992, ATL was awarded and successfully completed 7 SAS contracts. We received 6 contracts in 1993 and 3 in 1994 prior to the discontinuation of the program. Our goal has been to meet the stringent requirements set by EPA for these programs, in the belief that it will benefit all of our clients.

ATL's Quality Assurance (QA) Department administers a routine in-house and second source quality control (QC) check sample program and conducts annual internal laboratory audits. ATL's QA chemist has been trained as a certified lead Quality System Auditor utilizing the ISO9000 standard under the RAB Accreditation Program. The audits consist of extensive check-points encompassing both the technical and quality aspects of the laboratory operations. Any deficiencies noted are managed through a formal corrective action plan process.

Standard Operating Procedures (SOPs)

SOPs are prepared for all major laboratory functions and methodologies. Current SOPs are in place for laboratory operations as listed below:

TITLE	REV	DATE
Sample Receiving and Data Review - Evidentiary	6	4/7/94
Analysis of Volatile Organic Compounds in VOST Cartridges and Condensates by EPA Method 5041	11	1/8/98
Analysis of Petroleum Hydrocarbons by State of CA (LUFT) Leaking Underground Fuel Tank Field Manual 1988 Edition	1	11/20/89
Preparation of Tenax and Tenax/Charcoal Tubes for VOST or Ambient Air Samples	3	2/4/94
Analysis of Volatile Organic Compounds in Ambient Air using EPA Method TO-1 and TO-2	2	1/6/92
Analysis of Volatile Organic Compounds in Summa Polished Canisters by EPA Method TO-14	10	1/27/98
Preparation of Summa Canisters for Sampling	8	1/9/98
Analysis of Oxygen, Nitrogen, Methane, Carbon Monoxide, Carbon Dioxide and NMOC by ASTM Method D-3416	3	5/14/96
Analysis of Phenol and Cresols by EPA Method TO-8	1	6/10/94
Analysis of Semi-Volatile Compounds Collected on PUF/XAD Cartridges using EPA Method TO-13/8270	2	8/13/97
Analysis of Aldehydes and Ketones by EPA Method TO-11	0	8/1/92
Analysis of Aldehydes and Ketones by EPA Method TO-5	0	8/6/92
Analysis of Sulfur Compounds by EPA Method 15/16	1	3/25/94
Field Documentation Requirements	0	8/13/93
Field Sample Management	0	8/13/93
Sorbent Tube Tracking Procedures	2	1/18/97
Safe Lifting Procedures	1	1/18/94
Handling of Spills in the Sample Receiving Area	1	1/17/97
Refrigerator and Freezer Content and Temperature Monitoring Documentation	1	1/16/97
Analysis of Volatile Compounds in Air by EPA Method 18	0	11/19/93
Standard Preparation Documentation	2	1/20/97
Data Review	2	1/16/97
Diskette Deliverable Generation and Review	1	1/18/97
Storage and Disposal of Hazardous Wastes	0	1/14/94
Analysis of Aldehydes by EPA Method 0011 (BIF0011)	1	2/7/94
Analysis of Pesticides/PCBs Collected on PUF/XAD Cartridges using EPA Method 8080, TO-10 and TO-4	1	1/19/95

TITLE	REV	DATE
Internal Audit Program	3	4/28/97
Analysis of Volatile Organic Compounds in Summa Canisters by EPA Method TO-14 via Ion Trap GC/MS	0	6/3/94
Analysis of Low Level Vinyl Chloride in Summa Canisters by EPA Method TO-14 via Ion Trap GC/MS	0	6/3/94
Laboratory Security	1	1/16/97
Laboratory Safety	0	6/6/94
Generation, Updating and Distribution of Controlled Documents	1	1/17/97
Inventory of Neat Materials and Certified Compound Mixes	0	8/25/94
Certification of Test Equipment	1	1/3/96
Analysis of Formaldehyde & Acetaldehyde by CARB Method 430	0	9/30/94
Analysis of Non-Methane Organic Compounds (NMOC) by EPA Method TO-12	1	11/7/94
Analysis of Sulfur Compounds by Modified EPA Method 15/16 via GC/SCD	4	2/28/96
Analysis of Low Level Vinyl Chloride in Summa Polished Canisters via GC/MS-SIM EPA Method TO-14	0	6/26/95
Direct Injection of GC/MS Analysis of Volatile Organic Compouds by EPA Method 18	0	7/20/95
Analysis of Low Level Vinyl Chloride in Air via Prefractionator Interface EPA Method 18	0	7/21/95
Analysis of Benzene, Toluene, Ethylbenzene, Xylenes and Total Petroleum Hydrocarbons - EPA Method TO-3	2	1/7/98
Analysis of pptv Levels of Volatile Organic Compounds in Summa Polished Canisters Using Capillary Direct Quadrupole GC/MS - EPA TO-14	1	1/9/97
Preparation and Review of Laboratory Narratives	1	11/26/96
Writing Standard Operating Procedures	0	4/3/97
Analysis of Volatile Organic Compounds in Condensates by EPA Method 5030/8260A and Analysis of VOST Cartridges by EPA Method 5040A/8260A	0	6/24/96
Preparation and Review of Control Charts	0	11/22/96
Analysis of Oxygen, Nitrogen, Methane, Carbon Dioxide and NMO by EPA Method 25C	1	10/16/97
Receivivng and Tracking of Samples	0	1/7/97
Use of the Canister Asset Tracking System (CATS)	0	11/20/97
Manual Peak Integration – GC?MS	0	2/10/97
Canister Pressurization	0	12/9/97

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Experience

ATL works with a wide variety of clients in both the government and private sectors. Public sector clients include the US EPA, as well as State and local agencies. We have extensive Department of Defense (DOD) experience, conducting numerous projects for prime contractors, as well as contracting directly with the Army Corps of Engineers, Air Force AFCEE and Navy NFESC. We also work with Department of Energy sites, including Savannah River, Lawrence Livermore and Sandia National Laboratories. A partial DOD/DOE experience listing appears on the following pages.

ATL has established national relationships with numerous environmental engineering and consulting firms, as well as many industrial clients. Our laboratory has provided analytical support for many high profile projects nationwide, including dozens of hazardous waste incinerator trial burns.

We have made a concerted attempt to find unique, challenging projects to complement and enhance the work performed using the mainstream analytical protocols. This diversity allows ATL to stay on the leading edge of technology and offer a range of services unmatched by other air laboratories.

Air Toxics has unique mobile capability. Our mobile laboratory is equipped with state-of-the-art GC and GC/MS instrumentation, which is specially configured to analyze air samples. It is staffed by our most experienced analysts, who are able to both analyze samples and review data. The mobile laboratory has sufficient communications capability to send and receive data from our laboratory in Folsom, which means that raw data can be reviewed by QA personnel immediately, if necessary. Special report software has been written on a project specific basis, so that reports, in the client's format, are available at the end of the day.

In order to ensure that we are providing our clients with the best available service, we continually work to stay abreast of new technology. This means constantly evaluating and adopting new sampling and analytical techniques, or developing our own in-house equipment. Our Methods Development group is chartered with extending the range of existing methodologies through either chemical validation or hardware modifications. They have developed innovations such as a sorbent-based means of analyzing mid-boiling point range compounds that is more cost effective and easier to sample than conventional semi-volatile air sampling methods.

ATL is active in various public forums for disseminating technical information. Our personnel have published or presented over 30 papers at national and regional technical meetings, including 10 presentations at the annual AWMA Symposium on Measurement of Toxic and Related Air Pollutants.

The following selected project profile summaries present a brief overview of ATL's capabilities and experience in several different types of analytical programs.

MCCLELLAN AIR FORCE BASE, SOIL GAS SURVEY

Both Jacobs Engineering Group and Radian Corporation collected soil gas samples in support of the Air Force sub-surface contamination mapping project at the base. Wells were bored and soil gas samples collected at various depths. ATL received up to 25 soil gas samples per day for a two-year period for EPA Method 18 analysis. Since vinyl chloride at the ppbv level is difficult to detect in heavily contaminated soils gas utilizing Method 18, ATL developed a unique inlet system which allows only vinyl chloride to enter the GC. This eliminates the possibility of a false positive while significantly improving the measurement of vinyl chloride. Based on the Method 18 data, a number of full scan TO-14 analyses were performed.

Hard copy data reports were required within 24 hours, with a specially formatted diskette provided within 21 days. In addition to the analytical work, ATL supplied all the Tedlar bags and up to 20 certified Summa canisters per day for the project and participated in the base's monthly supplier meeting.

CLIENT: Radian Corporation, Sacramento, CA
Jacobs Engineering, Sacramento, CA

AIR FORCE BASE BIOVENTING SURVEY PROJECT

Parsons Engineering Science has been contracted to survey each US Air Force base that has an on-going bioventing project. A number of Summa canister samples are taken at each base. Each sample is analyzed for BTEX (DL = 1 ppbv) and TPH (Jet Fuel).

Air Toxics provides 1 Liter canisters on request. At any one time, there are approximately 50 canisters in the field. Analysis and reports are due within 72 hours of sample receipt. To date over 700 samples have been submitted from almost 40 bases, and the total number of samples will exceed 1,000. The project is expected to continue for another several years.

CLIENT: Parsons Engineering Science, Denver, CO

NAVAL UNDERSEA WARFARE STATION KEYPORT, WA and NAVAL WHIDBEY ISLAND, KEYPORT, WA AMBIENT AIR SAMPLING

Each of these two TO-14 projects required supplying 96 certified Summa canisters and 25 flow controllers. All of the requested sampling equipment was shipped within 48 hours of notification. Each ambient air sample was collected over 24 hours.

Samples were analyzed for a full target compound list + top 10 TICs. Full CLP documentation was required within 21 days.

CLIENT: SAIC, San Diego, CA

USPCI/LAIDLAW TRIAL BURN

USPCI/Laidlaw have been in the process of bringing on-line a new hazardous waste incinerator in Clive, Utah. Several miniburns were completed in preparation for the full burn in late Summer 1995. This project involved analyzing an extensive list of volatile organic compounds by the VOST protocol. Over 250 samples were analyzed over the course of the project.

ATL was successful in becoming certified for VOST in the State of Utah and passed rigorous audits by both the State of Utah and USPCI/Laidlaw.

CLIENT: Radian Corporation, Austin, TX

REGION IX; SUPERFUND SITE PERIMETER MONITORING PROJECT

This TO-14 project consisted of eight rounds of samples from the site perimeter taken over a six month period. ATL provided 150 Summa canisters and 40 flow controllers for each sampling round. The project required a 0.05 ppbv detection limit for vinyl chloride. Samples were also analyzed for methane by ASTM D-3416.

Each round of sampling required hard copy reports in 48 hours, with CLP data packages provided within 14 days. Canisters were cleaned, certified and returned to the field within 48 hours.

As a prerequisite for receiving this contract, ATL was required to successfully analyze three different low level (1.8 ppbv vinyl chloride) performance evaluation samples and pass an extensive on-site audit conducted by VIAR Corporation.

CLIENT: Viar Corporation, Alexandria, VA

TOOELE ARMY DEPOT SOUTH AREA RI/FS PROJECT

The project consisted of ambient air samples from the Tooele Army Depot Phase II Site Investigation. Over a two month period, 48 samples were collected during four rounds of sampling. ATL analyzed for VOCs, SVOCs and 7 PCB Aroclors utilizing EPA Methods TO-14, TO-13 and TO-4, respectively.

Air Toxics supplied prepared GPS-1 glassware with PUF/XAD cleaned down to ambient levels. ATL currently owns 150 pieces of glassware for the Graseby Model GPS-1 sampler.

CLIENT: EBASCO, Lakewood, CO

HUNTERS POINT ANNEX AIR MONITORING PROJECT

Hunters Point has operated as a shipyard and ship repair facility almost continuously since 1869 and a wide variety of solid and liquid wastes were generated and disposed at the site. This project consisted of 85 flux chamber and ambient air samples collected at 9 preselected sites at the facility. Summa canisters and PUF/XAD cartridges were provided by ATL. Analytical requirements included TO-14 and TO-13, with additional compounds requested by both methods. Several pesticides, 7 PCB Aroclors and the full 8270 analyte list were successfully quantitated from the same PUF/XAD sample train.

CLIENT: PRC Environmental Management, San Francisco, CA

BROOKFIELD AVENUE LANDFILL RI/FS PROJECT

New York City's Brookfield Avenue Landfill is classified as an Inactive Hazardous Waste Disposal Site, Class 2. Cyanide and several VOCs were illegally disposed at the site. The project involved ambient, flux and passive vent samples and consisted of 4 field events with 20 samples per round. ATL analyzed VOCs using TO-14, cyanide using NIOSH 6010, sulfur gases using EPA Method 15/16, and methane using ASTM D-3416.

ATL successfully analyzed several additional polar compounds using TO-14. The analyte list included methanol, acetonitrile, ethyl ether, and acrylonitrile, as well as several ketones.

CLIENT: Camp, Dresser & McKee, Woodbury, NY

PSE&G BAYONNE, NJ GAS WORKS, SOIL GAS AND AMBIENT AIR

Ambient air and flux chamber samples were analyzed for volatile organic compounds using EPA Method TO-14 and ASTM D-3416 (methane only). A detection limit of 0.1 ppbv was required for all target compounds except methane.

The project consisted of two sampling rounds. The first conducted in 1992 and the second in October of 1993. Each sampling event involved 65 samples collected over four days. Air Toxics provided the certified Summa canisters, the flow controllers and ancillary equipment such as vacuum gauges and electronic flow sensors.

Level III data was delivered approximately 10 days after receipt of the final round of samples.

CLIENT: Woodward-Clyde Consultants, Wayne, NJ

NORTON AIR FORCE BASE SOIL GAS PROJECT

Soil gas samples were analyzed for volatile organic compounds (EPA Method TO-14), methane (ASTM D-3416), total non-methane compounds (TO-12) and moisture using silica gel tubes.

The project consisted off approximately 11 samples for each type of analysis. Air Toxics provided the individually certified Summa canisters and ancillary equipment such as sampling lines with fittings, vacuum gauges and particulate filters.

Preliminary data was available five days after receipt, Level IV (CLP) data was delivered on schedule.

CLIENT: CDM Federal Programs, Fairfax, VA

TINKER AIR FORCE BASE, OKLAHOMA, ENVIRONMENTAL TREATABILITY STUDY

Parsons Engineering Science conducted an underground tank and piping removal project at Tinker Air Force Base in Oklahoma. The surrounding soil was contaminated with JP-4 jet fuel. ATL analyzed soil gas samples from a total of 15 multi-depth monitoring points and soil flux gases via a flux chamber in 9 different surface sampling positions.

ATL provided 6 liter canisters and 24-hour flow controllers. Over 180 samples were analyzed for volatile organic compounds by TO-14. Data was reported within 10 days with IRPIMS diskette due in 15 days.

CLIENT: Parsons Engineering Science, Fairfax, VA
LOCKHEED ENGINEERING & SCIENCE COMPANY

ATL's Research and Technical Services staff conducted a study of emissions from environmental chamber testing of various bag materials for Lockheed Engineering & Science Company. The sampling technique used was a variation of the ASTM D5116-90 Method. The purpose of this study was to demonstrate the bag materials' effectiveness for holding aldehyde-based fixative solutions, and to determine the relative permeability of these materials to off-gassing. ATL's laboratory conducted the chemical analysis of the air samples collected.

CLIENT: Lockheed Engineering & Sciences, Moffett Field, CA

NORWOOD, MA, PCB SITE, ONGOING MONITORING PROGRAM

Samples are collected for an ongoing monitoring program at this PCB contaminated site. Approximately 5-10 samples are analyzed each week for EPA Methods TO-14 and TO-4 (PCBs only). Results are reported within five days. Cans are 100% certified by EPA Method TO-12 and 10% certified by EPA Method TO-14.

CLIENT: Foster Wheeler Environmental, Boston, MA

MCCLELLAN AIR FORCE BASE, ON-SITE SOIL GAS ANALYSIS

This project required the extent of contamination to be determined in the field in order to make immediate drilling decisions. ATL's Research and Technical Services staff used a field portable Microsensor Technology Institute (MTI) microchip GC located in a van next to the drill rig to analyze up to 15 samples each day during this 3 month project. The target analyte list included 16 chlorinated and aromatic volatile organic compounds. Results were verbally communicated to the field geologist in less than 15 minutes from the time of sample collection.

CLIENT: Radian Corporation, Sacramento, CA

SACRAMENTO ARMY DEPOT: SEAMIST MONITORING PROGRAM

The SEAMIST project is part of an ongoing monitoring program taking place at the Sacramento Army Depot. Six soil gas wells, each with 10 sampling ports, were installed at the site. Sixty samples per month are analyzed by EPA Method TO-14, with results reported within two weeks. Over 600 samples have been analyzed to date.

CLIENT: Kleinfelder, Sacramento, CA

CLP SAS Program

ATL has always participated in the EPA's Special Analytical Services (SAS) program. This work is normally technically challenging and the QA is rigorous. The successful completion of a SAS contract clearly demonstrates the ability of the laboratory to perform at the highest level.

Each contract awarded is accompanied by a Performance Evaluation (PE) sample. The laboratory must report the correct value(s) often within 24 hours. ATL passed every audit sample submitted. In addition, there was often an on-site audit conducted by Viar Corporation. These audits are comprehensive and served to fine tune aspects of our procedures.

EPA SAS Contract Awards

1994
Contract number 8306B, Region II: TO-14
Contract number 8342E, Region V: TO-14
Contract number 8356F, Region II: TO-14

1993
Contract number 7727A, Region I
Contract number 7753A, Region I: TO-14
Contract number 7797A, Region I: TO-14
Contract number 78560A, Region I
Contract number 8073A, Region I: TO-14
Contract number 8245A, Region I: TO-14

1992
Contract number 7043-D, Region IV: TO-14
Contract number 7165-A, Region I: TO-14
Contract number 7276A, Region I
Contract number 7292A, Region I: TO-14
Contract number 7459A, Region I: TO-14
Contract number 7104A, Region I
Contract number 7589Y, Region IX: TO-14

1991
Contract number 6074-HQ, Method Validation: TO-14/TO-1
Contract number 6668-A, Region I

Selected Publications and Presentations

"pptv Determination of Reduced Sulfur Compounds: A Study of the critical Parameters," R.R. Freeman, Eric D. Winegar, D.C. Curtis and C.C. Crume, AWMA Symposium on Measurement of Toxic and Related Air Pollutants, Durham, NC, May 1995.

"Comparative Data Quality: Portable GC vs. Laboratory GC/MS," Marie Yates, Eric D. Winegar, D.C. Curtis and R.R. Freeman, AWMA Symposium on Measurement of Toxic and Related Air Pollutants, Durham, NC, May 1995.

"Air Measurement: The Forces Behind the Technology," Eric D. Winegar, Environmental Lab, pg 15, March/April 1995.

"Water Management in Sampling and Analysis: Problems and Progress," Eric D. Winegar, David B. Curtis, Source Evaluation Society, San Diego, CA, March 1995.

"Current Approaches to Point and Area Source Emission Measurements: Sampling and Analysis," Eric D. Winegar, Pittsburgh Conference on Analytical Chemistry and Spectroscopy, New Orleans, LA, March 1995.

"Field Screening vs. Field Analysis: Data Quality Objectives in the Age of Advanced On-Site Instrumentation," Eric D. Winegar, Marie Yates, David B. Curtis, EPA/AWMA Field Screening Conference, February 1995.

"Methyl Bromide Users Search for Science," Eric D. Winegar, Environmental Protection, January 1995.

"An Integrated Approach to Parts-Per-Trillion Measurements of Volatile Organic Compounds in Air," Eric D. Winegar, R.R. Freeman, Craig C. Crume, AWMA Symposium on Measurement of Toxic and Related Air Pollutants, Durham, NC, May 1994.

"Beyond Canister Cleaning...What About the Surface Chemistry?" R.R. Freeman, Eric D. Winegar, Craig C. Crume, AWMA Symposium on Measurement of Toxic and Related Air Pollutants, Durham, NC, May 1994.

"Methyl Bromide: Simple Chemical, Complex Issues," Eric D. Winegar, Environmental Protection, June 1994.

"Area and Point Source Sampling Approaches for the 1990's," Eric D. Winegar, American Chemical Society, San Diego, CA, March 1994.

"ppbv Determination of Vinyl Chloride in Soil Gas Using Modified EPA Method TO-14," R.R. Freeman and D.C. Curtis, AWMA Current Issues in Air Toxics, Sacramento, CA, November 1993.

"The Analysis of Acrolein Using CARB Method 430: What Works and What Doesn't Work," R.R. Freeman and D.C. Curtis, AWMA Current Issues in Air Toxics, Sacramento, CA, November 1993.

"Field Instruments for Air Sampling," Eric D. Winegar, Environmental Protection, July 1993.

"Practical Aspects of Using Summa Canisters for Ambient Air Sampling," R.R. Freeman, Eric D. Winegar, Craig C. Crume, AWMA Symposium on Measurement of Toxic and Related Air Pollutants, Durham, NC, May 1993.

"The Use of Perdeuterated Sampling Surrogates (d_6-butadiene and d_6-benzene) in Comparing Tedlar Bags and Canisters for Source Samples," Eric D. Winegar and R.R. Freeman, Source Evaluation Society Meeting, San Diego, March 1993.

Sampling and Analysis of Airborne Pollutants, Eric D. Winegar and Lawrence H. Keith, eds., Lewis Publishers, 1993.

"Ion Trap GC/MS - Applications to TO-14 Ambient Air Monitoring," L.L. Freeman and I.C. Crume, Current Issue in Air Toxics, AWMA, Sacramento, CA, November 1992.

"Assessment of Indoor Air Exposure to Medical Waste Incineration Emissions by Extractive Fourier Transform Infrared Spectroscopy and Conventional Sampling," Eric D. Winegar, Jeff B. Hicks and William B. Herget, IAQ '92, San Francisco, CA, October 1992 and Symposium on the Measurement of Toxic and Related Air Pollutants, Raleigh, NC, May 1992.

"Air Toxics Sampling and Analysis: A User's Perspective," Eric D. Winegar, ASTM E-6 Meeting, San Diego, CA, October 1992.

"Ion Trap GC/MS Applications to Air Monitoring," L.L. Freeman and R.R. Freeman, AWMA Symposium on the Measurement of Toxic and Related Air Pollutants, Durham, NC, May 1992.

"What is a Good 'Hold Time' for Canisters?Stability Studies and Degradation/Formation Questions," AWMA Symposium on the Measurement of Toxic and Related Air Pollutants, Raleigh, NC, May 1992.

"Simultaneous In-Plume and Stack Sampling for Detached Plume Formation Analysis at a Cement Plant," Larry O. Edwards, Eric D. Winegar, Lee Cover, AWMA Symposium on the Measurement of Toxic and Related Air Pollutants, Raleigh, NC, May 1992.

"Data Analysis and Interpretation for Source Apportionment in Integrated and Directional Ambient Air Sampling," Eric D. Winegar, Lesley Matsumoto, Dave-Paul Dayton, AWMA Symposium on the Measurement of Toxic and Related Air Pollutants, Raleigh, NC, May 1992.

"Approaches to the Measurement and Modeling of Volatile Organic Compound Emissions from Industrial Wastewater Treatment Plants," Eric D. Winegar and Judy A. Nottoli, Proceedings of Hazmacon 1992, pg. 608, April 1992 and California Water Pollution Control Association Annual Conference, April 1992.

"Extractive and Open-Path Fourier Transform Infrared Spectroscopy with Conventional Sampling: Uses for Assessment of Exposure to Air Toxics," Eric D. Winegar, Jeff B. Hicks and Carol S. Sim, Proceedings of Hazmacon 1992, pg. 597, April 1992.

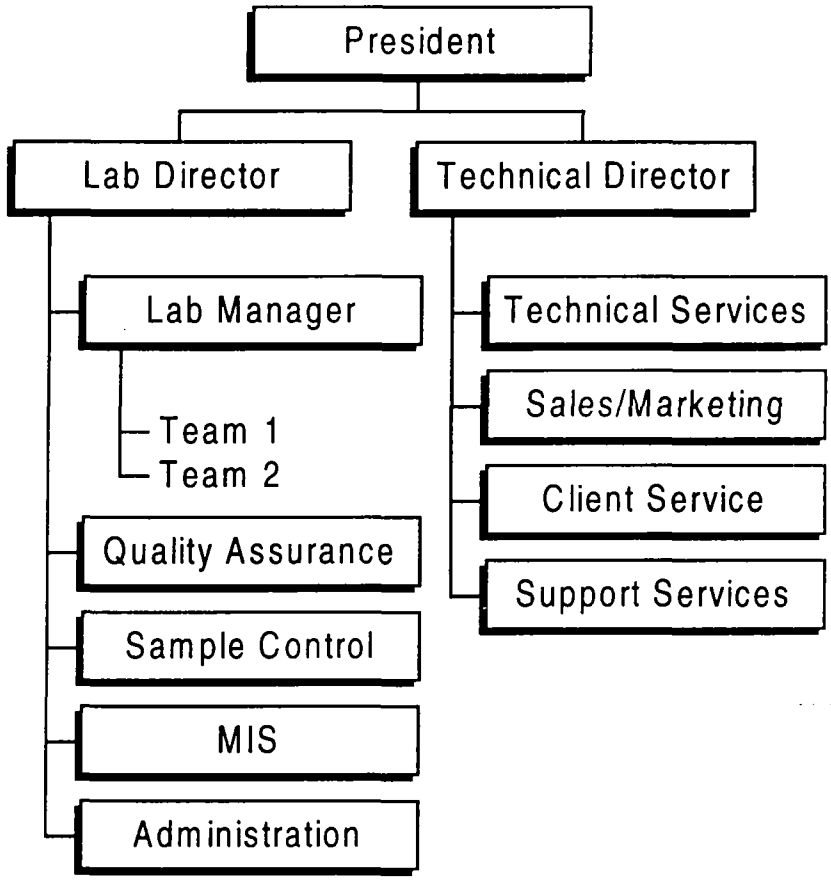
"Chromatographic Considerations for the Analysis of Acid-Extractable Priority Pollutants," R.R. Freeman, Advances in the Identification & Analysis of Organic Pollutants in Water, Volume 1, ed. L.H. Keith.

"Practical Factors Affecting QA/QC of TO-5/CARB 430 Measurements" R.R. Freeman, Proceedings of the 84th Annual AWMA Meeting, p 91-78.9, Vancouver, BC, 1991.

High Resolution Gas Chromatography, R.R. Freeman, Hewlett-Packard Company, 2nd Edition, 1981.

Air Toxics Ltd.

ORGANIZATION



AIR TOXICS LTD.

Key Personnel Profiles

LINDA L. FREEMAN **President and Laboratory Director**

Ms. Freeman founded Air Toxics Ltd. in 1989. She serves as the company's CEO and Laboratory Director. Other activities have included Contract Administrator for key clients and Quality Assurance Manager. Ms. Freeman is a member of the Air and Waste Management Association and the American Chemical Society.

Prior to founding ATL, Ms. Freeman was a staff chemist at Radian Corporation. She served as Chromatography Lab Supervisor and Client Services Coordinator in a production chemistry setting. She played a key role in establishing Standard Operating Procedures for multiple sites. As Client Services Coordinator, she was responsible for program management activities relating to sample flow and data package QC for several five and six figure analytical projects.

ACADEMIC AND PROFESSIONAL DEGREES
University of Wisconsin-Madison (Cum Laude), M.S. Analytical Chemistry-Toxicology, 1980

Boston College (Magna Cum Laude) B.S. Analytical Chemistry, 1978

ROBERT R. FREEMAN **Vice President and Technical Director**

Dr. Freeman co-founded Air Toxics Ltd. and currently serves as Vice President and Technical Director. His focus is identifying new business opportunities and validating new methods and services. He is responsible for the design, fabrication and testing of the various pieces of innovative air sampling equipment used by ATL.

Prior to starting Air Toxics, Dr. Freeman worked with both Varian Instruments and Hewlett Packard, where he was involved in new product development and technical marketing. He later became Director of Marketing and Sales at J&W Scientific. In this capacity, he was responsible for world wide distribution as well as all other marketing activities. Dr. Freeman has published two books and over 60 papers on various aspects of environmental chemistry and separation science.

Dr. Freeman is a member of the Air & Waste Management Association and the American Chemical Society.

ACADEMIC AND PROFESSIONAL DEGREES
National Institute of Health, Postdoctoral Fellowship, Texas Research Institute of Mental Sciences, 1971-1973

University of Houston, Ph.D Physical Chemistry, 1971

University of Illinois, B.S. Zoology, 1966

MARIEJEANNE DEFORET **Quality Assurance Manager**

Ms. deForet has seven years experience as an environmental consultant, QA/QC Group Leader and air sampling and onsite analytical scientist. As QA Manager, Ms. deForet develops strategies and implements programs intended to ensure the laboratory is producing data of known and acceptable quality. She oversees QC activities including various independent checks of laboratory systems, SOP generation, and corrective action procedures, as well as monitoring laboratrory certification programs.

Prior to assuming the QA position, Ms. deForet spent 3 years as a staff scientist in the Research and Technical Services / Field Analytical group. She was responsible for air sampling, onsite analysis using gas chromatography, data interpretation, reporting, and quality assurance program development and implementation for all field projects.

Ms. deForet previously was the group leader for the Radian-Sacramento Chemistry Group, where she was responsible for developing and implementing analytical and QA/QC programs associated with a variety of air, water and soil sampling programs, for reviewing and evaluating analytical results and for interpreting and researching causes of unexpected data points.

ACADEMIC AND PROFESSIONAL DEGREES
Norfolk State University (Summa Cum Laude), B.S. Chemistry, 1989

MIKA GREENFIELD **GC/MS Team Leader**

Ms. Greenfield has over six years experience in performing GC and GC/MS analyses for volatile and semivolatile organic compounds. She has provided analyst training on GC instrumentation and analysis and developed custom techniques for special projects. Ms. Greenfield currently serves as the GC/MS Team Leader and is responsible for the oversight of analysts performing volatile organics analysis by EPA Method TO-14 and VOST projects.

In her previous experience with Ross Analytical and Wadsworth Alert Laboratories, Ms. Greenfield performed analysis of ambient and source level air samples using TO-14, VOST and TO-1 methodologies, in addition to analyzing water, soil and landfill gas samples.

ACADEMIC AND PROFESSIONAL DEGREES
University of Pittsburgh, B.S. Chemistry, 1990
Graduate Level GC/MS Training, Finnegan Institute

HEIDI HAYES **GC and Extractables Team Leader**

Ms. Hayes has six years experience in methods development and trace level analyses of environmental samples using various GC, GC/MS and ICP instrumentation. She is the team leader supervising laboratory analysts performing GC and Extractables analyses, including semi-volatiles by EPA Method TO-13, BTEX analysis by EPA Method TO-3 and atmospheric gas analysis by ASTM Method D-3416.

Prior to joining ATL, Ms. Hayes was the supervisor of contract chemists at the Rocky Mountain Arsenal Laboratory Support Division. In that capacity, she developed preparation techniques and GC methods, certifying the procedures via U.S. Army performance based protocols. Her previous experience also includes positions as a GC and GC/MS chemist with Phoenix Analytical Laboratories.

ACADEMIC AND PROFESSIONAL DEGREES
Colorado School of Mines, M.S. Applied Chemistry, 1993
Luther College, B.S. Chemistry and Math, Summa Cum Laude, 1990

DEBBIE PEARCE **Project Manager**

Ms. Pearce has over 16 years experience in the environmental arena, working with analytical testing laboratories as well as consulting engineer firms. She is Air Toxics' lead project manager/ client service coordinator. Her duties include managing clients' projects from initial scoping, through setup, media shipping, sample tracking, and final data reporting. She aids clients in understanding analytical methodologies, field sampling issues and data reports. As Project Manager, Ms. Pearce serves as liaison between our clients, Quality Assurance and laboratory personnel.

Prior to joining Air Toxics, Ms. Pearce was Client Service Manager for Chem West Laboratories in Sacramento, CA. She previously spent 5 years with Radian Corporation, where she served as QA Coordinator for a number of large-scale projects, as well as several years experience as a GC chemist.

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A.W. Beattie Technical, A.S. Bacteriology, 1975

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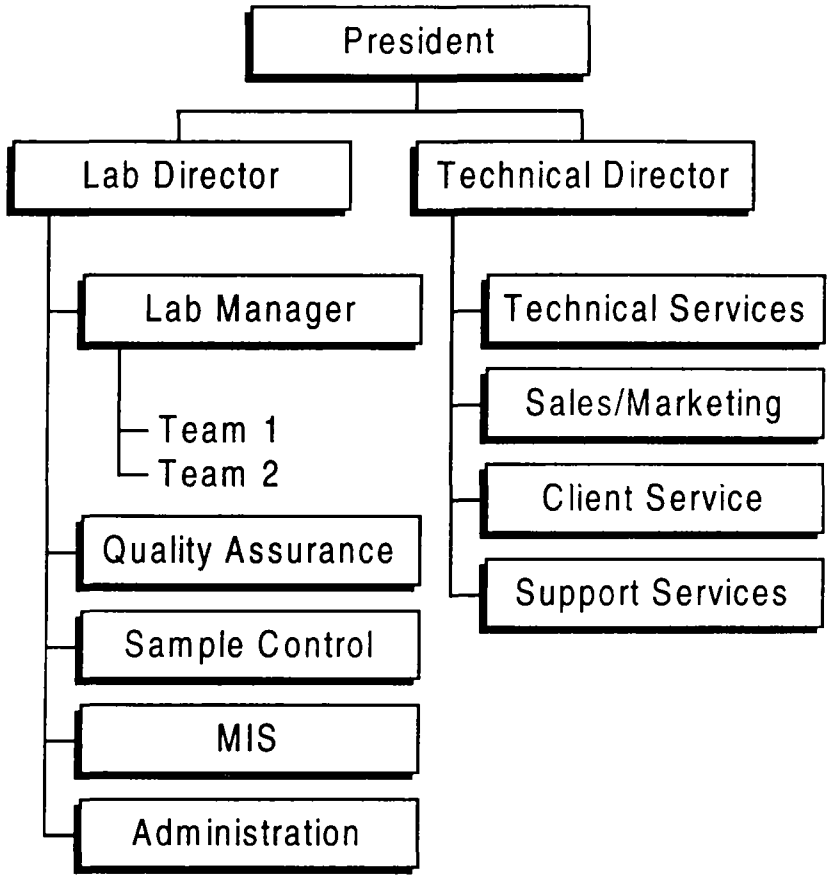

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