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OFFICE COPY

22 May 1992

Ms. Betty Lavis
Remedial Project Manager (HSRW-6J)
U.S. Environmental Protection Agency
77 West Jackson Boulevard
Chicago, Illinois 60604-3590

Subject: Response to the Second Round of Comments on the Draft Interim Quality Assurance Project Plan Moss-American Site, Milwaukee, Wisconsin

Dear Ms. Lavis:

Roy F. Weston, Inc. (WESTON®), on behalf of the settling defendant, Kerr-McGee Chemical Corporation (KMCC), is hereby responding to U.S. EPA's Quality Assurance Section (QAS) comments dated 14 April 1992; State of Wisconsin, Department of Natural Resources (WDNR) comments dated 30 March 1992 from Mr. Gary Edelstein; and WDNR comments dated 20 March 1992 from Mr. Duane Schuettpelz pertaining to the Interim Quality Assurance Project Plan for the Moss-American site. Each comment provided by the aforementioned parties is addressed herein. Also enclosed as attachments are the following:

- Those portions of the QAPP which have been revised as a result of these comments and any previous Agency comments that could not be addressed until completion of the analytical Method Performance Study.
- A revised Standard Operating Procedure (SOP) for Method 8270 SIMS.
- Data and findings of the analytical Method Performance Study performed by WESTON's Lionville Laboratory.

Should you require any additional clarification of this transmittal, please contact either of the undersigned. WESTON and KMCC look forward to the Agency's approval of the Interim Predesign QAPP.

Very truly yours,

ROY F. WESTON, INC.

Gary J. Deigan
Senior Project Manager

Kurt S. Stimpson
Project Director

GJD:KSS/slr
Attachments (3)

cc: Mr. Mark Krippel, Project Manager
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Milwaukee, WI 53212

ATTACHMENT 1

RESPONSE TO U.S. EPA/WDNR COMMENTS AND SUMMARY OF INTERIM QAPP REVISIONS

RESPONSE TO
COMMENTS FROM THE U.S. EPA
QUALITY ASSURANCE SECTION

Comments on the QAPP

I. Signature places for the Laboratory Project Manager and Quality Assurance Manager have been included as requested.

Comments on the SOP

II.A The requested information has been included in the SOP.

II.B This comment has been addressed as requested.

II.C Section 9.5 of the SOP has been reinstated with appropriate revisions.

II.D The base wash described in the SOP in Section 10.3.3 was utilized during the method performance study and will remain in place for sample analysis. The Alumina column cleanup for PAHs described in Method 3611 (SW846) was also utilized during the study and has been added to the SOP.

II.E Section 11.4 only describes the procedure for quantifying target peaks which are outside the calibration range. Only the internal standard amount is increased for dilutions and, as stated, the re-analysis is only used to quantify the large (i.e., out of calibration range) peaks. If many or all peaks are out of range, then a smaller sample size and/or higher spike levels may be used for a re-extraction.

II.F The method does not provide for a calibration for the PAHs other than those requested. In addition, the SIM analysis does not provide enough information to make defensible identification of non-target compounds. The scope of the Predesign Task 2 method development and analysis (per the Consent Decree) is limited to the eight carcinogenic PAHs, and therefore, the method was calibrated to meet this requirement.

II.G The SOP has been modified to reflect that standards used for spiking MS/MSD samples will be from a different source than those used for calibration. Matrix spikes and matrix spike duplicates will be prepared at the frequency specified in the QAPP. Blank spikes may be prepared for internal laboratory QA purposes.

RESPONSE TO
COMMENTS FROM WDNR
(Mr. Gary Edelstein)

1. No response required.

2. No response required.

3. WESTON and KMCC agree that the FSP should outline data use objectives for the background, downstream tributary sediment sampling data and that such data uses should be consistent with the statement of work requirements. WESTON and KMCC had proposed these data uses in a meeting of 26 September 1991 between WESTON, KMCC, U.S. EPA, and WDNR representatives. At this meeting however, it was agreed that data uses for downstream tributary sediment sampling would be deferred until further discussions between U.S. EPA and WDNR had taken place.

4. The proposed background sampling locations were presented to U.S. EPA and WDNR in a letter from WESTON dated 30 March 1992. As of this writing, WESTON and KMCC are awaiting U.S. EPA and WDNR approval of these sample locations.

RESPONSE TO
COMMENTS FROM WDNR
(Mr. Duane Schuettpelz)

Normalizing to Organic Carbon Content

The CD states that:

> *"During construction of the new river, Settling Defendant will remove concentrations of total CPAHs in the soils of the new channel which exceed background or Sediment Quality Criteria (SQC), whichever is greater. The SQC is defined in the ROD as 3 mg/kg total CPAHs."*

As presented above, the cleanup standard for sediments in the new river channel is defined in the CD in a clear and precise manner, that is, 3 mg/kg or background (whichever is greater) without reference to total organic carbon (TOC) content. An SQC of 3 mg/kg is defined in the ROD by assuming an average TOC content for the Little Menomonee River. The suggestion by WDNR that the sediment cleanup standards are TOC-dependent is inconsistent with previous site-related activities in the following respects:

- Neither the ROD nor the CD define the sediment cleanup standards to be TOC-dependent.
- The objectives of Predesign Task 1, as defined in the SOW, include an evaluation of potential rapid-turnaround analytical procedures for CPAHs, to be used in subsequent extent of contamination predesign tasks. There is no requirement for concurrent TOC analyses of sediment samples.
- The objectives of Predesign Task 2, as defined in the SOW, include a determination of background concentrations of CPAHs in soils and sediments. There is no requirement for concurrent TOC analyses of sediment samples.
- The RI/FS sediment background data are reported without reference to TOC.
- Sediment background data recently collected and analyzed by WDNR are also reported without reference to TOC (25 October 1991 memo from T. Janisch to W. Wawrzyn).

WESTON and KMCC believe that WDNR's comment introduces an additional analytical requirement and a potential adjustment to the cleanup standards that is not currently part of the ROD or the CD.

Analysis of Outliers

In general, data values that are much different from the rest can be referred to as outliers. There are many statistical tests that can determine whether outliers are present within a data set. The main concern is how to deal with outliers after they have been identified. Guidance from U.S. EPA (Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities, Interim Final, EPA/530/SW-89/026) indicates:

> *"The test for an outlier provides a statistical basis for determining whether an observation is statistically different from the rest of that data. If it is, then it is a statistical outlier. However, a statistical outlier may not be dropped or altered just because it has been identified as an outlier. The test provides a formal identification of an observation as an outlier, but does not identify the cause of the difference."*

Possible circumstances that might produce outliers include:

- A small area of contamination that is not really a member of the population under study.
- Anomalous sampling or analytical methodology.
- Errors in the transcription of data.
- True, but extreme measurements of the population under study.

WESTON and KMCC expect that the process of selection of background locations with Agency approval will act to minimize the first category of outliers. Background sampling and analysis consistent with the Agency-approved QAPP will act to minimize the second category of outliers. In order to deal with the third category of outliers, WESTON and KMCC propose that any individual data point more than three standard deviations above or below the mean of the others will be rechecked with the analytical laboratory to ensure that no identifiable procedural or transcriptional errors have occurred. If no specific error can be documented, then the outlier is assumed to be a member of the fourth category and retained in the data set. Treatment of outliers in the background studies is expected to be consistent with treatment of outliers that will occur during later stages of the RD/RA (e.g., when attempting to demonstrate that specific areas of the site have achieved the applicable cleanup standard).

ATTACHMENT 2

REVISED PAGES OF THE INTERIM PREDESIGN QAPP

QUALITY ASSURANCE PROJECT PLAN

FOR

THE MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

Prepared by

Roy F. Weston, Inc.
Three Hawthorn Parkway
Vernon Hills, Illinois

21 May 1992

QUALITY ASSURANCE PROJECT PLAN
FOR
THE MOSS-AMERICAN SITE
MILWAUKEE, WISCONSIN

21 MAY 1992

Prepared and
Approved By:

Gary J. Deigan, WESTON
Senior Project Manager

Approved By:

Kurt S. Stimpson, WESTON
Project Director

Approved By:

Mark S. Krippel, Kerr McGee Chemical Corporation
Project Manager

Approved By:

Joseph M. Loeper, WESTON Lionville Laboratory
Project Manager

Approved By:

Diane S. Therry, WESTON Lionville Laboratory
Quality Assurance Section Manager

Approved By:

Betty Lavis, U.S. EPA
Remedial Project Manager

Approved By:

Valerie J. Jones, U.S. EPA
Regional Quality Assurance Manager

4.2 ACCURACY, PRECISION, AND SENSITIVITY OF ANALYSIS

The fundamental QA objective with respect to accuracy, precision, and sensitivity of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols.

The standard operating procedure (SOP) for PAHs is provided in Appendix B. As part of the scope of work for this project, precision, accuracy, and method detection limits (MDLs) were determined via a MDL study. The laboratory followed U.S. EPA guidance for conducting MDL studies and provided a MDL study report. This report is also presented in Appendix B.

Sensitivity

The sensitivity for the CPAH analyses will be the achievable detection limits. Table 8-1 in Section 8 presents the MDLs for each contaminant of concern as determined from the MDL study.

Precision

In general, precision is the level of agreement among repeated independent measurements of the same characteristic, usually under a prescribed set of conditions (e.g., under the same analytical protocols). The most commonly used estimates of precision are the relative percent difference (RPD) for when only two measurements are available, and the percent relative standard deviation (% RSD) for when three or more measurements are available.

~~Precision of laboratory analysis will be assessed by comparing the analytical results between matrix spike and matrix spike duplicate samples. The relative percent difference (RPD) will be calculated for each target analyte pair. The RPD of the MS/MSD will be recorded and evaluated by statistically-generated control limits. For the Moss-American site project, a control limit of ±20 percent will be targeted for precision criteria. For the Moss-American Site project, QA objectives for precision have been omitted until the completion of a method performance study. The method performance study has been designed to evaluate method performance with an off-site Milwaukee-area background soil and sediment matrix. Results of this method performance study will be utilized to specify appropriate ranges for precision.~~ Precision of laboratory analysis will be assessed by comparing the analytical results between matrix spike and matrix spike duplicate samples. The relative percent difference (RPD) will be calculated for each target analyte pair. For the Moss-American site project, a goal of 50 percent will be targeted for precision criteria. Outliers for RPD will be evaluated and flagged on a case by case basis.

Accuracy

Accuracy is the degree of agreement of the analytical measurement with the true or expected concentration. When applied to a set of observed values, accuracy will be a combination of a random component and of a systematic error (or bias) component.

Analytical accuracy is expressed as the percent recovery of an analyte which has been used to fortify an investigative sample or a standard matrix (e.g., blank soil, analyte-free water, etc.) at a known concentration prior to analysis. See Section 13.2.2 for calculation of percent recovery.

The fortified concentration will be at 50 ng/g. Fortified standard matrices prepared in the laboratory are referenced as a blank spike, while fortified field (i.e., investigative) samples are referenced as matrix spikes.

For this project, all eight target analytes will be used as matrix spike compounds. QC limits for recovery will be 50 to 150 percent.

Recovery outliers will be evaluated on a case by case basis. If it is determined that the outliers are a result of lab error, the sample batch will be re-extracted and re-analyzed.

4.3 COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the Weston Lionville Laboratory will provide data meeting QC acceptance criteria for 95 percent or more for all samples tested using the PAH SOP provided in Appendix B. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

Calibration data, to include linearity verification, will be maintained in the laboratory's permanent records of instrument calibrations.

GC/MS - Continuing Calibration

During each operating shift, a single calibration standard may be analyzed to verify that the instrument responses are still within the initial calibration determinations. The response factor for each target compound in the daily standard is calculated and recorded, then compared to the average RF from the initial calibration. For the Moss-American Site Predesign Task 2 analyses, calibration standards will be prepared as discussed in Appendix B of the SOP. The SOP, Section 8.4, contains additional information regarding the frequency of continuing calibration.

If significant (>30 percent deviation) RF drift is observed for any analyte, appropriate corrective actions will be taken to restore confidence in the instrumental measurements. If criteria cannot be met, an acceptable five-point initial calibration must be re-established.

to the investigative samples to demonstrate acceptable method performance, independent of the investigative sample matrix. To facilitate comparison to the actual field samples, final results for the blank spike will be calculated as nanogram per gram (ng/g), assuming 100 percent solids and a weight equivalent to the aliquot used for the corresponding investigative samples. Blank spikes will only be analyzed and reported if the associated matrix spikes yield poor results or if the preparation batch includes no matrix spikes.

~~Blank spikes are performed in duplicate for each preparation batch of 20 or fewer samples. Duplicate analysis allows precision and accuracy data to be generated~~.

9.3.2 Matrix QC Indicators

Matrix QC indicators include duplicates and matrix spikes (MS). Over the last several years, matrix spike duplicates (MSD) have become popular replacements for laboratory duplicates, as they provide measurement data for precision assessment when no target compounds are indigenous to the sample selected for duplicate analysis.

A matrix spike is an aliquot of an investigative sample which is fortified (spiked) with the analytes of interest and analyzed with an associated sample batch to monitor the effects of the investigative sample matrix (matrix effects) on the analytical method.

For this project, MS/MSDs analyses will be performed at a rate of 5 percent (1 per 20 samples of the same matrix). All eight analytes of interest will be spiked into the sample at a mid-range calibration level.

9.3.3 Surrogates and Internal Standards

~~Two surrogates will be spiked into all samples prior to sample preparation to assess extraction and analysis efficiency. For Method 8270 modified, the surrogate compounds to be used are: chrysene-d12 and dibenzo (a, h) anthracene-d14. Surrogate recoveries below 20 percent or above 150 percent will be re-extracted and reanalyzed.~~

~~Three internal standards will be added to the sample prior to analysis but after sample preparation. Internal standards are indicators of instrument performance and are used to quantitate analyte concentrations in the samples. For Method 8270, the internal standards to be used are: pyrene-d10, benzo (a) pyrene-d12 and benzo (g, h, i) perylene-d12~~.

Two surrogates will be spiked into all samples prior to sample preparation to assess extraction and analysis efficiency. The surrogate compounds to be used are: chrysene-d12 and dibenzo(a,h)anthracene-d14. Samples with surrogate recoveries of less than 50 percent or greater than 120 percent will be re-extracted and re-analyzed if it is determined that the outliers are not due to matrix effects.

Three internal standards will be added to the sample prior to analysis but after sample preparation. The internal standards to be used are: pyrene-d10, benzo(a)pyrene-d12, and benzo(g,h,i)perylene-d12.

Solvent/Reagent Water Approval

Pre-purchase approval of solvents, including bottled water purchased for field sampling projects, is performed for all solvents purchased in large quantities. This includes, but is not limited to, acetone, acetonitrile, ethyl ether, freon, hexane, isooctane, methanol, methylene chloride,, toluene, bottled deionized water, and bottled HPLC water. Prior to purchase, a candidate lot of solvent is put in reserve at the vendor's warehouse. A sample case of the lot of solvent is provided by the vendor to the laboratory for testing. If the solvent passes acceptance criteria, the vendor is notified and holds the sample in reserve for laboratory use. The approved lot of solvent is shipped to the laboratory in increments until the entire lot has been received. Prior to exhaustion of the reserve lot, the process will be repeated with a new lot to ensure a constant supply of approved solvent.

The laboratory's on-tap deionized water supply is similarly tested on a monthly basis for selected parameters. Samples are collected and submitted for analysis by laboratory personnel.

Balances, Refrigerators

All sample/standards storage refrigerators and freezers are monitored daily. Refrigerators are monitored twice daily, and include the walk-in coolers in the sample receipt areas as well as those located within the individual laboratories. Balance calibration for all analytical balances is checked daily per WESTON OP21-06-102, "Daily Balance Check."

Instrument Time Check Verifications

An independent check of GC and GC/MS instrument time clocks is performed randomly and at a minimum prescribed frequency by the Laboratory QA Section.

The Section Manager and/or the Laboratory Project Manager review data to ensure consistency with laboratory QC requirements, to verify reasonableness with other generated data, and to determine if program requirements have been satisfied. Selected hard copy output of data (chromatograms, spectra, etc.) will be reviewed to ensure that results are interpreted correctly. Unusual or unexpected results will be reviewed, and a resolution will be made as to whether the analysis should be repeated. In addition, the Laboratory Project Manager or Section Manager will recalculate selected results to verify the calculation procedure. ~~The SOP in Appendix B will contain control limits for surrogates and MS/MSDs to be used in laboratory data review following completion of the method performance study~~. The SOP in Appendix B contains guidance on the evaluation of surrogate and MS/MSD recovery data.

Prior to final review/sign-off by the Laboratory Project Manager, the Data Reporting Section will verify that the report deliverable is complete and in proper format, screen the report for compliance to laboratory and client QA/QC requirements, and ensure that the case narrative covers any noted deficiencies The Laboratory Project Manager will be the final laboratory review prior to reporting the results to the client's Project Manager (Project Manager).

The Laboratory Quality Assurance Section independently conducts a complete review of selected reports to determine if laboratory and client quality assurance/quality control requirements have been met. The Laboratory QA Section will also review 10 percent of the data packages. Discrepancies will be reported to the appropriate Section Manager and/or Laboratory Project Manager for resolution.

Data Reporting

Reports will contain final results (uncorrected for blanks and recoveries), blank and recovery results, methods of analysis, levels of detection, surrogate recovery data, and method blank data. In addition, special analytical problems, and/or any modifications of referenced methods will be noted. The number of significant figures reported will be consistent with the limits of uncertainty inherent in the analytical method. Consequently, more analytical results will be reported to no more than two (2) or three (3) significant figures. Data are normally reported in units commonly used for the analyses performed. Concentrations in solid or semi-solid matrices are expressed in terms of weight per unit weight of sample (e.g., nanograms per gram [ng/g]).

Standard Operating Procedures for Low Concentration Analysis of Carcinogenic Polycyclic Aromatic Hydrocarbons via Method 8270 SIMS

ORGANIC ANALYSIS PROTOCOL
POLYNUCLEAR AROMATIC HYDROCARBONS (PAH) BY CAPILLARY COLUMN GC/MS SELECTED ION MONITORING (SIM) TECHNIQUES FOR MOSS-AMERICAN SITE

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Full Signature Approvals Are Kept on File
with WESTON's Analytics Division
QA Standard Practice Records

DRAFT NUMBER: 05/15/92

1.0 PURPOSE/APPLICATION

1.1 This method is designed for the determination of polynuclear aromatic hydrocarbons (PAH) in soil and sediment. Table 1 lists the analytes determined by this method.

1.2 The practical quantitation limit (PQL) of this method for the determination of an individual compound is 2 ng/g for soil and sediment. PQLs for a specific sample may be different from that listed depending upon the nature of interferences in the sample matrix, percent moisture, and dilutions required for analysis.

2.0 REFERENCE

2.1 EPA SW846, "Test Methods for Evaluating Solid Waste Physical/Chemical Methods", 3rd Edition, Method 8270, Method 3611.

2.2 EPA Method 1625 Revision B, "Semivolatile Organic Compounds by Isotope Dilution GC/MS", January, 1985.

3.0 SUMMARY OF METHOD

3.1 A measured amount of sample (10 g for soil and sediments) is extracted with methylene chloride using a Soxhlet. The methylene chloride extract is concentrated to a volume of 1 mL. Internal standards are then added and a

2 µL aliquot is injected for GC/MS analysis.

3.2 The method provides selected column chromatographic cleanup procedures to aid in the elimination of interferences that may be encountered.

3.3 The method specifies the use of a capillary column gas chromatograph (GC) interfaced to a mass spectrometer (MS) operated in selected ion monitoring (SIM) mode. Data is acquired utilizing SIM descriptors which are switched in sequence according to retention time data derived from a calibration standard.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated backgrounds at the masses (m/z) monitored. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analyses by running laboratory reagent blanks.

4.1.1 Glassware must be scrupulously cleaned to ensure low detection limits. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Refer to Appendix A (SP No. 21-16-0001) for detailed cleaning instructions.

4.1.2 After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

NOTE: Volumetric glassware should not be heated in a kiln.

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

4.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 10.3 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to eliminate false positives and achieve the PQL listed above.

5.0 SAFETY

5.1 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 OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) should also be made available to all personnel involved in the chemical analysis.

5.2 A fully fastened lab coat, latex gloves, and safety glasses should be worn whenever working with samples, extracts, or standards. All chemical containers should be properly labeled according to "Right-To-Know" guidelines.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzo[a]anthracene, benzo[a]-pyrene, and dibenzo[a,h]anthracene. Primary standards of all toxic compounds should be prepared in a hood.

6.0 APPARATUS AND MATERIALS

6.1 Glassware and Supplies

6.1.1 Soxhlet Continuous Extraction Device.

6.1.2 5 mL Disposable serological pipets.

6.1.3 Evaporative Flask, Kuderna-Danish: 500 mL.

6.1.4 Concentrator Tubes, Kuderna-Danish: 10 mL. Attach to K-D flask with plastic clips.

6.1.5 Snyder Column, Kuderna-Danish: three ball.

6.1.6 Vials: 12 and 16 mL with Teflon®-lined screw cap.

6.1.7 Disposable pipets, 5 3/4" pasteur.

6.1.8 Teflon boiling chips: Wash with methylene chloride prior to use.

6.1.9 Nitrogen blowdown apparatus (N-Evap® Analytical Evaporator Model 111, Organomation Associates Inc., Northborough, Massachusetts or equivalent). Teflon tubing connection to trap and gas regulator is required.

6.1.10 Filter paper (Whatman No. 41, or equivalent).

6.1.11 Water bath: heated, with concentric ring cover, capable of maintaining temperature 60-100°C. The bath must be used in a well ventilated hood.

6.1.12 Glass funnels: Glass, wide mouthed.

6.1.13 Heating mantle

6.1.14 Analytical balance capable of accurately weighing ±0.01 g.

6.1.15 Glass wool: baked at 400°C for a minimum of 4 hours before use.

6.1.16 Assorted Class A volumetric flasks including 5, 10, and 100 mL.

6.2 Gas Chromatograph/Mass Spectrometer (GC/MS) System

6.2.1 Gas Chromatograph: An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gasses. The injection port must be designed for splitless injection onto capillary columns. The column should be inserted directly into the source of the MS.

6.2.2 Capillary Column: 30 m long x 0.32 mm ID fused silica DB-5 with 0.25 um film thickness. Refer to Table 2 for complete operating conditions.

6.2.3 Mass Spectrometer: Low resolution mass spectrometer capable of scanning masses up to 500 amu with a cycle time of 1 second or less in the electron impact mode. The MS must be equipped with a 70 eV (nominal) ion source and be capable of acquiring m/z abundance data in real time selected ion monitoring for groups of two or more masses with cycle time of 1 second or less.

6.2.4 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained for the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. The SIM data acquired during the chromatographic program is defined as the Selected Ion Current Profile (SICP). Software must also be available that allows integrating the abundances in any SICP between specified time or scan-number limits, as well as performing routine calculations (i.e.; RF, RRT, amount detected - see Sect. 13).

7.0 REAGENTS

7.1 Sodium Sulfate, granular, anhydrous: purified by heating at 400°C for 4 hours in a shallow tray.

7.2 Alumina: neutral, 80/200 mesh (Woelm-Super A or equivalent). Dry Alumina overnight at 130°C prior to use.

7.3 Sodium Hydroxide Solution: 0.5 N.

7.4 Stock Standard Solutions: Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions. Methylene chloride (dichloromethane; DCM) is used as solvent for all solutions. Refer to Appendix B for standard preparation.

7.5 Acetone, Methylene Chloride, Hexane: pesticide quality or equivalent.

8.0 CALIBRATION

8.1 Using stock standards, prepare calibration standards that will allow measurement of relative response factors (RRFs) for five concentration ratios of each analyte of interest relative to internal standards. Internal standards, surrogates and analytes are listed in Table 1. All solutions should be discarded six months after the date prepared.

8.2 Initial Calibration: Using a 2 μL injection, analyze each calibration standard according to Section 11.1. Tabulate area responses against concentration for each compound and internal standard. Calculate RRFs for each analyte and surrogate.

$$RRF = \frac{[A_s][C_{is}]}{[A_{is}][C_s]}$$

where:

A_s = Area of quantitation ion for compound of interest.
A_{is} = Area of quantitation ion for internal standard.
C_{is} = Concentration of the internal standard, ng/mL.
C_s = Concentration of the compound of interest, ng/mL.

If the RRF value over the working range is a constant ($\leq$25% RSD), the RRF can be assumed to be invariant and the RRFs for the middle concentration will be used for calculations for the remainder of the 12-hour period.

If the RSD is greater than 25.0% or if any RRF is less than 0.25, the calibration may not be used.

8.3 Continuing Calibration: The RRFs must be verified on each working day by the measurement of the middle level calibration standard. If resulting RRFs vary from RRFs of initial calibration by more than ± 30.0% or if any RRF is less than 0.25, the test must be repeated using a freshly prepared calibration standard. If %D criteria still fail, a new initial calibration must be analyzed. If minimum RRF criteria still fail, the instrument or GC column requires service.

8.4 The injection of the first initial calibration standard or the continuing calibration standard initiates a 12-hour analytical period. The instrument is considered calibrated for 12 hours from the time of this first injection, and data for any samples injected during this period will be considered valid.

9.0 QUALITY CONTROL

9.1 Before processing any sample, the analyst must demonstrate through the analysis of a method blank that all glassware and reagents are interferant-free at the method detection limit of the matrix of interest. Each time a set of samples is extracted, or there is a change in reagents, a method blank must be processed as a safeguard against laboratory contamination.

A laboratory "method blank" must be run along with each extraction batch (20 or fewer samples). A method blank is performed by executing all of the specified extraction and cleanup steps, except for the introduction of a sample. The method blank is also dosed with a surrogate solution (see Section 9.3). Sodium sulfate will be used as the method blank for soil and sediment matrices.

9.2 The laboratory will analyze performance evaluation samples as provided by Kerr/McGee. Additional sample analysis will not be permitted if the performance criteria are not achieved. Corrective action must be taken and acceptable performance must be demonstrated before sample analyses may resume.

9.3 Each sample will be dosed with two surrogates (Table 1) just prior to the extraction process. Surrogate recoveries are used to asses method performance; samples with surrogate recoveries of less than 50% or greater than 120% will be re-extracted and re-analyzed if it is determined that the outliers are not due to matrix effects.

9.4 Matrix spikes and matrix spike duplicates will be analyzed at a rate of one per 20 samples of the same matrix. All analytes will be spiked at a level of 50 ng/g. If higher background levels are consistently encountered, the spike level will be adjusted. The QC limits for the MS/MSD recoveries are 50-150%. The Precision goal, expressed as Relative Percent Difference (RPD) is 50%. Recovery and/or RPD outliers will be evaluated and flagged on a case by case basis. If it is determined that the outliers are a result of lab error, the batch will be re-extracted and re-analyzed.

9.5 Individual chromatographic runs will be evaluated on a case by case basis for evidence of carryover. Corrective action (e.g. re-analysis, insertion of blanks, etc.) will be performed as appropriate.

10.0 EXTRACTION AND CLEAN-UP PROCEDURES

10.1 Extraction of Soil and Sediment: Record all extraction information in a bound logbook and label glassware accordingly. Rinse all glassware with acetone and DCM and dispose of washes properly. Decant any obvious liquid layer and stir the sample to ensure homogeneity. Dispose of the liquid in a safe manner. Weigh 10 grams of sample into a tared glass jar and record the weight to the nearest tenth of a gram. Add an equivalent amount of granular anhydrous sodium sulfate, or enough to give the sample a dry consistency.

Fill a 500 mL roundbottom flask approximately 2/3 full with DCM. Add a few boiling chips. Stopper the bottom of a Soxhlet with glass wool and attach the Soxhlet to the roundbottom flask. Place the sample into the Soxhlet and label properly. Add 100 µL of surrogate to each sample and add 100 µL of spike solution to BS, MS, and MSD. Reflux the system for a minimum of 16 hours.

After the system has refluxed and cooled, quantitatively transfer the extract into a K-D through a glass funnel lined with filter paper containing sodium sulfate. Rinse the roundbottom flask with DCM to insure quantitative transfer. Add a few boiling chips and a 3-ball Snyder column to the K-D and concentrate the extract on a bath at 90°to 100°C to an apparent volume of 10 mL.

Dispose of the remaining soil in a fiber waste drum.

Proceed to clean-up procedure (10.3).

10.2 Determination of Percent Solids:

Decant any obvious liquid layer and stir the sample to ensure homogeneity. Dispose of the liquid in a safe manner. Determine the weight of an aluminum weighing dish to the nearest tenth of a gram and record it in a bound notebook. Add approximately 10 grams of sample to the dish and record the pan + sample weight (again, to the nearest tenth of a gram). Subtracting the weight of the pan will give the wet weight of the sample. Place the dish in an oven (in a hood!) at 105°C for a minimum of 12 hours. Re-weigh and, subtracting the weight of the pan as before, determine the dry weight of the sample.

Calculation:

$$\frac{\text{Weight of dry sample (g)}}{\text{Weight of "Wet" sample (g)}} \times 100\% = \%\ \text{Solids}$$

% moisture = 100% - % Solids
(reported on Form 1)

Percent solids should be determined at the time samples are weighed for extraction to ensure an accurate representation of the sample being analyzed.

10.3 Cleanup Procedures:

For maximum PAH recovery, the samples must be extracted with DCM and boiled down without solvent exchange in the K-D apparatus. Solvent exchange is to be performed in a 16 mL vial without much heating. Care will be taken during column chromatography to avoid UV irradiation ie., columns will be covered with foil or dark glass columns will be employed.

10.3.1 Transfer the extract from concentrator tube (in DCM) to a 16 mL vial (A) using a disposable pipet. Rinse the concentrator tube with DCM and add to vial A to ensure quantitative transfer.

10.3.2 Solvent exchange to hexane: Rinse the tip of the blow down apparatus with DCM before use. Blow down the extract to about 3 mL. The Reacti-Therm heater setting must never exceed 3.5 on low! Add about 4 mL of hexane to the 16 mL vial (A) and mix it well. Again blow down to about 3 mL.

10.3.3 Base wash: Bring up the volume of the extract to about 5 mL by adding hexane. Add 2.5 mL of 0.5 N NaOH to the vial (A). Cap and shake the vial vigorously for 1 minute. Wait for the two layers to separate. Transfer the top layer to a clean 12 mL vial (B). A centrifuge may be necessary to assist in separating layers. Add 3 mL of hexane to vial A. Cap and shake for 30 seconds. Again transfer the top layer to vial B. Blow the extract in vial B down to about 2 mL. Dispose of remaining base fraction.

10.3.4 Alumina Column Cleanup

Fill the glass chromatographic column to about 20 cm with hexane. Weigh out 10.0 g of alumina and add the alumina to the column. Gently tap the column to distribute the alumina evenly (minimize chromatographic voids). Alternatively, a slurry of alumina in hexane may be used to pack the column. Allow the alumina to settle and then add 1.0 g of anhydrous sodium sulfate on top of the alumina. Elute the column with 50 mL of hexane. Let the solvent flow through the column until the head of the liquid in the column is just above the sodium sulfate layer. Close the stopcock to stop solvent flow. Transfer 1.0 mL of sample extract onto the column. Rinse out extract vial with 1 mL hexane and add it to the colum immediately. To avoid overloading the column, it is suggested that no more than 300 mg of extractable organics be placed on the column. Just prior to exposure of the sodium sulfate to the air, elute the column with a total of 15 mL of hexane. If the extract is in 1 mL of hexane, and if 1 mL of hexane was used as a rinse, then 13 mL of additional hexane should be used. Collect this fraction in a beaker, and then dispose of the eluent appropriately. Next, elute the column with 100 mL of methylene chloride and collect the effluent in a 250-mL flask. Label this fraction PAH's. Concentrate the extracts by the standard K-D and N_2 blowdown techniques to a volume of 1.0 mL.

NOTE: Some extracts (viscous or very dark) may be difficult to blow down to 1 mL. In these cases, a final volume of 5 or 10 mL (as appropriate) may be used.

Caution: Never blow down the extract to less than 0.5 mL at any stage of cleanup as analytes may be lost!

11.0 GC/MS ANALYSIS

11.1 The mass spectrometer will be calibrated with perfluorophenanthrene (FC-5311) before each 12 hour analytical period to ensure correct mass assignment. Establish proper selected ion monitoring (SIM) windows by analyzing a calibration standard to determine retention times of analytes and standards. Refer to Table 1 for SIM conditions and Table 2 for GC conditions.

11.2 Establish acceptable calibration according to section 8.2 or 8.3.

11.3 Add internal standard mix (IS) to the sample extract prior to injection onto the GC/MS. Add 100 µL of internal standard solution for each 1 mL of sample extract. Refer to Appendix B for IS preparation.

11.4 Extracts will be diluted if peaks outside of the calibration range are encountered to bring the largest peak to within the calibration range. After dilution, additional internal standard mix will be added to the extract at the amount described in 11.3. The extract will be re-analyzed in order to quantify large peaks.

One result for each compound will be reported, with a maximum of two analyses per sample reported. Details of how the dilutions are prepared will be documented in the instrument run log.

12.0 IDENTIFICATION CRITERIA

A peak will be identified as positive if it meets the following criteria:

12.1 The calculated RT relative to the appropriate internal standard must be within ± 0.005 RRT units when compared to the continuing calibration standard (or the middle standard of an initial calibration for samples analyzed in the same 12-hour period as the ICAL).

12.2 Peaks with proper RRT occurring at masses monitored for a given compound must maximize simultaneously (±2 scans) and produce a signal at least 2.5 times background. If the peak at confirmation mass does not meet 2.5 times background, but meets all other criteria, and in the judgement of the GC/MS analyst the peak is positive, the compound can be quantified and reported as positive with an explanation written on the chromatogram and a suitable flag qualifying any tabulated results (i.e., Form 1 and the data summary or spreadsheet).

The ratio between the quantitation and confirmation mass (see Table 1) is used to assist the analyst in determining levels of interference. Confirmation masses are not used for quantitation purposes. If the confirmation to quantitation ratio is not within the range specified in Table 1 but, in the judgement of the GC/MS analyst the peak is positive, the compound can be quantified and reported as positive with a suitable flag qualifying any tabulated results (i.e., Form 1 and the data summary or spreadsheet).

13.0 CALCULATIONS

13.1 Concentrations are calculated according to the equation:

$$\text{Conc.} = \frac{[A_c][Q_{is}]\ [V]}{[A_{is}][RRF][W][P]}$$

where:

A_c = Area of target compound quantitation ion.
A_{is}= Area of internal standard quantitation ion.

P = % Solids ÷ 100
Q_{is}= Amount (ng) of internal standard added.
RRF= Relative Response Factor (Section 8.2)
V = Volume of extract in mL (= dilution factor)
W = Sample amount in grams.

Results are reported in ng/g.

13.2 Surrogate Recoveries are calculated according to the equation:

$$\% \text{ Rec} = \frac{[A_s]\ Q_{is}}{[A_{is}][RRF][Q_s]} \times 100$$

where:

A_s = Area of surrogate compound quantitation ion.
Q_s = Amount (ng) of surrogate added.

13.3 Spike Recoveries are calculated according to the equation:

$$\% \text{ Rec} = \frac{[A_{sp}]\ Q_{is}}{[A_{is}][RRF][Q_{sp}]} \times 100$$

where:

A_{sp} = Area of spike compound quantitation ion.
Q_{sp} = Amount (ng) of spike added.

13.4 The relative percent difference between MS and MSD analyses is calculated according to the following equation:

$$\%\text{RPD} = \frac{S - D}{(S + D)/2} \text{ X } 100$$

Where: S = First Sample value (MS value)
D = Second sample value (MSD value)

14.0 Data Reporting

Quantitation reports from the GC/MS system will be transferred to WESTON's Laboratory Information Management System (LIMS) where calculations will be performed and final reports generated.

Typical semivolatile EPA CLP - type forms will be provided (1 through 7) in addition to a data summary and case narrative. Raw data (i.e., Selected Ion Current

Profiles and Quantitation Reports) for all samples and standards will be included as per typical CLP deliverable requirements.

Appendix A

ORGANIC ANALYSIS PROTOCOL
GLASSWARE CLEANING - ORGANICS
WESTON SP NO 21-16-0001
REVISION NO 01

1.0 PURPOSE

Establish procedures for cleaning analytical glassware to ensure that sample integrity is not violated by contaminated glassware.

2.0 PROCEDURE: EXTRACTABLES AND GENERAL PURPOSE GLASSWARE

2.1 Wash glassware with a phosphate-free detergent (e.g., Alconox). Rinse with tap water five (5) times and deionized water five (5) times.

2.2 Rinse with acetone (once). If the glassware still appears dirty, consult the Section Supervisor.

2.3 Rinse with hexane (once).

2.4 Kiln dry at 450°C for a minimum of four (4) hours.

Appendix B

PREPARATION OF STANDARDS

NOTE: All solutions prepared in Class A volumetric flasks.

1.0 Preparation of Internal Standard Solution

1.1 Purchase the following mixture:

Cambridge Isotope Laboratories (CIL) Catalog No. ES-2044
Deuterium Labeled PAH Surrogate Cocktail
Contains the following at 200 µg/mL in dichloromethane-d_2/Methanol-d_4 (1:1):

Pyrene-d_{10}	(0.98%)
Benzo[a]pyrene-d_{12}	(0.98%)
Benzo[g,h,i]perylene-d_{12}	(0.98%)

1.2 Dilute 1 mL (ES-2044) to 10 mL with methylene chloride to make an internal standard (IS) Stock Solution at 20 µg/mL

$$1\text{ mL} \times \frac{200\ \mu g}{\text{mL}} \times \frac{1}{10\text{ mL}} = \frac{20\ \mu g}{\text{mL}}$$

1.3 Dilute the IS Stock by 20x with methylene chloride to make an Internal Standard Working Solution:

example: $0.5\text{ mL} \times \frac{20\ \mu g}{\text{mL}} \times \frac{1}{10\text{ mL}} = \frac{1\ \mu g}{\text{mL}}$

1.4 Add the IS Working Solution to all sample extracts and standards at a rate of 100 µL per 1 mL of extract/standard. This results in 100 ng of each IS added to 1 mL.

1.5 Other convenient dilutions may be used to reach the final Working Solution concentration of 1 µg/mL.

2.0 Preparation of Surrogate Spiking Solution.

2.1 Purchase the following compound as a pure solid:

Dibenz[a,h]anthracene-d_{14}
(CIL Cat. No. DLM-677, D_{14} = 97%)

and purchase the following solution:

1000 μg/mL Chrysene-d_{12} in methylene chloride

(EPA - NOTE: future purchases will probably be Supelco Cat. No. 4-8416M at 2000 μg/mL and will require different dilutions to make a Working Standard)

2.2 Weigh approximately 10 mg of the dibenz[a,h]anthracene-d_{14} to the nearest .1 mg in a 10 mL Class A volumetric flask and dilute to volume with methylene chloride (final conc. = 1000 μg/mL)

2.3 Dilute 1 mL of each solution above (2.1 and 2.2) to 10 mL with methylene chloride to make a Surrogate Standard (SS) Stock Standard:

$$1\ \text{mL} \times \frac{1000\ \mu\text{g}}{\text{mL}} \times \frac{1}{10\ \text{mL}} = \frac{100\ \mu\text{g}}{\text{mL}}$$

NOTE: If the solution in 2.2 is not exactly 1000 μg/mL, adjust the volume used accordingly.

2.4 Dilute the SS stock by 20x with methylene chloride to make a Surrogate Standard Spiking Solution:

example: $$1.25\ \text{mL} \times \frac{100\ \mu\text{g}}{\text{mL}} \times \frac{1}{125\ \text{mL}} = \frac{5\ \mu\text{g}}{\text{mL}}$$

2.5 Add the SS spike solution to all samples and blanks before extraction at a rate of 100 μL per sample. This results in 500 ng of each SS added to each of the samples (10 g).

2.6 Other convenient dilutions may be used to reach the final SS spike solution concentration of 5 μg/mL.

3.0 Preparation of Matrix/Blank Spiking Solution.

3.1 Purchase the following mixture:

Supelco Cat. No. 4-8905 Polynuclear Aromatic Hydrocarbon Mix (or equivalent - adjust concentrations and dilutions as necessary). Contains all analytes (See Table 1) at 2000 μg/mL in methylene chloride/benzene (1:1).

3.2 Dilute 1 mL of the above to 10 mL with methylene chloride to make an Analyte Stock Solution:

$$1\ \text{mL} \times \frac{2000\ \mu g}{\text{mL}} \times \frac{1}{10\ \text{mL}} = \frac{200\ \mu g}{\text{mL}}$$

3.3 Dilute the analyte stock by 40x with methylene chloride to make a Matrix/Blank Spiking Solution:

example: $$2.5\ \text{mL} \times \frac{200\ \mu g}{\text{mL}} \times \frac{1}{10\ \text{mL}} = \frac{5\ \mu g}{\text{mL}}$$

3.4 Add the Matrix/Blank Spiking Solution to the required samples and/or blanks at a rate of 100 µL per sample. This results in 500 ng of each spike compound (i.e., each analyte) added to the appropriate samples and blanks.

3.5 Other convenient dilutions may be used to reach the final analyte concentration of 5 µg/mL in the Matrix/Blank Spiking Solution.

4.0 Preparation of Calibration Standards

4.1 Calibration Standard will be prepared from the IS & SS stocks (prepared in Sections 1.2 and 2.3) and an Analyte Stock (Section 3.2) different than the stock used for the preparation of the Matrix/Blank spiking solution.

4.2 Any convenient serial dilutions may be used to make the solutions below. If a particularly direct series is adopted, it will be documented. Otherwise, refer to the Standard Prep Log ID Number to determine the exact sequence used for a particular stock.

4.3 A "modular" approach is used to prepare the calibration standards so as to allow a given component (IS, SS, or analyte) to be changed and easily checked verses the other components.

4.4 Prepare five solutions of surrogate compounds and five of analytes at the following concentrations in methylene chloride:

40, 100, 400, 1000, 4000 µg/mL

4.5 Add the corresponding surrogate and analyte solutions together at a 1:1 ratio to make the Calibration Standards at:

20, 50, 200, 500, 2000 µg/mL

(These may, at times, be referred to as CC1...CC5)

The 200 μg/mL solution will be used as a Continuing Calibration Standard.

4.6 Before analysis, 100 μL of IS Working Solution (1 μg/mL) will be added to 1 mL of each Calibration Standard (or other similar ratio such as 10 μL to 100 μL, etc.). This will simulate the addition of 100 μL IS Working Solution to a 1 mL sample extract.

TABLE 1
PAH COMPOUNDS

SIM DESC	QUAN REF	COMPOUND	CAS #	QUAN MASS	CONF. MASS	C/Q RATIO	SPIKE AMOUNT
H4	IS#1	Pyrene-d10 (Int. Std. #1)	1718-52-1	212.14	NA	NA	NA
H4	IS#1	Chrysene-d12 (Surr. Std. #1)	1719-03-5	240.17	NA	NA	500 ng
H4	IS#1	Benzo(a)Anthrancene	56-55-3	228.09	226.09	.12-.50	500 ng
H4	IS#1	Chrysene	218-01-9	228.09	226.09	.13-.52	500 ng
H5	IS#2	Benzo(b)Fluoranthene	205-99-2	252.09	126.05	.04-.18	500 ng
H5	IS#2	Benzo(k)Fluoranthene	207-08-9	252.09	126.05	.04-.18	500 ng
H5	IS#2	Benzo(a)Pyrene-d12 (Int. Std. #2)	63466-71-7	264.17	NA	NA	NA
H5	IS#2	Benzo(a)Pyrene	50-32-8	252.09	126.05	.04-.17	500 ng
H6	IS#3	Indeno(1,2,3-cd)Pyrene	193-39-5	276.09	274.09	.11-.46	500 ng
H6	IS#3	Dibenz(a,h)Anthracene-d14 (Surr. Std. #2)	13250-98-1	292.17	NA	NA	500 ng
H6	IS#3	Dibenz(a,h)Anthracene	53-70-3	278.09	279.09	.12-.50	500 ng
H6	IS#3	Benzo(g,h,i)Perylene-d12 (Int. Std. #1)	93951-66-7	288.32	NA	NA	NA
H6	IS#3	Benzo(g,h,i)Perylene	191-24-2	276.09	274.09	.11-.46	500 ng

NA = Not Applicable

TABLE 2
GC/MS OPERATING CONDITIONS

Mass Spec.: 0.75 sec/scan
SIM acquisition (See Table 1)

Column: 30 m x 0.32 mm ID x 0.25 μm df DB-5
(J&W Scientific)

Carrier Gas: Helium

Column Head Pressure: 13 psi

Injection: Splitless (Splitter opened after 1 min.)

Injection Volume: 2 μL

Injector Temperature: 280°C

Transfer Line Temperature: 250-300°C

Column Oven Temperature: 60°C for 1 min
60°C to 240°C at 10°C/min
240° to 300°C at 15°C/min
Hold at 300°C for the duration of the analysis (approx. 5 min)

Total Analysis Time: Approx. 27 min

ORGANIC ANALYSIS PROTOCOL
POLYNUCLEAR AROMATIC HYDROCARBONS (PAH) BY CAPILLARY COLUMN GC/MS, SELECTED ION MONITORING (SIM) TECHNIQUES FOR MOSS-AMERICAN SITE

These Approval Signatures Are Kept on File
with WESTON®'s Analytics Division
QA Standard Practice Records

DRAFT NUMBER: 05/15/92

Printed Name: Signature/Date:

Initiated by: Dianne S. Therry
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Draft Number 04: 02/25/92
Draft Number 05: 05/15/92

Reasons for Change, Draft 05/15/92:

- client requested modification to the method (project-specific): Sections 2.1, 4.1.1, 7.0, 8.0, 9.0, 10.1, 10.3.4, delete 13.2 and re-number rest of section; update Appendix A; update Appendix B Sections 2.4-2.6, 3.3-3.5, and 4.1

ORGANIC ANALYSIS PROTOCOL POLYNUCLEAR AROMATIC HYDROCARBONS (PAH) BY CAPILLARY COLUMN GC/MS, SELECTED ION MONITORING (SIM) TECHNIQUES FOR MOSS-AMERICAN SITE

> I have read and understood this in-house SOP. I agree not to deviate from this in-house SOP without my supervisor's approval. Any approved deviations will be documented with the raw data and co-signed by my supervisor; with copy to the appropriate client file and the laboratory manager.

DRAFT NUMBER: 05/15/92

Printed Name:	Signature/Date:

ATTACHMENT 3

RESULTS OF THE ANALYTICAL METHOD PERFORMANCE STUDY

SUMMARY OF
METHOD PERFORMANCE STUDY FOR METHOD 8270 GC/MS
WITH SELECT ION MONITORING (SIM)

Moss-American Superfund Site
Milwaukee, Wisconsin

The method performance study was conducted by WESTON's Lionville Laboratory in accordance with the following study objectives and scope. Results of the method performance study are presented herein.

Scope and Objectives

Study Objectives

- Further validate the use of the capillary column GC/MS SIM method for determining background concentrations of CPAHs in soils and sediments.
- Establish a database of achievable precision and accuracy QC limits for the SIM method using site soil. Sediment samples will be evaluated for comparability to the soil data.
- Evaluate the use of Method 8310 (HPLC) in determining "higher" background concentrations.

Sample Collection

- WESTON will obtain approximately 1 kg of background soil at an off-site location approximately 1/2 mile north of Brown Deer Road.
- WESTON will obtain approximately 1 kg of background sediment at an off-site location within the Little Menomonee River approximately 1/2 mile north of Brown Deer Road.
- WESTON will obtain approximately 1 kg of on-site (CNW railroad property) soils with total CPAH concentrations on the order of 20 ppm.

Lab Analysis

- GC/MS SIM analysis will be run in triplicate and Method 8310 (HPLC) analysis will be performed to determine background concentrations of total CPAHs in soils.
- Samples will be spiked at 2x, 5x, 10x the background concentration and analyzed as described above.
- Background sediment will be analyzed by both, the GC/MS method and Method 8310 HPLC.
- The on-site soil (~20 ppm CPAH) will be analyzed by Method 8310 HPLC, and by Method 8270 SIMs with dilutions, as necessary.

Data Evaluation/Reporting

The report will include:

- All analysis data.
- All spikes will be reported as concentrations and percent recovery.
- The average percent recovery and standard deviation for each spike level will be reported.
- Precision will be expressed as a relative standard deviation for each spike level.
- All data will be used to assess method performance and the need for any changes to the method.
- Recommendation based on the data will be made regarding data quality objectives for the GC/MS SIM method.
- Apparent deficiencies in meeting QC criteria, if any, will be discussed.
- Results of a background sediment with an associated MS/MSD will be presented relative to the comparability of data with the soil data.
- A limited evaluation of the HPLC Method (8310) will be performed and data will be reported.

Findings

The results of the method performance study for PAH analysis by GC/MS SIM are tabulated in Table 1. Recoveries obtained for the low level (10 ppb) spike are biased low, with average recoveries for all target compounds ranging from 58 to 81 percent. Average recoveries for the medium-level (50 ppb) ranged from 80 to 129 percent, and the average high level recoveries (100 ppb) were biased high ranging from 98 to 127 percent. Our recommendation is to use a 50 ppb spike of each target compound for the background soils and sediments which exhibited concentration levels of approximately 20 ppb for each of the target compounds. If higher background levels are encountered, higher spike levels will be used.

Considering these variations in recoveries (overall range for 50 ppb is 53 percent to 164 percent) for a fairly well characterized sample material (triplicate analysis) and the inherent non-homogenous characteristics of an environmental soil matrix, MS/MSD recovery ranges of 50 to 150 percent appear to be appropriate for the inclusion in the QAPP. A surrogate recovery range of 50 to 120 percent appears to be warranted by the data.

It is important to note that the 50 and 100 ppb spikes exhibit average MS/MSD recoveries around 100 percent, i.e., the method does not exhibit a low bias. The recovery ranges recommended above are appropriate for a multi-analyte spike into an inhomogeneous matrix containing varying levels of the spike compounds.

To briefly summarize, the results obtained with Method 8310 (HPLC analysis), all target compound results with this analysis were biased higher than the GC/MS SIM results. This is to be expected as the HPLC detector exhibits less selectivity than the Selected Ion Monitoring technique.

Appendix 1 of this attachment presents additional sample collection information. Appendix 2 of this attachment presents the raw data package for the completed method performance study.

TABLE 1

METHOD VALIDATION FOR
PAH ANALYSIS BY GC/MS SIM
KERR McGEE, MOSS AMERICAN SITE

ANALYTE	Z030614	Z030913	Z031202	AVERAGE		%RSD
SS #1	56.731	80.475	73.793	70.3	%	17.4
SS #2	54.212	69.733	61.065	61.7	%	12.6
Benzo[a]anthracene	4.1274	6.3220	7.5791	6.0 ng/g	(wet wt.)	29.1
Chrysene	6.9119	10.9578	12.1408	10.0 ng/g	"	27.4
Benzo[b]fluoranthene	6.4840	8.3632	12.9500	9.3 ng/g	"	35.9
Benzo[k]fluoranthene	6.1552	8.8406	7.9070	7.6 ng/g	"	17.9
Benzo[a]pyrene	5.5417	9.0357	11.4832	8.7 ng/g	"	34.4
Indeno[1,2,3-cd]pyrene	4.7697	7.0228	8.0819	6.6 ng/g	"	25.5
Dibenz[a,h]anthracene	1.1367	1.4788	1.7593	1.5 ng/g	"	21.4
Benzo[g,h,i]perylene	4.6345	6.0071	7.6601	6.1 ng/g	"	24.8

ANALYSIS OF LEVEL ONE SPIKE (10 ng/g)

	Z031009	Z030616	Z030912	Z031203	Z031204	
Benzo[a]anthracene	13.2756	13.1813	12.0821	14.5585	15.0800	ng/g (wet wt.)
Chrysene	17.3774	18.4610	16.4088	18.2938	19.9907	"
Benzo[b]fluoranthene	20.6377	15.7826	12.3043	13.6709	13.3601	"
Benzo[k]fluoranthene	17.7627	16.9850	15.3370	9.5721	10.9040	"
Benzo[a]pyrene	20.1433	16.6550	14.7848	15.5232	15.0626	"
Indeno[1,2,3-cd]pyrene	18.0906	13.0093	10.8640	12.1997	11.9117	"
Dibenz[a,h]anthracene	8.9970	7.8713	6.3735	6.7757	6.2574	"
Benzo[g,h,i]perylene	16.8298	12.9940	10.7392	11.6027	11.1130	"

% RECOVERY OF LEVEL ONE SPIKE

	Z031009	Z030616	Z030912	Z031203	Z031204	AVERAGE	%RSD
SS #1	64.557	45.968	80.627	78.107	NS	67.3	23.6
SS #2	70.842	42.349	67.152	54.255	NS	58.6	22.1
Benzo[a]anthracene	72.6610	71.7180	60.7260	85.4900	90.7050	76.3	15.6
Chrysene	73.7390	84.5750	64.0530	82.9030	99.8720	81.0	16.5
Benzo[b]fluoranthene	113.7197	65.1687	30.3857	44.0517	40.9437	58.9	56.4
Benzo[k]fluoranthene	101.2843	93.5073	77.0273	19.3783	32.6973	64.8	56.7
Benzo[a]pyrene	114.5643	79.6813	60.9793	68.3633	63.7573	77.5	28.3
Indeno[1,2,3-cd]pyrene	114.6580	63.8450	42.3920	55.7490	52.8690	65.9	43.0
Dibenz[a,h]anthracene	75.3873	64.1303	49.1523	53.1743	47.9913	58.0	20.1
Benzo[g,h,i]perylene	107.2923	68.9343	46.3863	55.0213	50.1243	65.6	37.9

ANALYSIS OF LEVEL TWO SPIKE (50 ng/g)

	Z031011	Z031012	Z031013	Z031014	Z031015	
Benzo[a]anthracene	73.0677	86.6849	68.5179	59.8031	50.8813	ng/g (wet wt.)
Chrysene	81.1269	94.3079	76.6912	65.9506	54.7417	"
Benzo[b]fluoranthene	78.4447	78.3467	54.8656	47.5773	44.3970	"
Benzo[k]fluoranthene	74.7267	74.6372	49.2486	47.3660	42.2903	"
Benzo[a]pyrene	81.1766	81.2749	65.4578	55.3167	49.1666	"

TABLE 1

Cont'd.

METHOD VALIDATION FOR
PAH ANALYSIS BY GC/MS SIM
KERR McGEE, MOSS AMERICAN SITE

Indeno[1,2,3-cd]pyrene	80.2202	55.6948	43.8813	36.9988	36.5493	"
Dibenz[a,h]anthracene	65.2277	46.9647	38.7364	31.9548	32.2629	"
Benzo[g,h,i]perylene	75.7439	50.3663	40.0950	33.5750	32.4993	"

% RECOVERY OF LEVEL TWO SPIKE

	Z031011	Z031012	Z031013	Z031014	Z031015	AVERAGE	%RSD
SS #1	99.685	124.703	96.027	93.467	89.358	100.6	13.9
SS #2	87.225	98.368	75.665	74.609	76.956	82.6	12.3
Benzo[a]anthracene	134.1164	161.3508	125.0168	107.5872	89.7436	123.6	21.9
Chrysene	142.2468	168.6088	133.3754	111.8942	89.4764	129.1	23.3
Benzo[b]fluoranthene	138.3579	138.1619	91.1997	76.6231	70.2625	102.9	32.2
Benzo[k]fluoranthene	134.1849	134.0059	83.2287	79.4635	69.3121	100.0	31.5
Benzo[a]pyrene	144.9795	145.1761	113.5419	93.2597	80.9595	115.6	25.4
Indeno[1,2,3-cd]pyrene	147.1908	98.1400	74.5130	60.7480	59.8490	88.1	41.4
Dibenz[a,h]anthracene	127.5389	91.0129	74.5563	60.9931	61.6093	83.1	33.3
Benzo[g,h,i]perylene	139.2867	88.5315	67.9889	54.9489	52.7975	80.7	44.2

ANALYSIS OF LEVEL THREE SPIKE (100 ng/g)

	Z032606	Z032607	Z032608	Z032609	Z032610	
Benzo[a]anthracene	112.5080	155.4050	136.1230	112.2290	149.4180	ng/g (wet wt.)
Chrysene	120.0260	124.4760	140.5220	117.2190	149.3060	"
Benzo[b]fluoranthene	117.3990	129.4760	147.6650	127.8990	124.5500	"
Benzo[k]fluoranthene	101.8850	128.9250	103.7550	85.7764	108.3860	"
Benzo[a]pyrene	117.3370	133.7340	142.0960	125.6760	122.8990	"
Indeno[1,2,3-cd]pyrene	130.4880	129.1680	132.1340	113.3170	142.3370	"
Dibenz[a,h]anthracene	107.2060	105.5100	114.3090	94.5075	117.0160	"
Benzo[g,h,i]perylene	116.5460	117.0430	123.2240	104.2360	130.1490	"

% RECOVERY OF LEVEL THREE SPIKE

	Z032606	Z032607	Z032608	Z032609	Z032610	AVERAGE	%RSD
SS #1	62.976	67.036	73.594	55.766	89.104	69.7	18.1
SS #2	62.381	62.620	63.548	51.690	68.096	61.7	9.8
Benzo[a]anthracene	106.4985	149.3955	130.1135	106.2195	143.4085	127.1	15.9
Chrysene	110.0225	114.4725	130.5185	107.2155	139.3025	120.3	11.6
Benzo[b]fluoranthene	108.1333	120.2103	138.3993	118.6333	115.2843	120.1	9.3
Benzo[k]fluoranthene	94.2507	121.2907	96.1207	78.1421	100.7517	98.1	15.8
Benzo[a]pyrene	108.6501	125.0471	133.4091	116.9891	114.2121	119.7	8.1
Indeno[1,2,3-cd]pyrene	123.8632	122.5432	125.5092	106.6922	135.7122	122.9	8.5
Dibenz[a,h]anthracene	105.7477	104.0517	112.8507	93.0492	115.5577	106.3	8.3
Benzo[g,h,i]perylene	110.4454	110.9424	117.1234	98.1354	124.0484	112.1	8.5

APPENDIX 1

SAMPLE COLLECTION LOG

ROY F. WESTON, INC.
THREE HAWTHORN PARKWAY, SUITE 400
VERNON HILLS, ILLINOIS 60061
708-918-4000

MOSS AMERICAN SOIL AND SEDIMENT SAMPLING: METHOD VALIDATION STUDY SAMPLES

Sampling Date: 20 February 1992
Sampling Team: T. Klemp, J. Ososkie

1. Site soil - 0945 hours
Sample was collected from the site on Chicago and North Western (CNW) Transportation Company property in an area of suspected contamination. The sample location was on the eastern end of the CNW property, immediately north of the asphalt drive in the area of the former processing facilities. Sample was collected from a 0 - 6 inch depth with a stainless steel scoop, mixed in a stainless steel bowl, and similar aliquots were scooped into 2-250 mL glass jars. Dedicated sampling equipment was used for the collection of all samples.

2. Soil background - 1045 hours
Sample was collected from a forested wetland area approximately two miles north of the site in Ozaukee County, Wisconsin. The sampling location was approximately 100 feet north of Donges Bay Road, a two-lane county road, and 50 feet west of the Little Menomonee River. The surrounding area was predominantly agricultural. Sample was collected from a 0-6 inch depth with a stainless steel hand trowel. A composite sample was collected from several locations, mixed in a stainless steel bowl, and similar aliquots were scooped into 6-250 mL glass jars. Dedicated sampling equipment was used for the collection of all samples.

3. Sediment background - 1115 hours
Sample was collected from the stream bed of Little Menomonee River approximately two miles north of the site in Ozaukee County, Wisconsin. The sample location was approximately 50 feet south of Donges Bay Road. The stream bed was sandy, with many cobbles and large rocks. The surrounding area was predominantly agricultural. Sample was collected from a 0-6 inch depth with a stainless steel scoop, mixed in a stainless steel bowl, and similar aliquots were scooped into 2-250 mL glass jars. Dedicated sampling equipment was used for the collection of all samples.

APPENDIX 2

METHOD PERFORMANCE STUDY
DATA PACKAGE

ROY F. WESTON, INC.
Lionville Laboratory

CLIENT: KERR McGEE **SAMPLES RECEIVED:** 02-21-92
RFW #: 9202L422, POLYAROMATIC HYDROCARBONS
W.O. #: 2687-07-01

NARRATIVE

The set of samples consisted of two (2) soil samples and one (1) sediment sample collected on 02-20-92.

The samples were extracted on 02-28-92 and analyzed by GC/MS Selected ION Monitoring for Client Specified target compounds on 03-06,09,10-92.

The following is a summary of the QC results accompanying the sample results and a description of any problems encountered during their analyses:

1. All obtainable surrogate recoveries were within QC limits.
2. Three (3) of sixteen (16) sediment matrix spike recoveries were outside QC limits.
3. All blank spike recoveries were within QC limits.

04.02.92.

Jack R. Tuschall, Ph.D. **Date**
Laboratory Manager
Lionville Analytical Laboratory

PAH DATA FLAGS

B = Compound was detected in the associated method blank.

D = Indicates compounds that were quantitated from an analysis at a secondary dilution factor. Also used to indicate that surrogate recoveries were not obtained because the extract had to be diluted for analysis.

E = Indicates an estimated value. Used in cases where a target analyte is detected at a level greater than the upper quantification limit of instrument calibration.

H = Due to matrix interference, peak height (instead of peak area) was used to calculate the isotopic and to quantitate the analyte. Peak must meet all other identification criteria.

J = Indicates an estimated value. Used in cases where a target analyte is detected at a level less than the lower quantification limit of instrument calibration.

NA = Not applicable

ND = None Detected

NR = Not Required

P = Poor resolution of 2,3,7,8 substituted peak.

Q = Associated internal standard recovery is outside QC limits. However, signal to noise ratio is greater than 10 to 1 and data is valid.

S = Signal to noise ratio of M-COCL does not meet 2.5 S/N requirement, but peak was determined to be positive in the judgement of the GC/MS analyst.

U = Indicates that the compound was analyzed for but not detected. The minimum detection limit for the sample (not the method detection limit) is reported with the U.

X = Isotope ratio criteria are not met using either peak height or peak area (used for EMPC only).

Roy F. Weston, Inc. - Lionville Laboratory
Poly Aromatic Hydrocarbons by GCMS
Report Date: 04/09/92 15:12
RFW Batch Number: 9202L422
Client: KERR MCGEE
Work Order: 2687-07-01-0000
Page: 1a

Cust ID:	SITE SOIL	fl	SEDIMENT-BKG ND	fl	SEDIMENT-BKG ND	fl	SEDIMENT-BKG ND	fl	SEDIMENT-BKG ND	fl	SOIL-BKGND	fl
Sample Information RFW#:	001		002		002 REP		002 MS		002 MSD		003	
Matrix:	SOIL		SEDIMENT		SEDIMENT		SEDIMENT		SEDIMENT		SOIL	
D.F.:	20.0		1.00		1.00		1.00		1.00		1.00	
Units:	ug/Kg		ug/Kg		ug/Kg		ug/Kg		ug/Kg		ug/Kg	
Chrysene-d12	D	%	78	%	92	%	85	%	101	%	57	%
Surrogate benz[a,h]anthracene-d14	D	%	86	%	78	%	80	%	70	%	54	%
Benzo[a]anthracene	4500		19		16		44	%	90	%	9	
Chrysene	6600	E	28		25		55	%	110	%	16	
Benzo[b]fluoranthene	4900		34		33		123	%	17 *	%	15	
Benzo[k]fluoranthene	2800		18		18		82	%	73	%	14	
Benzo[a]pyrene	2800		26		20		28	%	55	%	13	
Indeno[1,2,3-cd]pyrene	2300		21		15		32	%	23	%	11	
Dibenz[a,h]anthracene	340		4		4		53	%	51	%	3	J
Benzo[g,h,i]perylene	2900		25		15		9 *	%	4 *	%	11	

Cust ID:	BLK	fl	BLK BS	fl
Sample Information RFW#:	92LS0016-MB1		92LS0016-MB1	
Matrix:	SOIL		SOIL	
D.F.:	1.00		1.00	
Units:	ug/Kg		ug/Kg	
Chrysene-d12	68	%	57	%
Surrogate benz[a,h]anthracene-d14	64	%	69	%
Benzo[a]anthracene	1	U	51	%
Chrysene	1	U	52	%
Benzo[b]fluoranthene	1	U	56	%
Benzo[k]fluoranthene	1	U	68	%
Benzo[a]pyrene	1	U	64	%
Indeno[1,2,3-cd]pyrene	1	U	54	%
Dibenz[a,h]anthracene	1	U	55	%
Benzo[g,h,i]perylene	1	U	53	%

U= Analyzed, not detected. J= Present below detection limit. B= Present in blank. NR= Not requested. NS= Not spiked.
%= Percent recovery. D= Diluted out. I= Interference. NA= Not Applicable. *= Outside of EPA CLP QC

Roy F. Weston, Inc. - Lionville Laboratory
PAH ANALYTICAL DATA PACKAGE FOR
KERR MCGEE

DATE RECEIVED: 02/21/92 RFW LOT # :9202L422

CLIENT ID	RFW #	MTX	PREP #	COLLECTION	EXTR/PREP	ANALYSIS
SITE SOIL	001	S	92LS0016	02/20/92	02/28/92	03/10/92
SEDIMENT-BKGND	002	SE	92LS0016	02/20/92	02/28/92	03/10/92
SEDIMENT-BKGND	002 REP	SE	92LS0016	02/20/92	02/28/92	03/09/92
SEDIMENT-BKGND	002 MS	SE	92LS0016	02/20/92	02/28/92	03/09/92
SEDIMENT-BKGND	002 MSD	SE	92LS0016	02/20/92	02/28/92	03/09/92
SOIL-BKGND	003	S	92LS0016	02/20/92	02/28/92	03/06/92
LAB QC:						
BLK	MB1	S	92LS0016	N/A	02/28/92	03/06/92
BLK	MB1 BS	S	92LS0016	N/A	02/28/92	03/09/92

WESTON Analytics Use Only
9202L422

Custody Transfer Record/Lab Work Request

WESTON
Page 1 of 1

Client KERR MCGEE /MOSS- AMERICAN
Est. Final Proj. Sampling Date 2-20-92
Work Order # 2687-07-01
Project Contact/Phone # GARY DIEGAN/x 4114
AD Project Manager Gail DeRuyter/Pete Hensley
QC STD Del STD TAT 14 day
Date Rec'd 2/21/92 Date Due 3/10/92
Account # KERRMGMDL

		VOA	BNA	Pest/PCB	Herb	PAH	PAH	HPLC Scan	Metal	CN						
Refrigerator #						6	6									
#/Type Container	Liquid															
	Solid					2GL	6GL	→								
Volume	Liquid															
	Solid					250mL	250mL	→								
Preservatives	Ice					Ice	Ice									

ANALYSES REQUESTED → ORGANIC: VOA, BNA, Pest/PCB, Herb; PAH; PAH; HPLC Scan; INORG: Metal, CN

WESTON Analytics Use Only: OPAH, OPAH, OLCSC

MATRIX CODES:
S - Soil
SE - Sediment
SO - Solid
SL - Sludge
W - Water
O - Oil
A - Air
DS - Drum Solids
DL - Drum Liquids
L - EP/TCLP Leachate
WI - Wipe
X - Other
F - Fish

Lab ID	Client ID/Description	MS	MSD	Matrix	Date Collected 1992	Time Collected	VOA	BNA	Pest/PCB	Herb	OPAH	OPAH	OLCSC	Metal	CN						
001	SITE SOIL			S	2-20	0445					X		✓								
002	SEDIMENT - BKGND			SE	2-20	1045					X		✓								
003	SOIL BKGND			S	2-20	1115						X	✓								

FIELD PERSONNEL: COMPLETE ONLY SHADED AREAS

Special Instructions:

Attn: Joe Loeper
** do not throw away extra sample
* Site soil - 2 - 250mL
Sediment bkgnd - 2-250mL
Soil bkgnd - 6- 250mL

DATE/REVISIONS:
3-12-92 1. add HPLC scan to samples #001 - #003
2. Per Joe Loeper [illegible] % sol to
3. Sample #002 [illegible]
4.
5.
6.

Relinquished by	Received by	Date	Time
T. Klimp	FEDEX	2-20-92	1830
Fed-Ex	Brian Shaffer	2-21-92	0930

Relinquished by	Received by	Date	Time
	BKS 2-21-92		

Discrepancies Between Samples Labels and COC Record? [✓ Y] or N
NOTES: Rec'd 3 Bottles for Site Soil but time says 1115

WESTON Analytics Use Only

Samples were:
1) Shipped ✓ or Hand Delivered ___ Airbill # ___
2) Ambient or [✓ Chilled]
3) Received in Good Condition [✓ Y] or N
4) Labels Indicate Properly Preserved [✓ Y] or N
5) Received Within Holding Times [✓ Y] or N

COC Tape was:
1) Present on Outer Package Y or [✓ N]
2) Unbroken on Outer Package Y or [✓ N]
3) Present on Sample Y or [✓ N]
4) Unbroken on Sample Y or [✓ N]
COC Record Present Upon Sample Rec't [✓ Y] or N

RFW 21-21-001/A-7/91 ___ L372 ___ L373 ___ L375 ___ L377 ___ L378 Ref# 5765 Cooler# N/A 381-596a

Roy F. Weston, Inc. - Lionville Laboratory
LCSC ANALYTICAL DATA PACKAGE FOR
KERR MCGEE

DATE RECEIVED: 02/21/92 **RFW LOT # :**9202L422

CLIENT ID	RFW #	MTX	PREP #	COLLECTION	EXTR/PREP	ANALYSIS
SITE SOIL	001	S	92LLC030	02/20/92	03/04/92	03/17/92
SEDIMENT-BKGND	002	SE	92LLC030	02/20/92	03/04/92	03/17/92
SOIL-BKGND	003	S	92LLC030	02/20/92	03/04/92	03/17/92
LAB QC:						
BLK	MB1	S	92LLC030	N/A	03/04/92	03/17/92
BLK	MB1 BS	S	92LLC030	N/A	03/04/92	03/17/92
BLK	MB1 BSD	S	92LLC030	N/A	03/04/92	03/17/92

WESTON

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WESTON

CHAIN OF CUSTODY

WESTON Analytics Use Only
9202L422

Custody Transfer Record/Lab Work Request

WESTON

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Client KERR MCGEE / MOSS-AMERICAN
Est. Final Proj. Sampling Date 2-20-92
Work Order # 2WB7-07-01
Project Contact/Phone # GARY DIEGAN / X 4114
AD Project Manager [illegible]
QC SH Del SH TAT 14 day
Date Rec'd 2/21/92 Date Due 3/10/92
Account # KERRMGMDL

		VOA	BNA	Pest/PCB	Herb	PAH	PAH		Metal	CN
Refrigerator #						6	6			
#/Type Container	Liquid									
	Solid					2GL	6GL			
Volume	Liquid									
	Solid					250mL	250mL			
Preservatives	Ice					Ice	Ice			

ANALYSES REQUESTED → ORGANIC: VOA, BNA, Pest/PCB, Herb; PAH; PAH; INORG: Metal, CN

WESTON Analytics Use Only

MATRIX CODES:
S - Soil
SE - Sediment
SO - Solid
SL - Sludge
W - Water
O - Oil
A - Air
DS - Drum Solids
DL - Drum Liquids
L - EP/TCLP Leachate
WI - Wipe
X - Other
F - Fish

Lab ID	Client ID/Description	Matrix QC Chosen (✓) MS	MSD	Matrix	Date Collected 1992	Time Collected	9PAH	0PAH
001	SITE SOIL			S	2-20	0445	X	
002	SEDIMENT - BKGND			SE	2-20	1045	X	
003	SOIL - BKGND			S	2-20	1115		X

FIELD PERSONNEL: COMPLETE ONLY SHADED AREAS

Special Instructions:

Attn: Joe Loeper

** do not throw away extra sample
* Site soil - 2 - 250mL
Sediment bkgnd - 2 - 250mL
Soil bkgnd - 6 - 250mL

DATE/REVISIONS:
1.
2.
3.
4.
5.
6.

Relinquished by	Received by	Date	Time
T. Klump	FEDEX	2-20-92	1830
Fed-Ex	Brian Dhoff	2-21-92	0930 am

Relinquished by	Received by	Date	Time
	BKS 2-21-92		

WESTON Analytics Use Only

Samples were:
1) Shipped ✓ or Hand Delivered ___
Airbill # ___
2) Ambient or [✓ Chilled]
3) Received in Good Condition [✓ Y] or N
4) Labels Indicate Properly Preserved [✓ Y] or N
5) Received Within Holding Times [✓ Y] or N

COC Tape was:
1) Present on Outer Package Y or [✓ N]
2) Unbroken on Outer Package Y or [✓ N]
3) Present on Sample Y or [✓ N]
4) Unbroken on Sample Y or [✓ N]
COC Record Present Upon Sample Rec't [✓ Y] or N

Discrepancies Between Samples Labels and COC Record ? [✓ Y] or N
NOTES: Rec'd 3 Bottles for Site Soil but have [illegible]

RFW 21-21-001/A-7/91 ___ L372 ___ L373 ___ L375 ___ L377 ___ L378 Ref# 5765 Cooler# N/A 381-596a

WESTON

DATA SUMMARY

Roy F. Weston, Inc. - Lionville Laboratory

HPLC scan

Report Date: 03/25/92 08:57

RFW Batch Number: 9202L422 Client: KERR MCGEE Work Order: 2687-07-01-0000 Page: 1

Cust ID:	SITE SOIL	fl	SEDIMENT-BKGND	fl	SOIL-BKGND	fl	BLK	fl	BLK BS	fl	BLK BSD	fl
Sample Information RFW#:	001		002		003		92LLC030-MB1		92LLC030-MB1		92LLC030-MB1	
Matrix:	SOIL		SEDIMENT		SOIL		SOIL		SOIL		SOIL	
D.F.:	100		1.00		1.00		1.00		1.00		1.00	
Units:	ug/kg		ug/kg		ug/kg		ug/kg		ug/kg		ug/kg	
Benzo(a)anthracene	3400		22		18		10	U	89	%	98	%
Chrysene	4300		46	J	40	J	50	U	82	%	96	%
Benzo(b)fluoranthrene	4200		49		26		10	U	111	%	113	%
Benzo(k)fluoranthrene	1900		19		14		10	U	131	%	139	%
Benzo(a)pyrene	3200		42		32		10	U	99	%	113	%
Dibenzo(a,h)anthracene	640		2.9	J	2.5	J	10	U	96	%	108	%
Benzo(ghi)perylene	3600		32		24		10	U	76	%	85	%
Indeno(1,2,3-cd)pyrene	4400		56		52		50	U	96	%	117	%

U= Analyzed, not detected. J= Present below detection limit. B= Present in blank. NR= Not requested. NS= Not spiked. %= Percent recovery. D= Diluted out. I= Interference. NA= Not Applicable. *= Outside of EPA CLP QC

CASE NARRATIVE

5A

ROY F. WESTON, INC.
Lionville Laboratory

CLIENT: KERR McGEE
RFW #: 9202L422
W.O. #: 2687-07-01

SAMPLES RECEIVED: 02-21-92

NARRATIVE

Samples have been prepared and analyzed for selected Polyaromatic Hydrocarbon target compounds by EPA Method 8310.

The QA/QC control samples have been analyzed concurrently with each extraction batch. Abbreviations noted below have been used in the data summary.

Abbreviation		Description
BLK	=	Reagent blank analyzed to provide an indication of lab contamination and its effect on reported analytical data.
BS	=	Designates reagent blank spiked with target compounds.
BSD	=	Designates reagent blank spiked with target compounds in duplicate.

NOTE: Spikes have been reported as percent (%) recovery.

NOTE: Sample site soil exhibited fairly high levels of late eluting non-target compounds. A gel-permeation clean-up is being investigated as a clean-up option for the removal of these potential interferences. Data related to this investigation will be submitted under separate cover.

Analysis Summary

Samples Collected: 02-20-92
Samples Prepared: 03-04-92
Samples Analyzed: 03-17-92

04.09.92
Jack R. Tuschall, Ph.D. — Date
Laboratory Manager
Lionville Analytical Laboratory

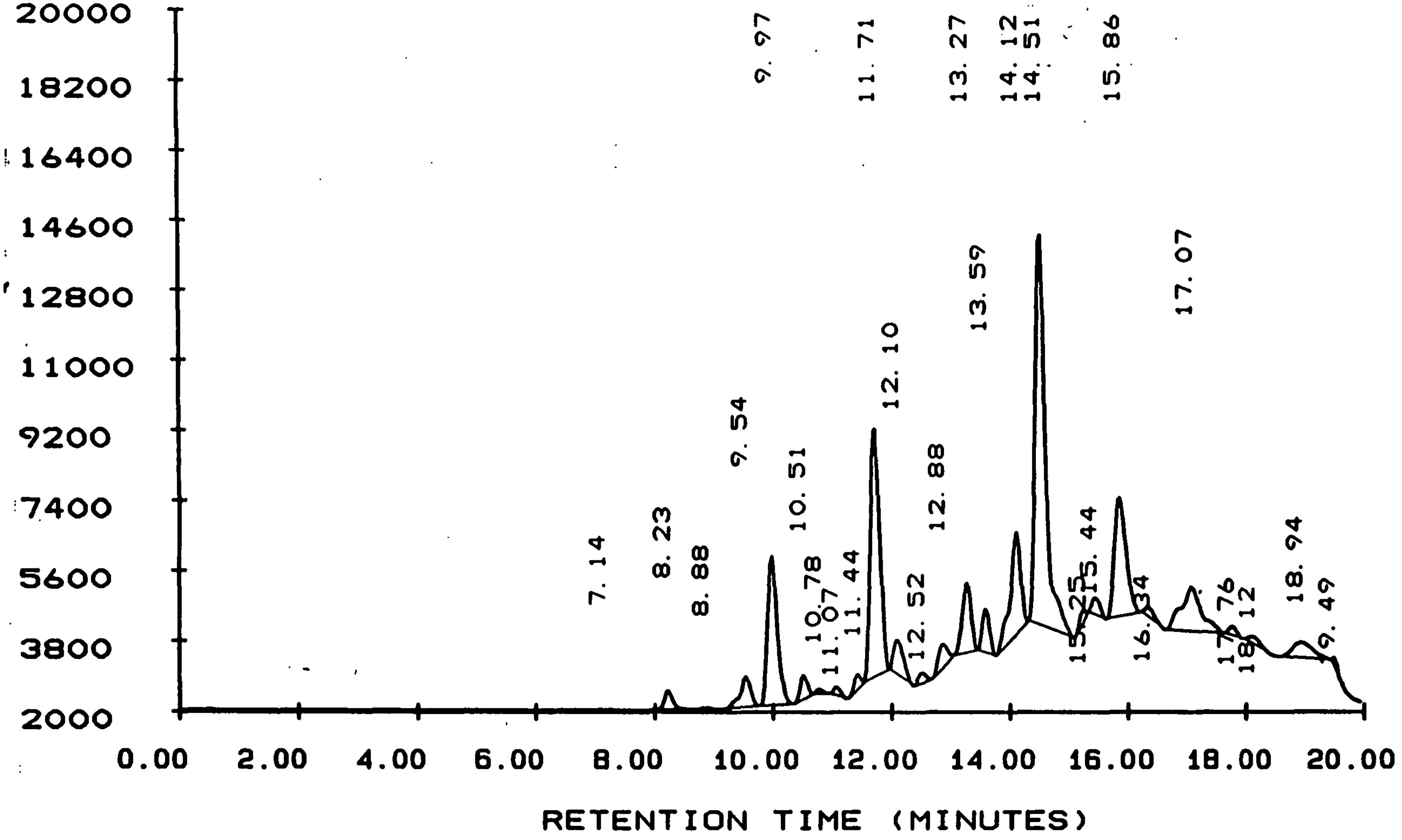
WESTON

SAMPLE DATA
in increasing RFW# order

9202L422-003R2

SAMPLE NO. : 03179217 .13
TEST NO. :
METHOD NO. : PAHF1 / PAHF1

INSTRUMENT: 17
DATE TIME: 03/17/92 15:53:43
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

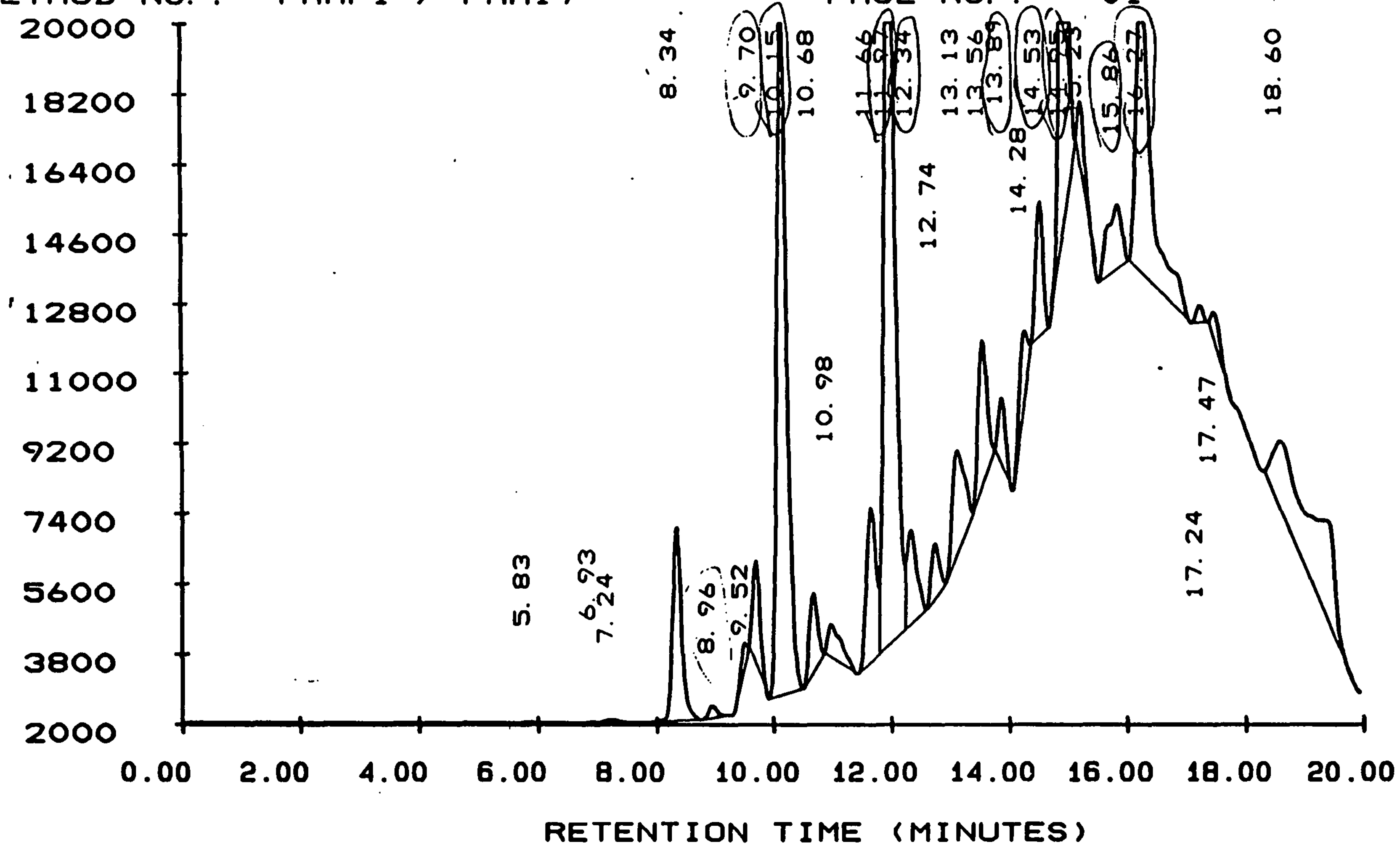
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                03/17/92 16:20:08

                           MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217    .13                        INST:17 VIAL:F0 SEQ NUMBER:013
TEST :                               DATE-TIME INJECTED   : 03/17/92 15:53:43
COLLECTION TIME :   19.96            DATE-TIME PROCESSED  : 03/17/92 16:20:08
METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG         SAMP RATE:   0.78
CLIENT ID: SOIL-BKGND                                         SAMPLE VOL:
CLIENT: KERR MCGEE                           COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: 9202L422-003                                RAW FILE: RAW2:CH243040
SAMPLE WT :               % MOISTURE   :            DILUTION FACTOR :  1.0000

                                                               HEIGHT
PK        PEAK       PEAK   BL    RT   GR COMPONENT             CONC
NO        AREA      HEIGHT     MINUTES #   NAME                 NG/UL
                            T    0.000
                            V    0.000
003       2147        133   V    7.140
004      48897       4644   V    8.229   PHENANTHRENE          0.294
005       4525        393   V    8.878   ANTHRACENE            0.187
006      98385       7501   V    9.538   FLUORANTHRENE         0.378
007     439891      37649   V    9.974   PYRENE                0.936
008      52839       5788   V   10.514
009       9130       1135   V   10.778
010      17191       2116   V   11.067
011      57446       5465   T   11.437
012     812915      67095       11.714   BENZO(A)ANTHRACENE    0.079 ✓
013     176400      12539   V   12.100   CHRYSENE              0.176 ✓
014      24972       2498   V   12.523
015      64022       5789   V   12.878
016     185878      17525   V   13.273
017      98896      10896   V   13.590   BENZO(B)FLUORANTHREN  0.115 ✓
018     317243      25412   V   14.116   BENZO(K)FLUORANTHREN  0.059 ✓
019    1179593      98951       14.512   BENZO(A)PYRENE        0.139 ✓
020      20829       2414   V   15.248
021      41730       4203   V   15.445   DIBENZO(A,H)          0.011 ✓
022     417746      30219   V   15.858   BENZO(GHI)            0.104 ✓
023      25515       2455   V   16.335
024     276097      11304   V   17.073
025      21402       2365   V   17.765
026      24707       1933   V   18.115                              PH 3/18/92
027      95181       3930   V   18.944
028       8910       1712       19.494


All compounds were quantitated using method PAH17
except those which are labeled.
```

9202L422-001R2

SAMPLE NO. : 03179217 .15
TEST NO. :
METHOD NO. : PAHF1 / PAH17

INSTRUMENT: 17
DATE TIME: 03/17/92 16:42:21
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

START TIME: 0.00
END TIME: 20.00

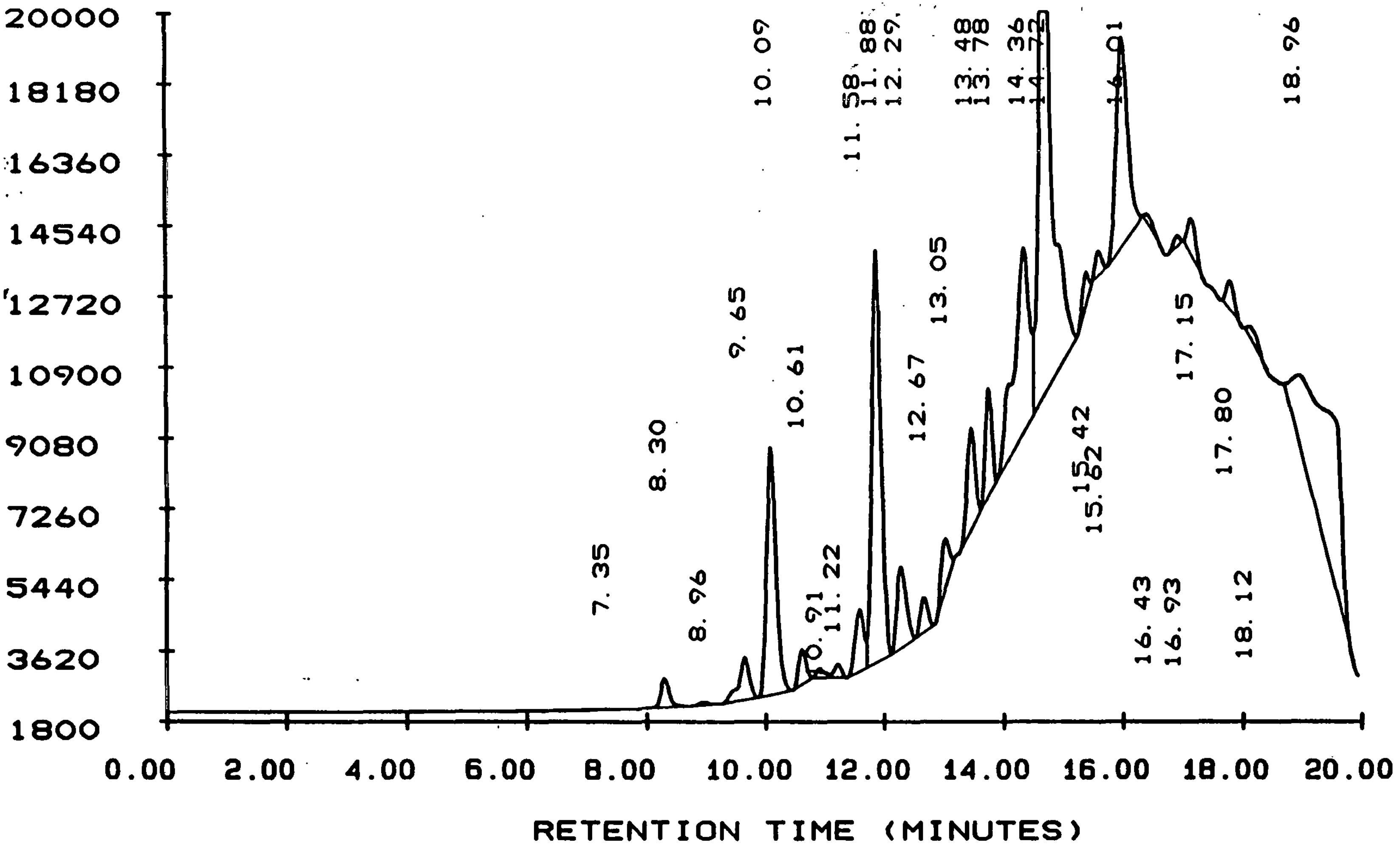
```
Roy F. Weston, Inc. - Lionville Laboratory                03/17/92 17:18:59

                        MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217    .15                         INST:17 VIAL:F0 SEQ NUMBER:015
TEST :                                   DATE-TIME INJECTED  : 03/17/92 16:42:21
COLLECTION TIME :   19.96                DATE-TIME PROCESSED : 03/17/92 17:18:59
METHOD:  PAHF1 / PAH17   REV #: 00062  ANALYST: HUANG         SAMP RATE:   0.78
CLIENT ID: SITE SOIL                                         SAMPLE VOL:
CLIENT: KERR MCGEE                                    COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: 9202L422-001                                     RAW FILE: RAW2:CH243077
SAMPLE WT :                 % MOISTURE  :              DILUTION FACTOR :100.0000

                                                                 HEIGHT
PK       PEAK      PEAK   BL    RT   GR COMPONENT                  CONC
NO       AREA     HEIGHT   MINUTES  #   NAME                      NG/UL
002      3904       289   V   5.834
003      1182       154   V   6.929
004     10362       766   V   7.243
005    534366     48700   V   8.343     PHENANTHRENE             154.198
006     32832      3064   V   8.957     ANTHRACENE                33.935
007     41128      3514   V   9.522
008    223347     25992   V   9.697     FLUORANTHRENE            118.026
009   2110785    180481   V  10.146     PYRENE                   417.991
010    190467     19440   V  10.680
011    140042      8408   V  10.984
012    446947     38881   T  11.663
013   2622491    202260   T  11.968     BENZO(A)ANTHRACENE        23.212 ✓
014    282168     23592   V  12.337     CHRYSENE                  29.086 ✓
015    135303     13493   T  12.743
016    494092     29539   T  13.129
017    651248     48908   T  13.565
018    305454     27416   V  13.887     BENZO(B)FLUORANTHREN      28.327 ✓
019    392618     33301   T  14.283
020    738628     56982   T  14.534     BENZO(K)FLUORANTHREN      13.179 ✓
021   2166623    154827   T  14.955     BENZO(A)PYRENE            21.939 ✓
022    756306     57757   V  15.226
023    289799     16337   V  15.858     DIBENZO(A,H)               4.353 ✓
024   1313787     72901   V  16.275     BENZO(GHI)                24.460 ✓
025     35519      4417   V  17.242
026     78451      7844   V  17.474
027   1190927     18295      18.605


All compounds were quantitated using method PAH17
except those which are labeled.
```

3/18/92

9202L422-002R2

SAMPLE NO. : 03179217 .16
TEST NO. :
METHOD NO. : PAHF1 / PAH17

INSTRUMENT: 17
DATE TIME: 03/17/92 17:06:40
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1800.

START TIME: 0.00
END TIME: 20.00

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    Roy F. Weston, Inc. - Lionville Laboratory                03/18/92 13:52:54

                          MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217    .16                       INST:17 VIAL:F0 SEQ NUMBER:016
TEST :                              DATE-TIME INJECTED  : 03/17/92 17:06:40
COLLECTION TIME :   19.96           DATE-TIME PROCESSED : 03/18/92 13:52:54
METHOD:  PAHF1 / PAH17   REV #: 00063  ANALYST: HUANG         SAMP RATE:   0.78
CLIENT ID: SEDIMENT-BKGND                                     SAMPLE VOL:
CLIENT: KERR MCGEE                                  COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: 9202L422-002                                      RAW FILE: RAW2:CH243094
SAMPLE WT :                 % MOISTURE  :               DILUTION FACTOR :  1.0000

                                                                  HEIGHT
PK      PEAK       PEAK   BL   RT   GR COMPONENT                  CONC
NO      AREA      HEIGHT   MINUTES  #   NAME                       NG/UL
003     2026        126   V   7.348
004    75318       7147   V   8.300   PHENANTHRENE                0.365
005     9308        781   V   8.962   ANTHRACENE                  0.209
006   136176      10557   V   9.647   FLUORANTHRENE               0.510
007   695821      62494   V  10.085   PYRENE                      1.500
008    79520       8692   V  10.614
009    25639       2346   T  10.914
010    31091       3447   V  11.217
011   170116      15553   T  11.582
012  1136127     104385   V  11.876   BENZO(A)ANTHRACENE          0.122 ✓
013   228933      20320   T  12.292   CHRYSENE                    0.257 ✓
014    78895       8348   V  12.673
015   111666      11346   V  13.048
016   232242      25088   V  13.478
017   201812      26310   V  13.778   BENZO(B)FLUORANTHREN        0.272 ✓
018   844750      46589   T  14.365   BENZO(K)FLUORANTHREN        0.108
019  2193479     164105   V  14.715   BENZO(A)PYRENE              0.233
020    56080       7011   V  15.416
021    49890       5971   V  15.623   DIBENZO(A,H)                0.016
022   706120      52948   V  16.010   BENZO(GHI)                  0.179
023    22686       2630   V  16.430
024    21235       2586   V  16.931
025    91534       9900   V  17.155
026    65142       7465   V  17.804
027    40004       2782   V  18.121
028  1562831      17987      18.962

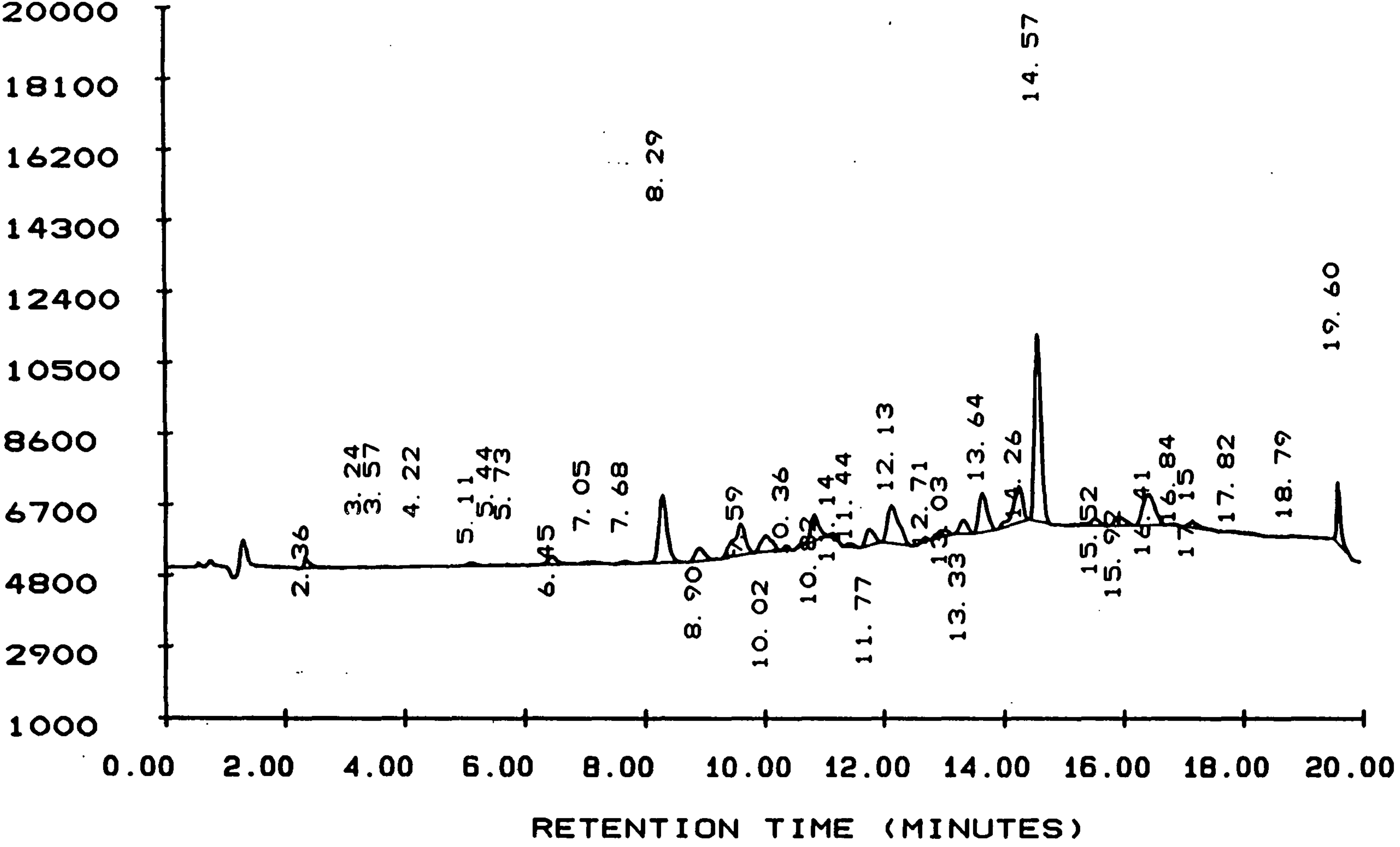
All compounds were quantitated using method PAH17
except those which are labeled.
```

3/18/92

9202L422-003R2

SAMPLE NO. : 03179218 .13
TEST NO. :
METHOD NO. : PAHU1 / PAHU1

INSTRUMENT: 18
DATE TIME: 03/17/92 15:53:43
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                    03/17/92 16:21:15

                            MULTILEVEL EXTERNAL STANDARD

  SAMPLE: 03179218    .13                          INST:18 VIAL:F0 SEQ NUMBER:013
  TEST :                                   DATE-TIME INJECTED  : 03/17/92 15:53:43
  COLLECTION TIME :   19.96                DATE-TIME PROCESSED : 03/17/92 16:21:15
  METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG           SAMP RATE:   0.78
  CLIENT ID: SOIL-BKGND                                           SAMPLE VOL:
  CLIENT: KERR MCGEE                                  COLUMN TYPE: PAH'S BY HPLC UV DET
  LAB ID: 9202L422-003                                      RAW FILE: RAW2:CH243041
  SAMPLE WT :                % MOISTURE  :              DILUTION FACTOR :  1.0000

                                                                  HEIGHT
  PK      PEAK      PEAK   BL    RT   GR COMPONENT                 CONC
  NO      AREA     HEIGHT     MINUTES #   NAME                    NG/UL
  001     18718      2333  V    2.364
  002      2274       165  V    3.245
  003      1132       129  V    3.570
  004      5500       226  V    4.215
  005     10874       922  V    5.115
  006      1746       202  V    5.443
  007      3450       270  V    5.729
                                5.880    NAPHTHALENE
  008     27856      2338  V    6.454
                                6.590    ACENAPHTHYLENE
  009     10804       592  V    7.050
                                7.430    ACENAPHTHENE
  010      6566       633  V    7.683    FLUORENE
  011    180481     17704  V    8.286
  012     41762      3446  V    8.900
  013    117999      8165  V    9.592
  014     59565      4289  V   10.017
  015      7681      1268  V   10.365
  016     74572      6858  V   10.825
  017     10210      1454  V   11.137
  018      6972       855  V   11.436
  019     42490      4184  V   11.766
  020    134951      9889  V   12.131
  021      8184      1017  V   12.707
  022     22390      1523  V   13.034
  023     34263      3570  V   13.334
  024    103206     10165  V   13.645
  025    109370      9160  V   14.263
  026    364994     47689  V   14.570
  027     28818      1800  V   15.521
  028     31898      2417  V   15.916
  029    119156      8181  V   16.411    INDENO(1,2,3-CD)        0.225
  030      3220       395  V   16.841
  031     19394      1333  V   17.148
  032     10742       411  V   17.823
  033      7433       290  V   18.790
  034     77626     13640      19.595

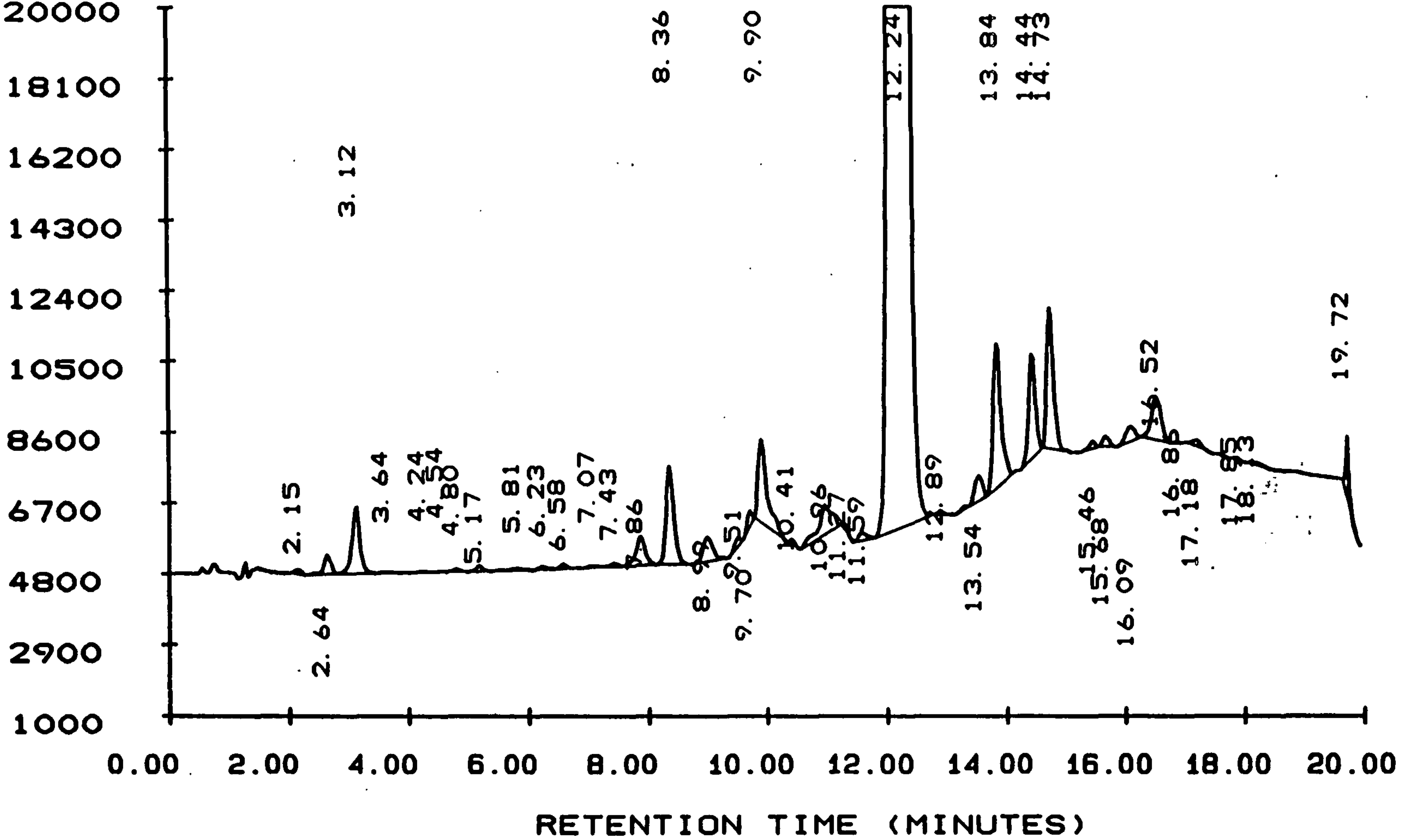

  All compounds were quantitated using method PAH18
  except those which are labeled.
```

✓ RH 3/18/92

9202L422-002R2

SAMPLE NO. : 03179218 .16
TEST NO. :
METHOD NO. : PAHU1 / PAH18

INSTRUMENT: 18
DATE TIME: 03/17/92 17:06:41
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                     03/17/92 17:32:45

                            MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179218    .16                             INST:18 VIAL:F0 SEQ NUMBER:016
TEST :                                     DATE-TIME INJECTED   : 03/17/92 17:06:41
COLLECTION TIME :   19.96                  DATE-TIME PROCESSED  : 03/17/92 17:32:45
METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG         SAMP RATE:    0.78
CLIENT ID: SEDIMENT-BKGND                                     SAMPLE VOL:
CLIENT: KERR MCGEE                                      COLUMN TYPE: PAH'S BY HPLC UV DET
LAB ID: 9202L422-002                                          RAW FILE: RAW2:CH243095
SAMPLE WT :                 % MOISTURE  :                DILUTION FACTOR :   1.0000

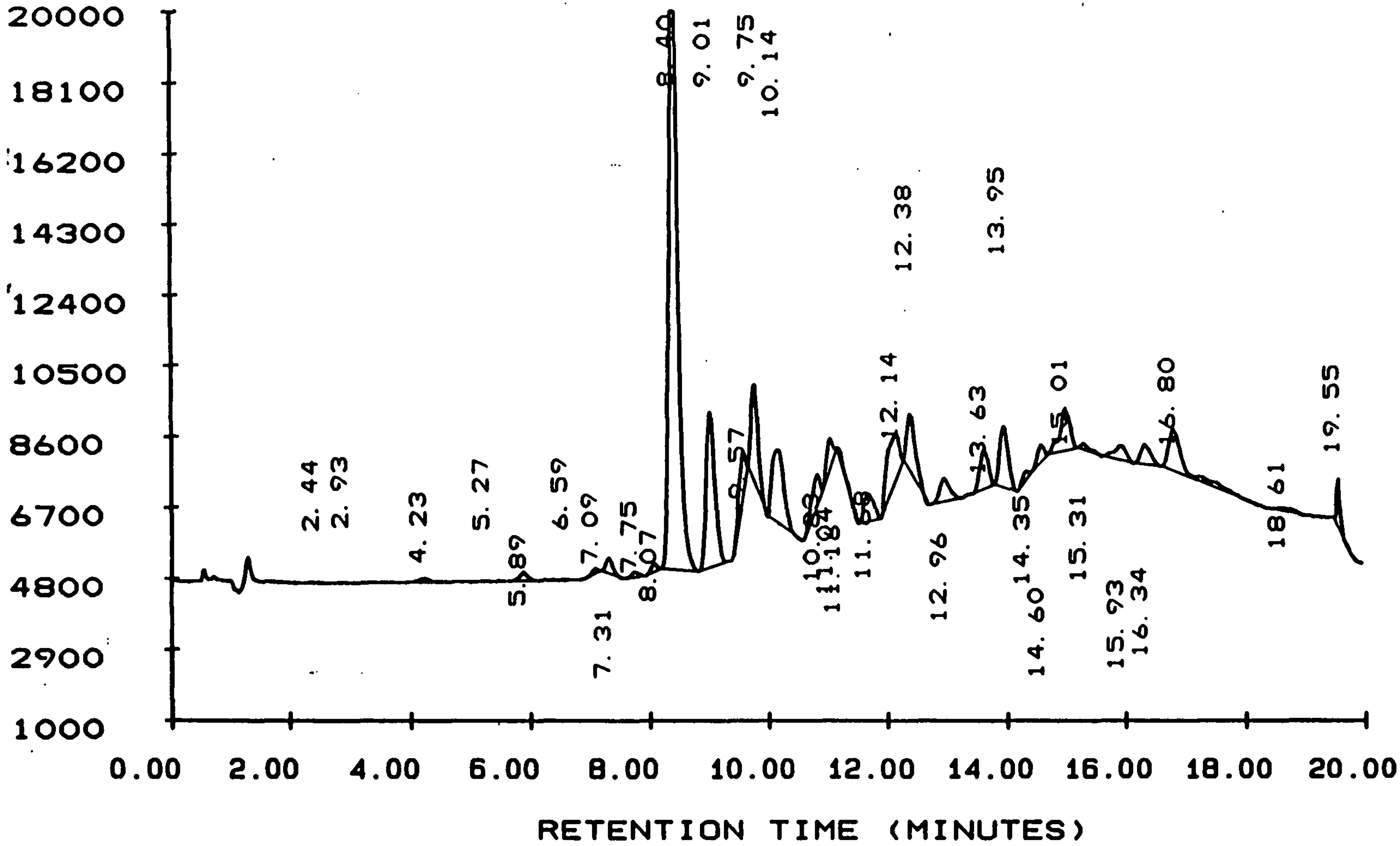
                                                              HEIGHT
PK        PEAK      PEAK  BL    RT   GR COMPONENT              CONC
NO        AREA     HEIGHT    MINUTES #   NAME                  NG/UL
001       9802      1155  V   2.153
002      44119      4845  V   2.640
003     165906     17203  V   3.118
004       3986       311  V   3.635
005       3116       281  V   4.242
006       1379       198  V   4.543
007       5325       681  V   4.797
008      12222      1401  V   5.168
009      10884       575  V   5.805   NAPHTHALENE
010       7134       671  V   6.230
011       8423      1064  V   6.576   ACENAPHTHYLENE          0.133
012       2867       331  V   7.072
013       6566       781  V   7.430   ACENAPHTHENE            0.047
014      89284      7709  V   7.859   FLUORENE                0.199
015     245457     25769  V   8.356
016      80522      6591  V   8.989
017      14938      1829  V   9.513
018      19499      3490  V   9.705
019     248415     22196  V   9.899
020       6038      1137  V  10.408
021     121866      7607  V  10.964
022       8527      1918  V  11.267
023      22978      2050  V  11.590
024    9460176    042651  V  12.245
025       5645       907  V  12.894
026      67863      6526  V  13.541
027     352512     37651  V  13.841
028     216878     26542  V  14.442
029     298743     36265  V  14.735
030      13646      1787  V  15.460
031      21686      2614  V  15.675
032      42170      3710  V  16.094
033     128806     11468  V  16.520   INDENO(1,2,3-CD)        0.314
034       1479       239  V  16.863
035      15826      1572  V  17.182
036       3255       476  V  17.850
037       7635       426  V  18.130
038      56000     12706     19.721


All compounds were quantitated using method PAH18
except those which are labeled.
```

9202L422-001R2

SAMPLE NO. : 03179218 .15
TEST NO. :
METHOD NO. : PAHU1 / PAH18

INSTRUMENT: 18
DATE TIME: 03/17/92 16:42:21
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0.00
END TIME: 20.00

```
Roy F. Weston, Inc. - Lionville Laboratory                 03/17/92 17:08:54

                    MULTILEVEL MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179218    .15                       INST:18 VIAL:F0 SEQ NUMBER:015
TEST :                                  DATE-TIME INJECTED  : 03/17/92 16:42:21
COLLECTION TIME :   19.96               DATE-TIME PROCESSED : 03/17/92 17:08:54
METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG        SAMP RATE:   0.78
CLIENT ID: SITE SOIL                                         SAMPLE VOL:
CLIENT: KERR MCGEE                              COLUMN TYPE: PAH'S BY HPLC UV DET
LAB ID: 9202L422-001                                 RAW FILE: RAW2:CH243078
SAMPLE WT :                 % MOISTURE  :          DILUTION FACTOR :100.0000

                                                                 HEIGHT
PK        PEAK      PEAK   BL    RT   GR COMPONENT                 CONC
NO        AREA      HEIGHT     MINUTES #   NAME                    NG/UL
001       2570        180   V   2.443
002        549        101   V   2.934
004      15405       1038   V   4.225
005       2599        147   V   5.275
007      21984       2176   V   5.887    NAPHTHALENE              13.696
008       1653        134   V   6.594    ACENAPHTHYLENE
009       8132       1066   V   7.092
010      34195       4115   V   7.308    ACENAPHTHENE            100.029
011      11110       1289   V   7.752    FLUORENE                  1.458
012      15392       2058   V   8.075
013    1708339     169562   V   8.399
014     411571      40709   V   9.011
015      86231       9469   V   9.573
016     207940      26096   V   9.751
017     271074      19678   V  10.144
018      77651       7221   V  10.828
019      50488       6857   V  11.042
020      22754       2438   V  11.163
021      86038       7283   V  11.683
022     180955      11013   V  12.139
023     153347      15511   V  12.383
024      76370       6155   V  12.959
025     106072      10082   V  13.628
026     164399      15972   V  13.950
027      15502       1674   V  14.352
028      36727       4126   V  14.596
029     135008      10853   V  15.011
030      16362       1572   V  15.309
031      55142       3920   V  15.935
032      64021       5228   V  16.335
033     192126      10839   V  16.803    INDENO(1,2,3-CD)         29.689
034      12491        714   V  18.613
035      52076      10626      19.549


All compounds were quantitated using method PAH18
except those which are labeled.
```

✓

PH 3/18/92

WESTON

STANDARD DATA

Fluorescence
Detection

METHOD NUMBER : PAH17
METHOD TITLE : PAH BY HPLC FLUORE
USER PROGRAMS : USER:MULTIPAH

ORDER OF FIT : 1
NUMBER OF LEVELS : 7
REPORT PARAMETERS :
NO.OF TIMES MODIFIED :
NO.OF TIMES CALIBRAT : 2

#	COMPONENT NAME	LEVEL A	LEVEL B	LEVEL C	LEVEL D	LEVEL E	LEVEL F	LEVEL G
1	PHENANTHRENE	0.3200	0.6400	1.2800	3.2000	6.4000	12.8000	32.0000
2	ANTHRACENE	0.3300	0.6600	1.3200	3.3000	6.6000	13.2000	33.0000
3	FLUORANTHRENE	0.1050	0.2100	0.4200	1.0500	2.1000	4.2000	10.5000
4	PYRENE	0.1350	0.2700	0.5400	1.3500	2.7000	5.4000	13.5000
5	BENZO(A)ANTHRACENE	0.0065	0.0130	0.0260	0.0650	0.1300	0.2600	0.6500
6	CHRYSENE	0.0750	0.1500	0.3000	0.7500	1.5000	3.0000	7.5000
7	BENZO(B)FLUORANTHREN	0.0090	0.0180	0.0360	0.0900	0.1800	0.3600	0.9000
8	BENZO(K)FLUORANTHREN	0.0085	0.0170	0.0340	0.0850	0.1700	0.3400	0.8500
9	BENZO(A)PYRENE	0.0115	0.0230	0.0460	0.1150	0.2300	0.4600	1.1500
10	DIBENZO(A,H)	0.0150	0.0300	0.0600	0.1500	0.3000	0.6000	1.5000
11	BENZO(GHI)	0.0380	0.0760	0.1520	0.3800	0.7600	1.5200	3.8000

```
MULTILEVEL CALIBRATION METHOD PAH17          03/17/92 13:51:32
1ST ORDER EXTERNAL STANDARD      CALIBRATION USING PEAK HEIGHT


TEST:

LEVEL     REPLICATE 1            REPLICATE 2        REPLICATE 3

  A       03179217.06
  B       03179217.05
  C       03179217.04
  D       03179217.03
  E
  F       03179217.01
  G

                                          COEFFICIENTS             SD OF    CORR
PEAK NAME                       a         b         c          d     FIT      COEFF

PHENANTHRENE                                  2.832E-05 1.628E-01  0.14606  0.99941
ANTHRACENE                                    5.713E-05 1.643E-01  0.14478  0.99946
FLUORANTHRENE                                 4.340E-05 5.221E-02  0.04929  0.99938
PYRENE                                        2.271E-05 8.119E-02  0.06954  0.99925
BENZO(A)ANTHRACENE                            1.130E-06 3.563E-03  0.00352  0.99917
CHRYSENE                                      1.035E-05 4.668E-02  0.04610  0.99894
BENZO(B)FLUORANTHREN                          1.018E-05 4.179E-03  0.00402  0.99943
BENZO(K)FLUORANTHREN                          2.303E-06 5.603E-04  0.00269  0.99971
BENZO(A)PYRENE                                1.434E-06-2.634E-03  0.00087  0.99998
DIBENZO(A,H)                                  2.674E-06-1.518E-04  0.00291  0.99989
BENZO(GHI)                                    3.291E-06 4.680E-03  0.01213  0.99971
```

STD PAH 20X

SAMPLE NO. : 03179217 . 01

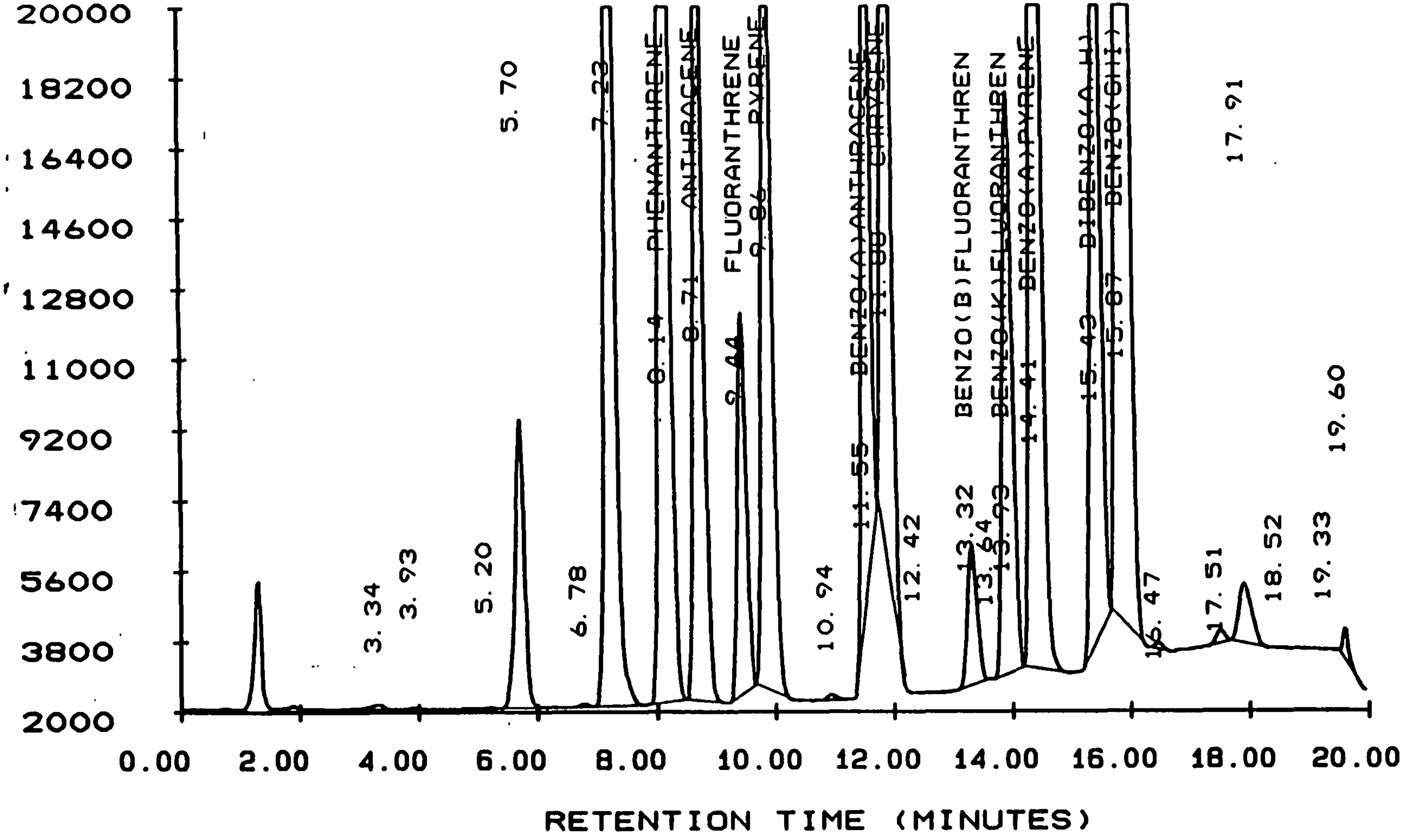
TEST NO. :

METHOD NO. : PAHF1 / PAHF1

INSTRUMENT: 17

DATE TIME: 03/17/92 10:50:44

PAGE NO. : 01



Y MAXIMUM: 20000.

Y MINIMUM: 2000.

START TIME: 0.00

END TIME: 20.00

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                         MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217    .01                          INST:17 VIAL:F0 SEQ NUMBER:001
TEST :                                 DATE-TIME INJECTED   : 03/17/92 10:50:44
COLLECTION TIME :   19.96              DATE-TIME PROCESSED  : 03/17/92 13:53:40
METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG          SAMP RATE:   0.78
CLIENT ID:                                                   SAMPLE VOL:
CLIENT:                                            COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: STD PAH 20X                                   RAW FILE: RAW2:CH242823
SAMPLE WT :                 % MOISTURE   :           DILUTION FACTOR :  1.0000

                                                                  HEIGHT
PK       PEAK      PEAK  BL    RT  GR COMPONENT                   CONC
NO       AREA    HEIGHT   MINUTES  #   NAME                       NG/UL
                          V   0.000
                          V   0.000
                          V   0.000
                          V   0.000
                          V   0.000
002     24896      1320   V   3.337
003      7098       463   V   3.933
004      4450       314   V   5.202
005    798090     72605   V   5.705
006      8330       916   V   6.780
007   3521115    327442   V   7.230
008   4616077    447781       8.139   PHENANTHRENE              12.844
009   2488231    228938       8.713   ANTHRACENE                13.244
010    993378     95904   V   9.435   FLUORANTHRENE              4.214
011   2657019    235014       9.861   PYRENE                     5.418
012     15638      1359   V  10.939
013   2563933    227871   T  11.552   BENZO(A)ANTHRACENE         0.261
014   3461655    286489   V  11.879   CHRYSENE                   3.012
015      3107       324   T  12.418
016    406064     35060   V  13.318   BENZO(B)FLUORANTHREN       0.361
017      1024       210   V  13.643
018   1722739    147216   V  13.928   BENZO(K)FLUORANTHREN       0.340
019   3956415    322708      14.407   BENZO(A)PYRENE             0.460
020   2587569    224774      15.433   DIBENZO(A,H)               0.601
021   5986277    461410   V  15.872   BENZO(GHI)                 1.523
022     16031      1599   V  16.472
023     34585      3197   V  17.508
024    233718     15149   V  17.914
025      3506       152   V  18.516
026       891       102   V  19.334
027     52346      7727      19.599


All compounds were quantitated using method PAH17
except those which are labeled.
```
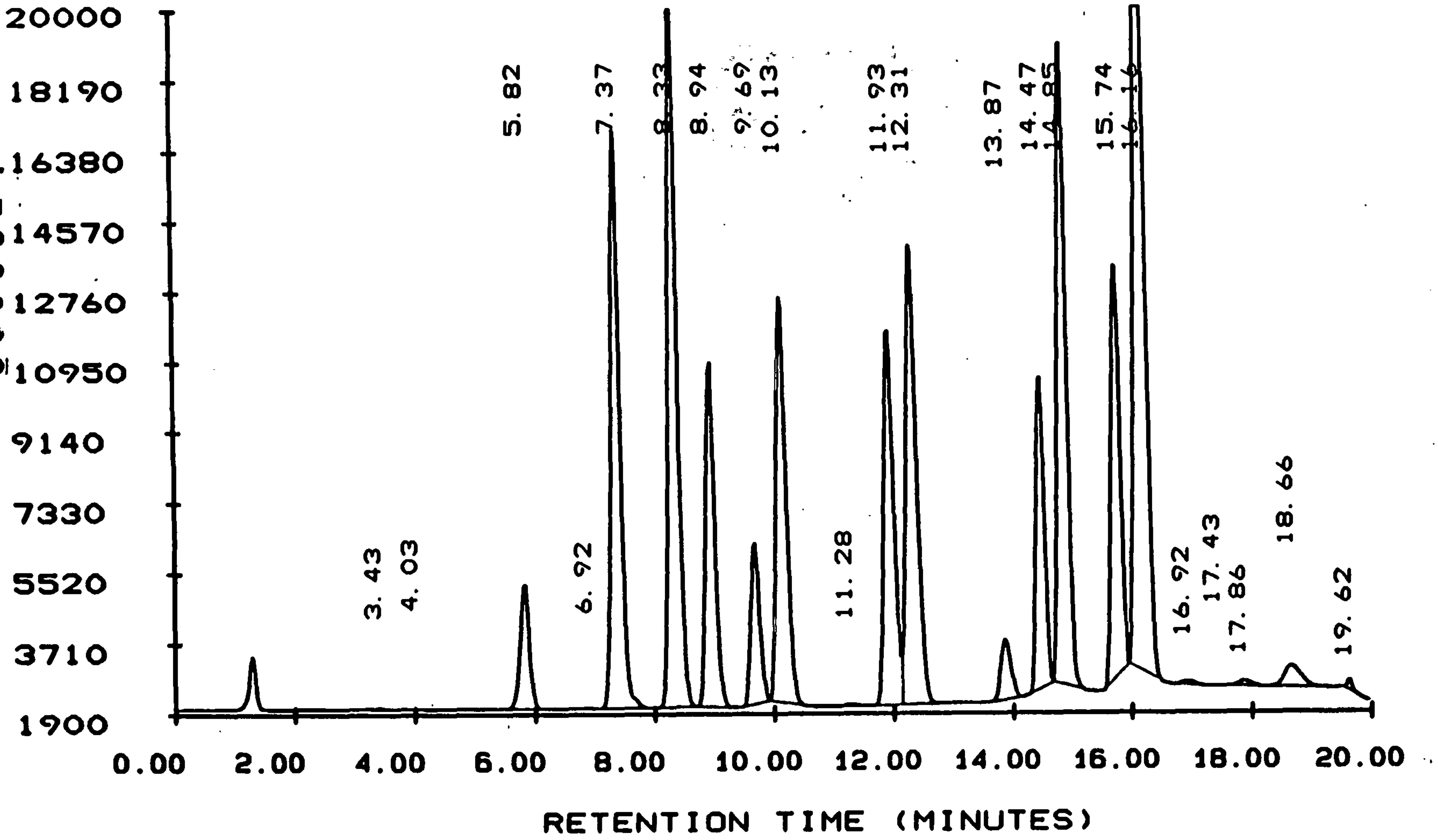

STD PAH 10X

SAMPLE NO. : 03179217 .07
TEST NO. :
METHOD NO. : PAHF1 / PAH17

INSTRUMENT: 17
DATE TIME: 03/17/92 13:16:42
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1900.

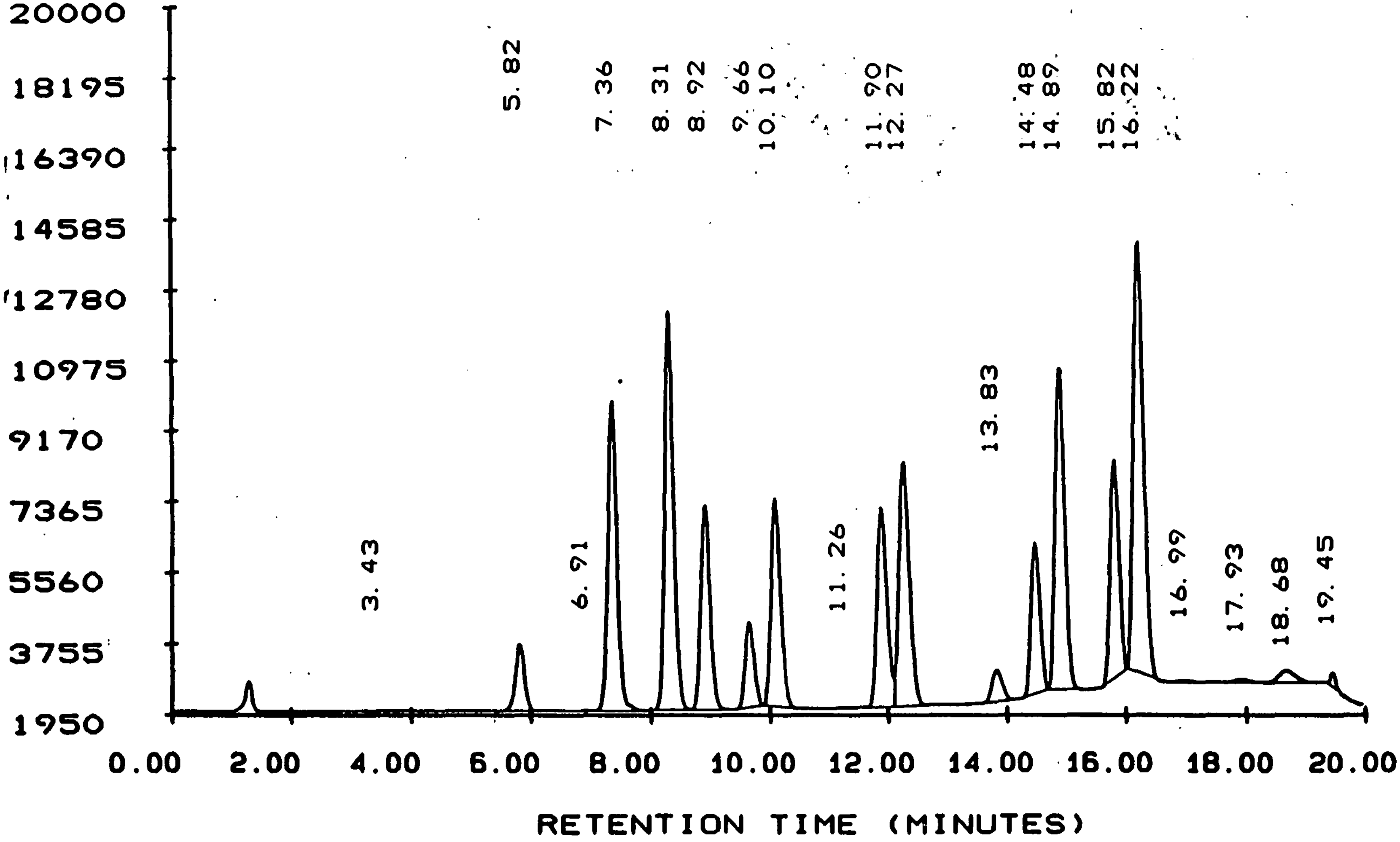
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                     03/17/92 13:55:16

                              MULTILEVEL EXTERNAL STANDARD

  SAMPLE: 03179217     .07                           INST:17 VIAL:F0 SEQ NUMBER:007
  TEST :                                   DATE-TIME INJECTED  : 03/17/92 13:16:42
  COLLECTION TIME :    19.96               DATE-TIME PROCESSED : 03/17/92 13:55:16
  METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG          SAMP RATE:    0.78
  CLIENT ID:                                                     SAMPLE VOL:
  CLIENT:                                          COLUMN TYPE: PAH BY HPLC FLUORESE
  LAB ID: STD PAH 10X                                  RAW FILE: RAW2:CH242915
  SAMPLE WT :                 % MOISTURE   :          DILUTION FACTOR :   1.0000

                          A                                        HEIGHT
  PK       PEAK         PEAK   BL    RT   GR COMPONENT                CONC
  NO       AREA        HEIGHT    MINUTES #   NAME                    NG/UL
  001       7198          359   V   3.428
  002       2803          160   V   4.030
  003     337062        31238   V   5.825
  004       2086          241   V   6.917
  005    1531483       146410   V   7.371
  006    1950363       189636   V   8.332   PHENANTHRENE             5.533
  007     936242        87351   V   8.943   ANTHRACENE               5.155
  008     425286        40496   V   9.690   FLUORANTHRENE            1.810
  009    1177581       102988   V  10.129   PYRENE                   2.420
  010       5883          457   V  11.285
  011    1120533        95609   T  11.928   BENZO(A)ANTHRACENE       0.112
  012    1484953       117159   V  12.306   CHRYSENE                 1.259
  013     181087        15179   V  13.867   BENZO(B)FLUORANTHREN     0.159
  014     776174        78846   V  14.466   BENZO(K)FLUORANTHREN     0.182
  015    1746137       163523   V  14.849   BENZO(A)PYRENE           0.232
  016    1110213       104548   V  15.745   DIBENZO(A,H)             0.279
  017    2695679       221827   V  16.156   BENZO(GHI)               0.735
  018      12674          934   V  16.920
  019       1032          108   V  17.427
  020      21001         1400   V  17.863
  021     111562         5451   V  18.664
  022      16422         2577      19.620


  All compounds were quantitated using method PAH17
  except those which are labeled.
```

STD PAH 5X

SAMPLE NO. : 03179217 .03
TEST NO. :
METHOD NO. : PAHF1 / PAH17

INSTRUMENT: 17
DATE TIME: 03/17/92 11:39:24
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1950.

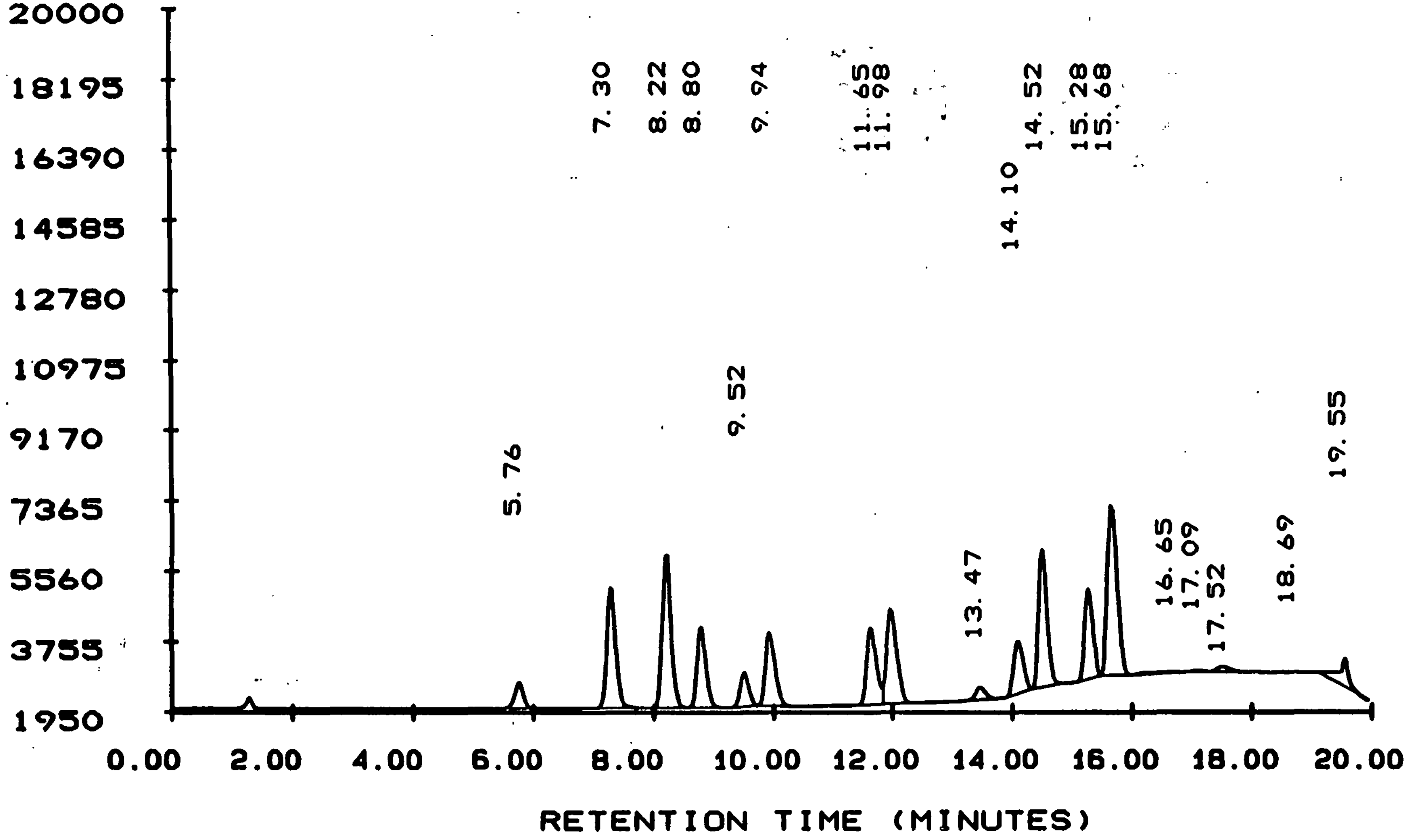
START TIME: 0.00
END TIME: 20.00

```
                    MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217     .03                     INST:17 VIAL:F0 SEQ NUMBER:003
TEST :                                 DATE-TIME INJECTED  : 03/17/92 11:39:24
COLLECTION TIME :   19.96              DATE-TIME PROCESSED : 03/17/92 13:54:17
METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG        SAMP RATE:   0.78
CLIENT ID:                                                   SAMPLE VOL:
CLIENT:                                           COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: STD PAH 5X                                  RAW FILE: RAW2:CH242860
SAMPLE WT :                 % MOISTURE  :         DILUTION FACTOR :  1.0000

                                                                 HEIGHT
PK       PEAK        PEAK   BL    RT   GR COMPONENT               CONC
NO       AREA      HEIGHT    MINUTES #   NAME                     NG/UL
001      3810         190   V   3.433
003    182035       16692   V   5.824
005      1420         139   V   6.915
006    834957       77736   V   7.364
007   1054631      100047   V   8.315    PHENANTHRENE             2.996
008    554838       51181   V   8.921    ANTHRACENE               3.088
009    226496       21360   V   9.662    FLUORANTHRENE            0.979
010    605286       52288   V  10.099    PYRENE                   1.269
011      2774         265   V  11.256
012    595810       49966   T  11.897    BENZO(A)ANTHRACENE       0.060
013    767034       61559   V  12.270    CHRYSENE                 0.684
014     96834        7860   V  13.828    BENZO(B)FLUORANTHREN     0.084
015    399617       37763   V  14.480    BENZO(K)FLUORANTHREN     0.088
016    923410       81332   V  14.893    BENZO(A)PYRENE           0.114
017    567194       54579   V  15.818    DIBENZO(A,H)             0.146
018   1327775      109445   V  16.216    BENZO(GHI)               0.365
019      6234         435   V  16.985
020     13848         800   V  17.925
021     60624        2940   V  18.682
022     21400        3489      19.448


All compounds were quantitated using method PAH17
except those which are labeled.
```

STD PAH 2X

SAMPLE NO. : 03179217 .04
TEST NO. :
METHOD NO. : PAHF1 / PAH17

INSTRUMENT: 17
DATE TIME: 03/17/92 12:03:42
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1950.

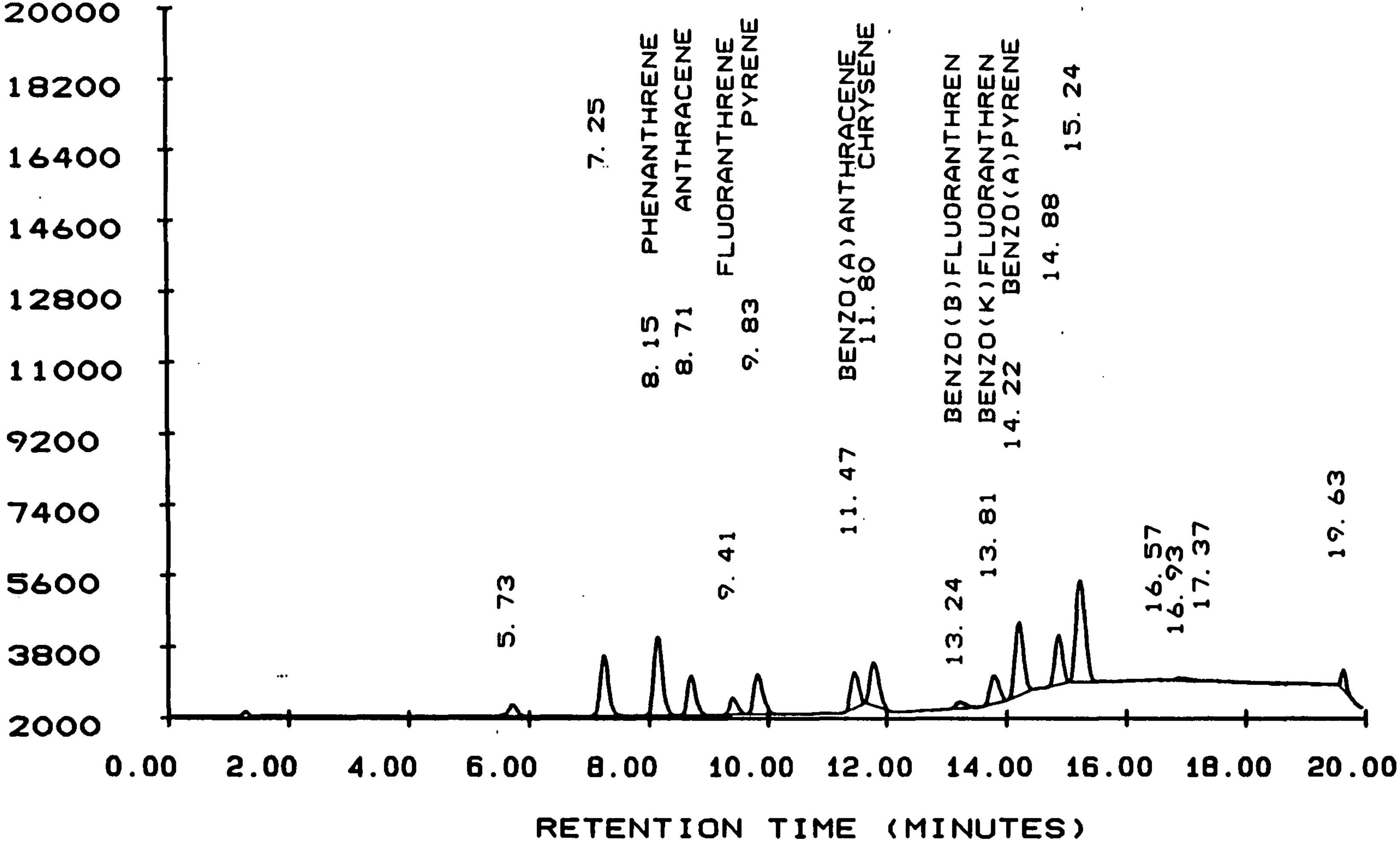
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                  03/17/92 13:54:32

                           MULTILEVEL EXTERNAL STANDARD

   SAMPLE: 03179217    .04                            INST:17 VIAL:FO SEQ NUMBER:004
   TEST :                                     DATE-TIME INJECTED   : 03/17/92 12:03:42
   COLLECTION TIME :   19.96                  DATE-TIME PROCESSED  : 03/17/92 13:54:32
   METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG           SAMP RATE:   0.78
   CLIENT ID:                                                      SAMPLE VOL:
   CLIENT:                                                   COLUMN TYPE: PAH BY HPLC FLUORESE
   LAB ID: STD PAH 2X                                           RAW FILE: RAW2:CH242873
   SAMPLE WT :                % MOISTURE  :                 DILUTION FACTOR :  1.0000

                                                                   HEIGHT
   PK       PEAK      PEAK   BL    RT   GR COMPONENT                 CONC
   NO       AREA     HEIGHT   MINUTES #   NAME                     NG/UL
                              T   0.000
                              V   0.000
                              V   0.000
   003     66219      6304   V   5.763
   004    318156     30141   V   7.295
   005    398524     38370   V   8.215   PHENANTHRENE              1.249
   006    211821     20104   V   8.795   ANTHRACENE                1.313
   007     87402      8324   V   9.515   FLUORANTHRENE             0.413
   008    209086     18371   V   9.938   PYRENE                    0.498
   009    223369     19283   V  11.646   BENZO(A)ANTHRACENE        0.025
   010    289401     23844      11.984   CHRYSENE                  0.293
   011     36712      3081   V  13.470   BENZO(B)FLUORANTHREN      0.036
   012    152506     13049   V  14.103   BENZO(K)FLUORANTHREN      0.031
   013    355543     34636      14.517   BENZO(A)PYRENE            0.047
   014    223392     22605   V  15.282   DIBENZO(A,H)              0.060
   015    496057     43068      15.675   BENZO(GHI)                0.146
   016     10273       209   V  16.650
   017      3919       288   V  17.087
   018     22515      1367   V  17.516
   019      2401       118   V  18.687
   020     93489      7136      19.551


   All compounds were quantitated using method PAH17
   except those which are labeled.
```

STD PAH 1X

SAMPLE NO. : 03179217 .05
TEST NO. :
METHOD NO. : PAHF1 / PAHF1

INSTRUMENT: 17
DATE TIME: 03/17/92 12:28:02
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

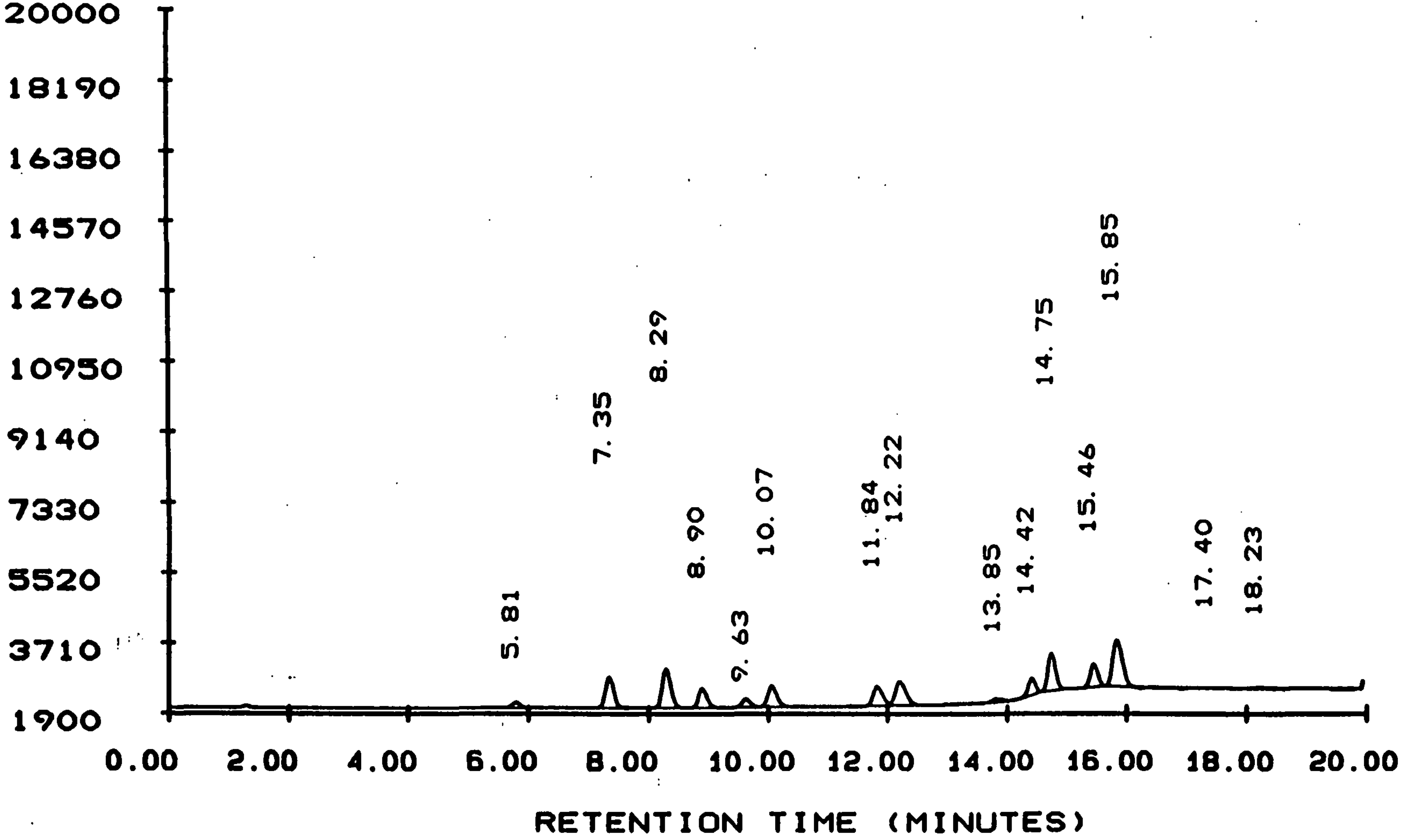
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                03/17/92 13:54:49

                         MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217    .05                         INST:17 VIAL:F0 SEQ NUMBER:005
TEST :                                  DATE-TIME INJECTED   : 03/17/92 12:28:02
COLLECTION TIME :   19.96               DATE-TIME PROCESSED : 03/17/92 13:54:49
METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG         SAMP RATE:   0.78
CLIENT ID:                                                  SAMPLE VOL:
CLIENT:                                               COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: STD PAH 1X                                          RAW FILE: RAW2:CH242884
SAMPLE WT :                % MOISTURE  :               DILUTION FACTOR :  1.0000

                                                                HEIGHT
PK       PEAK      PEAK   BL    RT   GR COMPONENT                 CONC
NO       AREA     HEIGHT   MINUTES #   NAME                     NG/UL
                           V   0.000
                           V   0.000
                           V   0.000
                           T   0.000
                           V   0.000
                           V   0.000
002     31900      3044    V   5.731
003    157563     15153    V   7.253
004    200134     19427        8.149   PHENANTHRENE            0.713
005    102616      9934        8.711   ANTHRACENE              0.732
006     43254      4245    V   9.414   FLUORANTHRENE           0.236
007    113097     10046        9.830   PYRENE                  0.309
008    114227     10099    V  11.473   BENZO(A)ANTHRACENE      0.015
009    151000     12368       11.795   CHRYSENE                0.175
010     18248      1544    V  13.241   BENZO(B)FLUORANTHREN    0.020
011     75475      6876    V  13.809   BENZO(K)FLUORANTHREN    0.016
012    179302     18013    V  14.218   BENZO(A)PYRENE          0.023
013    112410     12237       14.877   DIBENZO(A,H)            0.033
014    270618     25294       15.236   BENZO(GHI)              0.088
015      5553       222    V  16.568
016     11371       767    V  16.930
017      6758       139    V  17.365
018     30957      5211       19.635


All compounds were quantitated using method PAH17
except those which are labeled.
```

STD PAH 0.5X

SAMPLE NO. : 03179217 .06
TEST NO. :
METHOD NO. : PAHF1 / PAH17

INSTRUMENT: 17
DATE TIME: 03/17/92 12:52:23
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1900.

START TIME: 0.00
END TIME: 20.00

Roy F. Weston, Inc. - Lionville Laboratory 03/17/92 13:55:03

MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217 .06 INST:17 VIAL:F0 SEQ NUMBER:006
TEST : DATE-TIME INJECTED : 03/17/92 12:52:23
COLLECTION TIME : 19.96 DATE-TIME PROCESSED : 03/17/92 13:55:03
METHOD: PAHF1 / PAH17 REV #: 00061 ANALYST: HUANG SAMP RATE: 0.78
CLIENT ID: SAMPLE VOL:
CLIENT: COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: STD PAH 0.5X RAW FILE: RAW2:CH242901
SAMPLE WT : % MOISTURE : DILUTION FACTOR : 1.0000

PK NO	PEAK AREA	PEAK HEIGHT	BL	RT MINUTES	GR #	COMPONENT NAME	HEIGHT CONC NG/UL	
			V	0.000				
001	14990	1419	V	5.806				
002	78374	7666	V	7.347				
003	98701	9766	V	8.294		PHENANTHRENE	0.439	
004	49012	4733	V	8.896		ANTHRACENE	0.435	
005	20758	2057	V	9.634		FLUORANTHRENE	0.141	ug/mL
006	59943	5251	V	10.068		PYRENE	0.200	
007	58624	4956	T	11.841		BENZO(A)ANTHRACENE	0.009	.0065
008	76807	6092	V	12.218		CHRYSENE	0.110	.075
009	9053	783	V	13.851		BENZO(B)FLUORANTHREN	0.012	.009
010	39492	4241	V	14.421		BENZO(K)FLUORANTHREN	0.010	.008
011	92293	9597	V	14.751		BENZO(A)PYRENE	0.011	.0115
012	57154	5862		15.460		DIBENZO(A,H)	0.016	.015
013	138189	11776	V	15.848		BENZO(GHI)	0.043	.038
015	3663	145	V	17.403				
016	7329	337	V	18.225				

Indeno is on
UV detector .02 2ppb.

All compounds were quantitated using method PAH17
except those which are labeled.

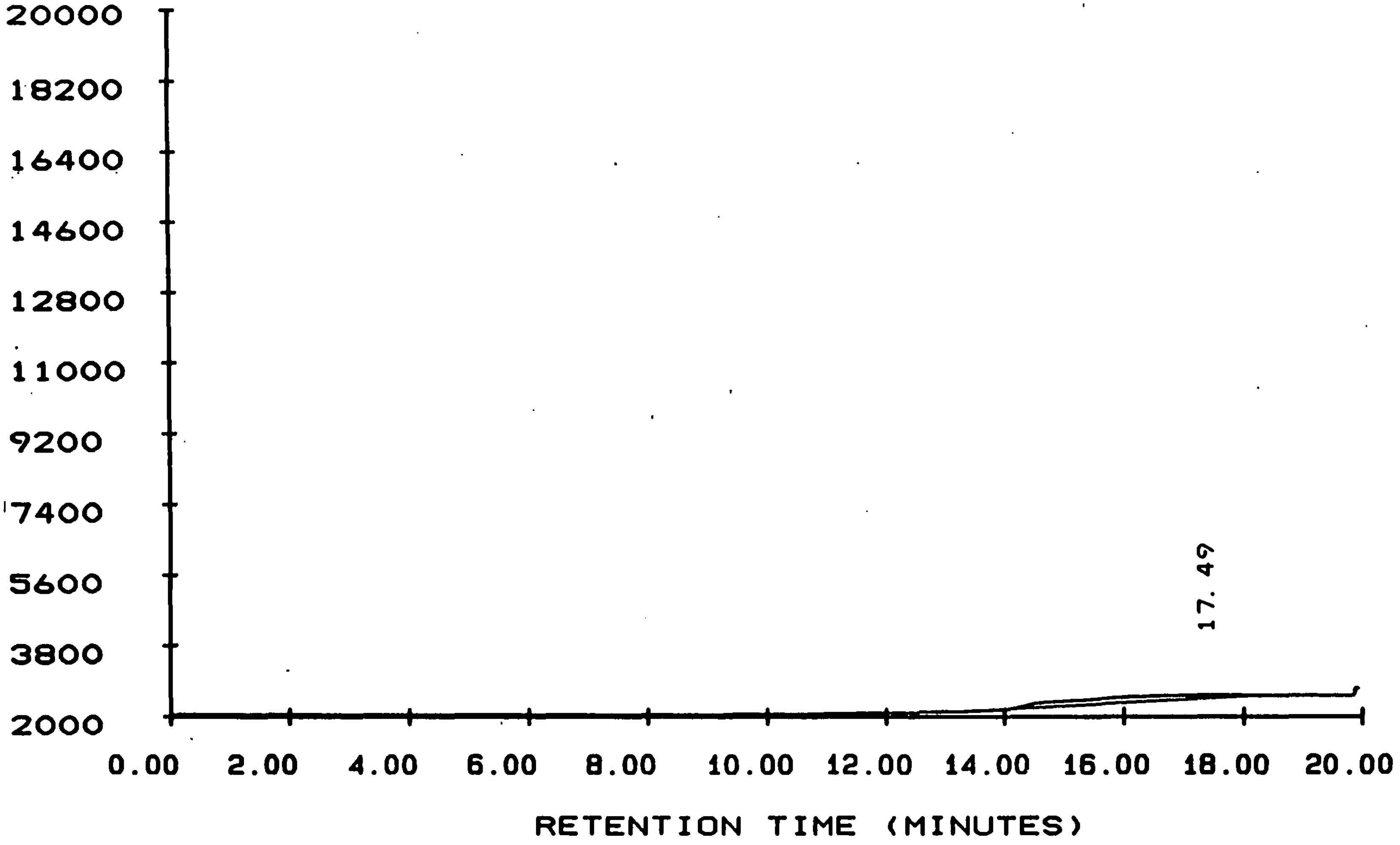
.01 ug/mL → 1ug/kg

.01 = 1ppb

OX

SAMPLE NO. : 03179217 . 08
TEST NO. :
METHOD NO. : PAHF1 / PAHF1

INSTRUMENT: 17
DATE TIME: 03/17/92 13:41:01
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

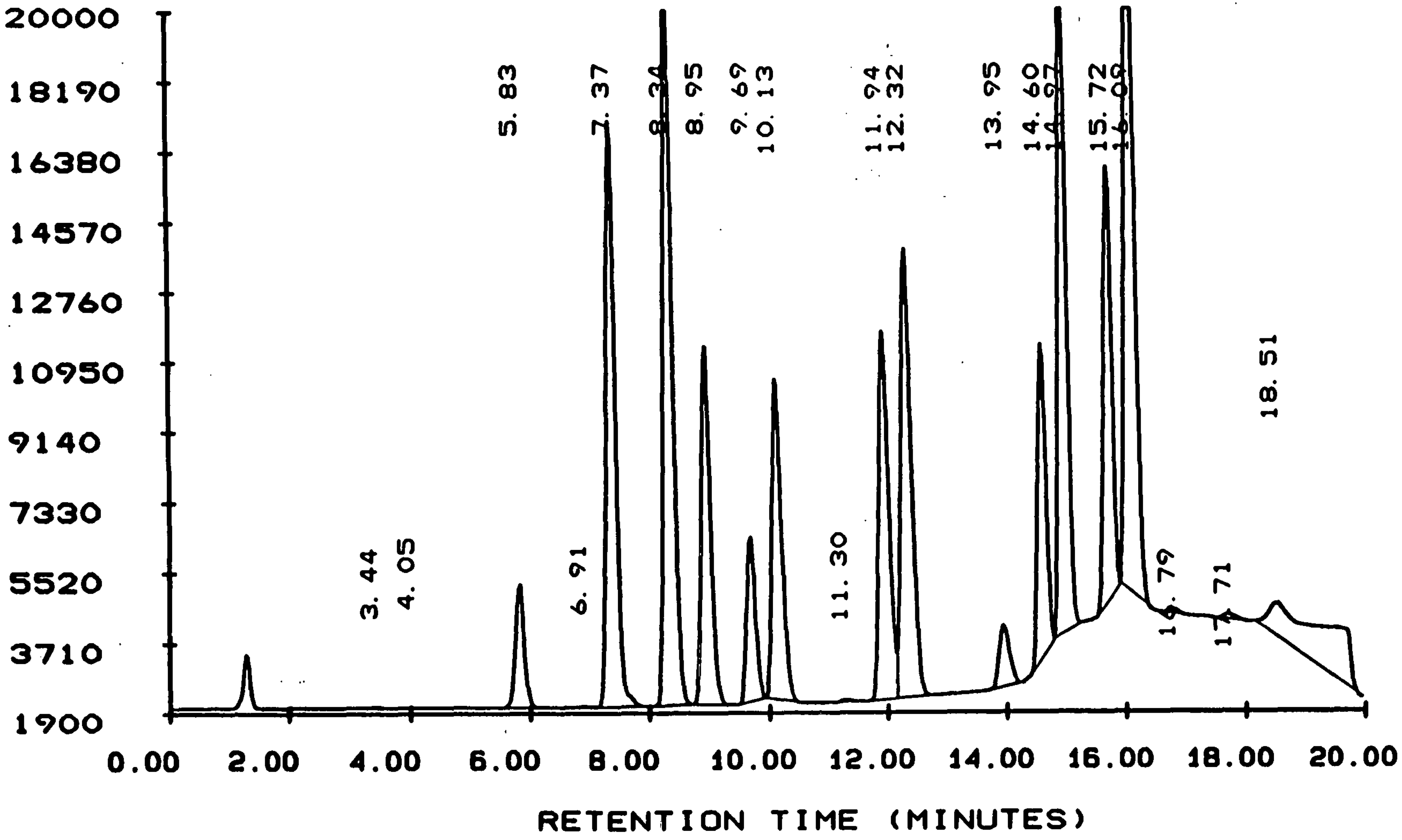
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory              03/17/92 14:39:44

                           MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217    .08                          INST:17 VIAL:F0 SEQ NUMBER:008
TEST :                                   DATE-TIME INJECTED  : 03/17/92 13:41:01
COLLECTION TIME :   19.96                DATE-TIME PROCESSED : 03/17/92 14:39:44
METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG          SAMP RATE:   0.78
CLIENT ID:                                                    SAMPLE VOL:
CLIENT:                                                  COLUMN TYPE: PAH BY HPLC FL
LAB ID: 0X                                                  RAW FILE: RAW2:CH242931
SAMPLE WT :                 % MOISTURE  :               DILUTION FACTOR :  1.0000

                                                                 HEIGHT
PK       PEAK      PEAK   BL    RT   GR COMPONENT                  CONC
NO       AREA     HEIGHT     MINUTES #   NAME                      NG/UL
================================================================================
                              8.330 M PHENANTHRENE
                              8.940 M ANTHRACENE
                              9.690 M FLUORANTHRENE
                             10.130 M PYRENE
                             11.930 M BENZO(A)ANTHRACENE
                             12.310 M CHRYSENE
                             13.870 M BENZO(B)FLUORANTHREN
                             14.470 M BENZO(K)FLUORANTHREN
                             14.850 M BENZO(A)PYRENE
                             15.740 M DIBENZO(A,H)
                             16.160 M BENZO(GHI)
003    238554      726   V   17.491
```

STD PAH 10X

SAMPLE NO. : 03179217 .18
TEST NO. :
METHOD NO. : PAHF1 / PAH17

INSTRUMENT: 17
DATE TIME: 03/17/92 17:55:17
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1900.

START TIME: 0.00
END TIME: 20.00

Roy F. Weston, Inc. - Lionville Laboratory 03/18/92 13:19:56

MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217 .18 INST:17 VIAL:F0 SEQ NUMBER:018
TEST : DATE-TIME INJECTED : 03/17/92 17:55:17
COLLECTION TIME : 19.96 DATE-TIME PROCESSED : 03/18/92 13:19:56
METHOD: PAHF1 / PAH17 REV #: 00062 ANALYST: HUANG SAMP RATE: 0.78
CLIENT ID: SAMPLE VOL:
CLIENT: COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: STD PAH 10X RAW FILE: RAW2:CH243141
SAMPLE WT : % MOISTURE : DILUTION FACTOR : 1.0000

PK NO	PEAK AREA	PEAK HEIGHT	BL	RT MINUTES	GR #	COMPONENT NAME	HEIGHT CONC NG/UL	Rec %
001	7561	388	V	3.445				
002	1996	135	V	4.052				
003	331016	31252	V	5.829				
004	2356	279	V	6.914				
005	1542465	148770	V	7.374				
006	1928841	188987	V	8.336		PHENANTHRENE	5.515	86.2
007	971648	91190	V	8.946		ANTHRACENE	5.374	81.4
008	436429	41731	V	9.692		FLUORANTHRENE	1.863	88.7
009	940731	81591	V	10.131		PYRENE	1.934	71.6
010	5722	501	V	11.298				
011	1099723	93970	T	11.942		BENZO(A)ANTHRACENE	0.110	85.6
012	1416383	114738	V	12.322		CHRYSENE	1.234	82.3
013	195896	15654	V	13.950		BENZO(B)FLUORANTHREN	0.164	91.1
014	815674	81255	T	14.604		BENZO(K)FLUORANTHREN	0.188	110.6
015	1705753	172562	V	14.965		BENZO(A)PYRENE	0.245	106.5
016	1064163	110124	V	15.718		DIBENZO(A,H)	0.294	98.0
017	2241035	195921	V	16.090		BENZO(GHI)	0.649	85.4
018	10581	967	V	16.789				
019	17028	1256	V	17.713				
020	837220	8686		18.511				

All compounds were quantitated using method PAH17
except those which are labeled.

3/18/92

UV
Detect

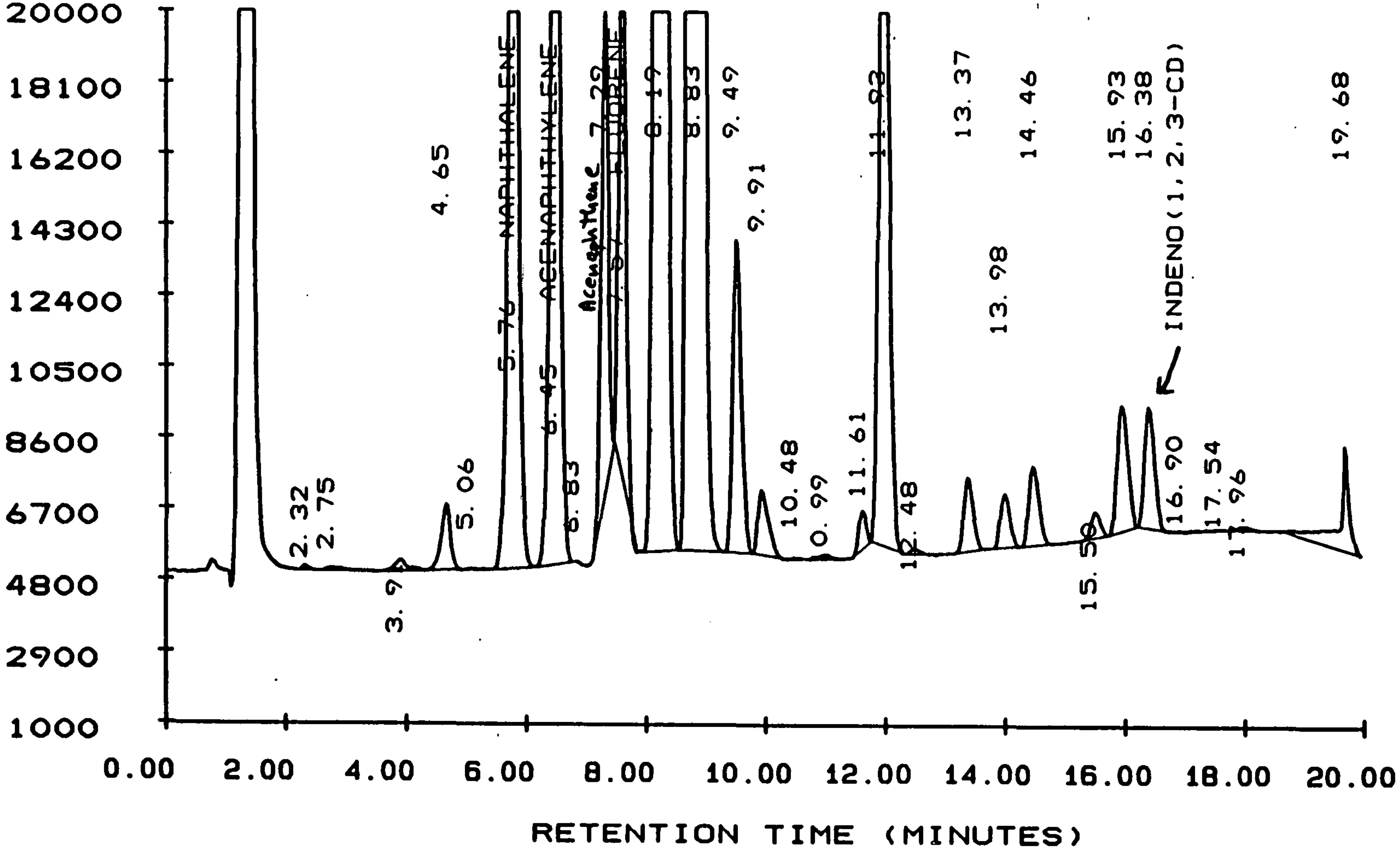
```
METHOD NUMBER                                   : PAH18
METHOD TITLE                                    : PAH'S BY HPLC UV D
USER PROGRAMS                                   : USER:MULTITST

ORDER OF FIT                                    : 1
NUMBER OF LEVELS                                : 7
REPORT PARAMETERS                               :
NO.OF TIMES MODIFIED                            :
NO.OF TIMES CALIBRAT                            : 3


# COMPONENT NAME        LEVEL A    LEVEL B    LEVEL C    LEVEL D    LEVEL E
                        LEVEL F    LEVEL G

1 NAPHTHALENE          0.9000     1.8000     3.6000     9.0000     18.0000
                       36.0000    90.0000

2 ACENAPHTHYLENE       1.1500     2.3000     4.6000     11.5000    23.0000
                       46.0000    115.0000

3 ACENAPHTHENE         0.9000     1.8000     3.6000     9.0000     18.0000
                       36.0000    90.0000

4 FLUORENE             0.1050     0.2100     0.4200     1.0500     2.1000
                       4.2000     10.5000

5 INDENO(1,2,3-CD)     0.0215     0.0430     0.0860     0.2150     0.4300
                       0.8600     2.1500
```

```
MULTILEVEL CALIBRATION METHOD PAH18          03/17/92 13:52:39
1ST ORDER EXTERNAL STANDARD      CALIBRATION USING PEAK HEIGHT


TEST:

LEVEL     REPLICATE 1            REPLICATE 2        REPLICATE 3

  A       03179218.06
  B       03179218.05
  C       03179218.04
  D       03179218.03
  E       03179218.07
  F       03179218.01
  G

                                     COEFFICIENTS          SD OF    CORR
PEAK NAME                 a         b          c          d       FIT     COEFF

NAPHTHALENE                              1.168E-04-1.172E-01  0.21049  0.99980
ACENAPHTHYLENE                           1.536E-04-3.065E-02  0.05151  0.99999
ACENAPHTHENE                             2.860E-04-1.766E-01  0.40443  0.99928
FLUORENE                                 2.865E-05-2.235E-02  0.06497  0.99864
INDENO(1,2,3-CD)                         2.692E-05 5.108E-03  0.01333  0.99864
```

STD PAH 20X

SAMPLE NO. : 03179218 . 01
TEST NO. :
METHOD NO. : PAHU1 / PAHU1

INSTRUMENT: 18
DATE TIME: 03/17/92 10:50:44
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

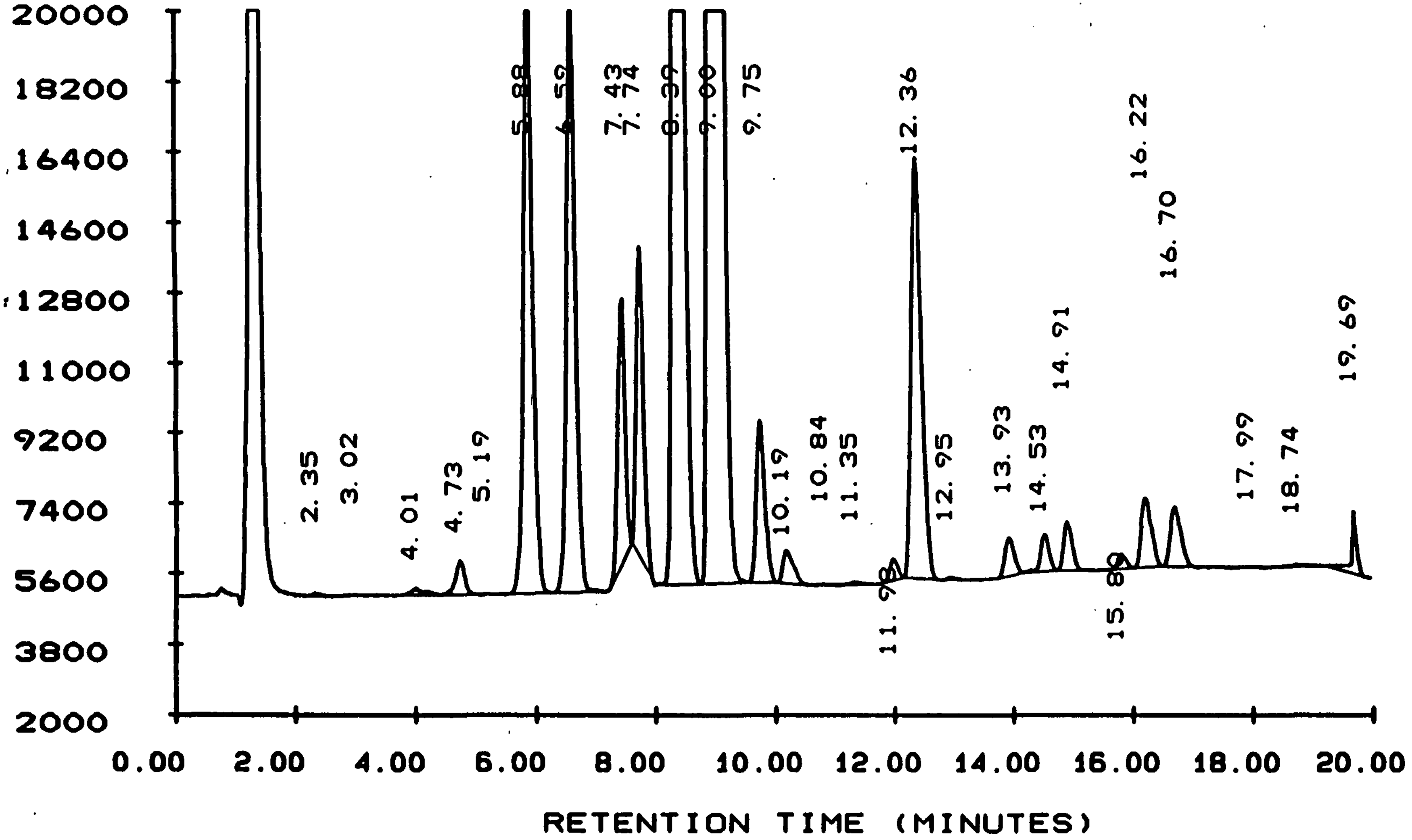
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory             03/17/92 13:56:11

                          MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179218    .01                      INST:18 VIAL:F0 SEQ NUMBER:001
TEST :                             DATE-TIME INJECTED  : 03/17/92 10:50:44
COLLECTION TIME :   19.96          DATE-TIME PROCESSED : 03/17/92 13:56:11
METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG        SAMP RATE:   0.78
CLIENT ID:                                                SAMPLE VOL:
CLIENT:                                        COLUMN TYPE: PAH'S BY HPLC UV DET
LAB ID: STD PAH 20X                                 RAW FILE: RAW2:CH242824
SAMPLE WT :                 % MOISTURE  :         DILUTION FACTOR :  1.0000

                                                               HEIGHT
PK       PEAK      PEAK  BL   RT   GR COMPONENT                 CONC
NO       AREA     HEIGHT  MINUTES  #   NAME                     NG/UL
                          V   0.000
                          V   0.000
                          V   0.000
001      7464       1082  V   2.321
002     19854        855  V   2.752
003     46515       3060  V   3.906
004    190521      17291  V   4.648
005      4151        294  V   5.061
006   3230419     307564      5.762  NAPHTHALENE              35.806
007   2994239     299530      6.455  ACENAPHTHYLENE           45.977
008      1565        368  V   6.826
009   1058785     125270  V   7.287  ACENAPHTHENE             35.651
010   1243281     145491      7.572  FLUORENE                  4.146
011   1836301     045159  V   8.192
012   7282672     048657  V   8.832
013    842688      82324  V   9.491
014    213745      16942  V   9.915
015      2373        253  V  10.484
016     14566       1175  V  10.990
017     87805       9784  V  11.607
018   2418187     213980  V  11.934
019     15255       1360  V  12.485
020    220572      19379  V  13.374
021    158816      14149  V  13.985
022    250458      20966  V  14.463
023     75222       6771  V  15.497
024    407514      33327  V  15.935
025    378259      32116  V  16.377  INDENO(1,2,3-CD)          0.870
026      1030        108  V  16.901
027      6146        198  V  17.545
028     15552        984  V  17.963
030    337062      24382     19.676


All compounds were quantitated using method PAH18
except those which are labeled.
```

STD PAH 10X

SAMPLE NO. : 03179218 .07
TEST NO. :
METHOD NO. : PAHU1 / PAH18

INSTRUMENT: 18
DATE TIME: 03/17/92 13:16:42
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

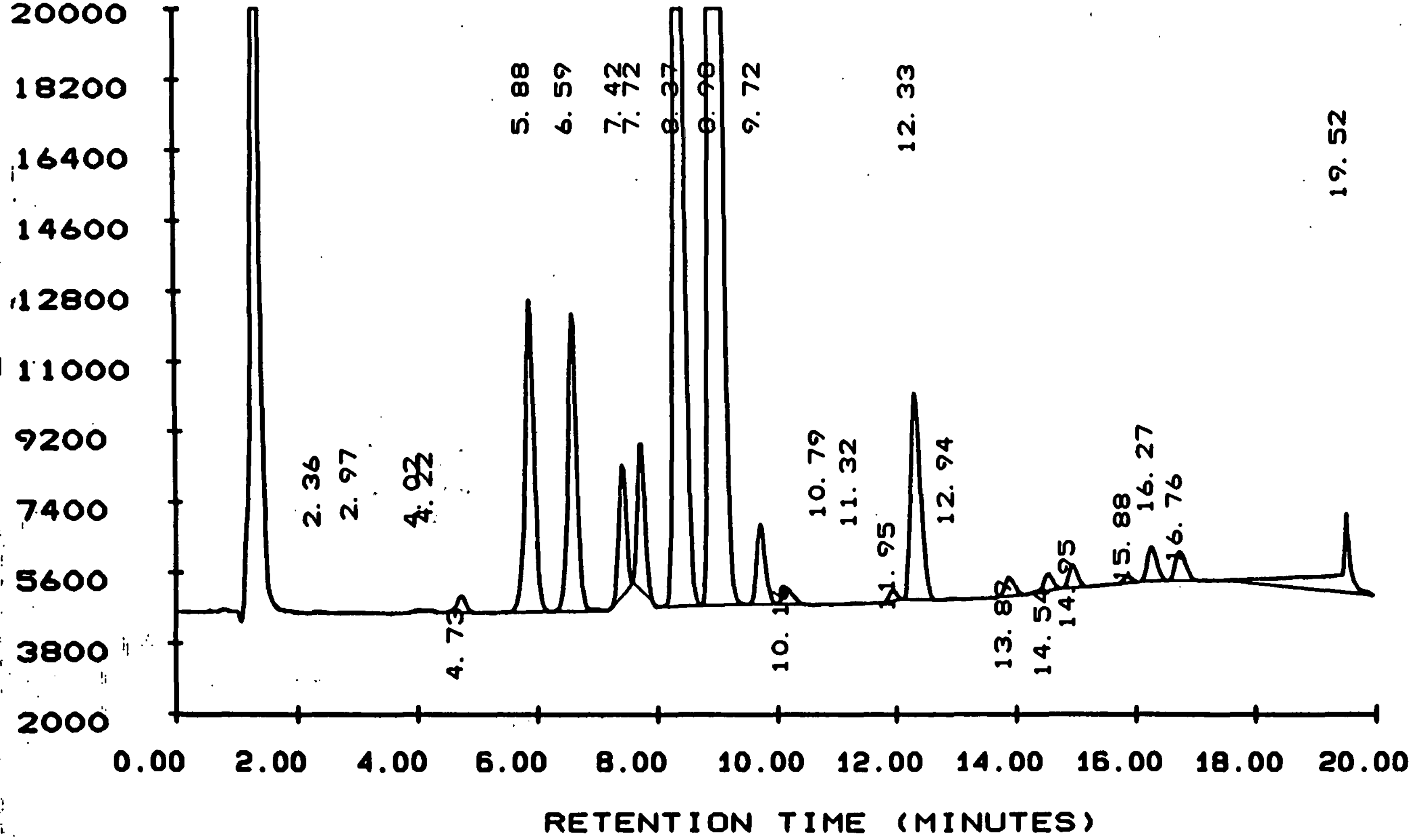
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                03/17/92 13:57:42

                          MULTILEVEL EXTERNAL STANDARD

  SAMPLE: 03179218    .07                         INST:18 VIAL:F0 SEQ NUMBER:007
  TEST :                                  DATE-TIME INJECTED  : 03/17/92 13:16:42
  COLLECTION TIME :    19.96              DATE-TIME PROCESSED : 03/17/92 13:57:42
  METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG          SAMP RATE:   0.78
  CLIENT ID:                                                   SAMPLE VOL:
  CLIENT:                                          COLUMN TYPE: PAH'S BY HPLC UV DET
  LAB ID: STD PAH 10X                                   RAW FILE: RAW2:CH242914
  SAMPLE WT :                % MOISTURE  :              DILUTION FACTOR :  1.0000

                                                                   HEIGHT
  PK       PEAK      PEAK   BL    RT   GR COMPONENT                  CONC
  NO       AREA     HEIGHT    MINUTES #   NAME                      NG/UL
  001      6132       672   V   2.347
  002      3635       193   V   3.015
  004     28439      1631   V   4.010
  005     93802      8627   V   4.730
  006       954       132   V   5.189
  007   1633369    158128   V   5.882    NAPHTHALENE              18.352
  008   1519405    150498   V   6.594    ACENAPHTHYLENE           23.086
  009    577465     66003   V   7.428    ACENAPHTHENE             18.700
  010    675424     78072   V   7.736    FLUORENE                  2.214
  011   6376769    641228   V   8.388
  012   3447835    048293   V   9.003
  013    421371     40816   V   9.746
  014    108684      8502   V  10.187
  015      3316       191   V  10.844
  016      8749       665   V  11.345
  017     53747      5480   V  11.985
  018   1303113    107062   V  12.364
  019      7217       670   V  12.949
  020    106819      9599   V  13.925
  021     92056      9105   V  14.526
  022    123264     12228   V  14.908
  023     38374      3727   V  15.804
  024    210692     17617   V  16.217
  025    191945     15099   V  16.703    INDENO(1,2,3-CD)          0.412
  027      2637       144   V  17.987
  028     12914       486   V  18.740
  029     98909     12427      19.689


  All compounds were quantitated using method PAH18
  except those which are labeled.
```

STD PAH 5X

SAMPLE NO. : 03179218 .03
TEST NO. :
METHOD NO. : PAHU1 / PAH18

INSTRUMENT: 18
DATE TIME: 03/17/92 11:39:24
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

START TIME: 0.00
END TIME: 20.00

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    Roy F. Weston, Inc. - Lionville Laboratory               03/17/92 13:56:43

                            MULTILEVEL EXTERNAL STANDARD

  SAMPLE: 03179218     .03                          INST:18 VIAL:F0 SEQ NUMBER:003
  TEST :                                   DATE-TIME INJECTED   : 03/17/92 11:39:24
  COLLECTION TIME :    19.96               DATE-TIME PROCESSED  : 03/17/92 13:56:43
  METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG          SAMP RATE:   0.78
  CLIENT ID:                                                     SAMPLE VOL:
  CLIENT:                                               COLUMN TYPE: PAH'S BY HPLC UV DET
  LAB ID: STD PAH 5X                                         RAW FILE: RAW2:CH242859
  SAMPLE WT :                 % MOISTURE  :                 DILUTION FACTOR :  1.0000

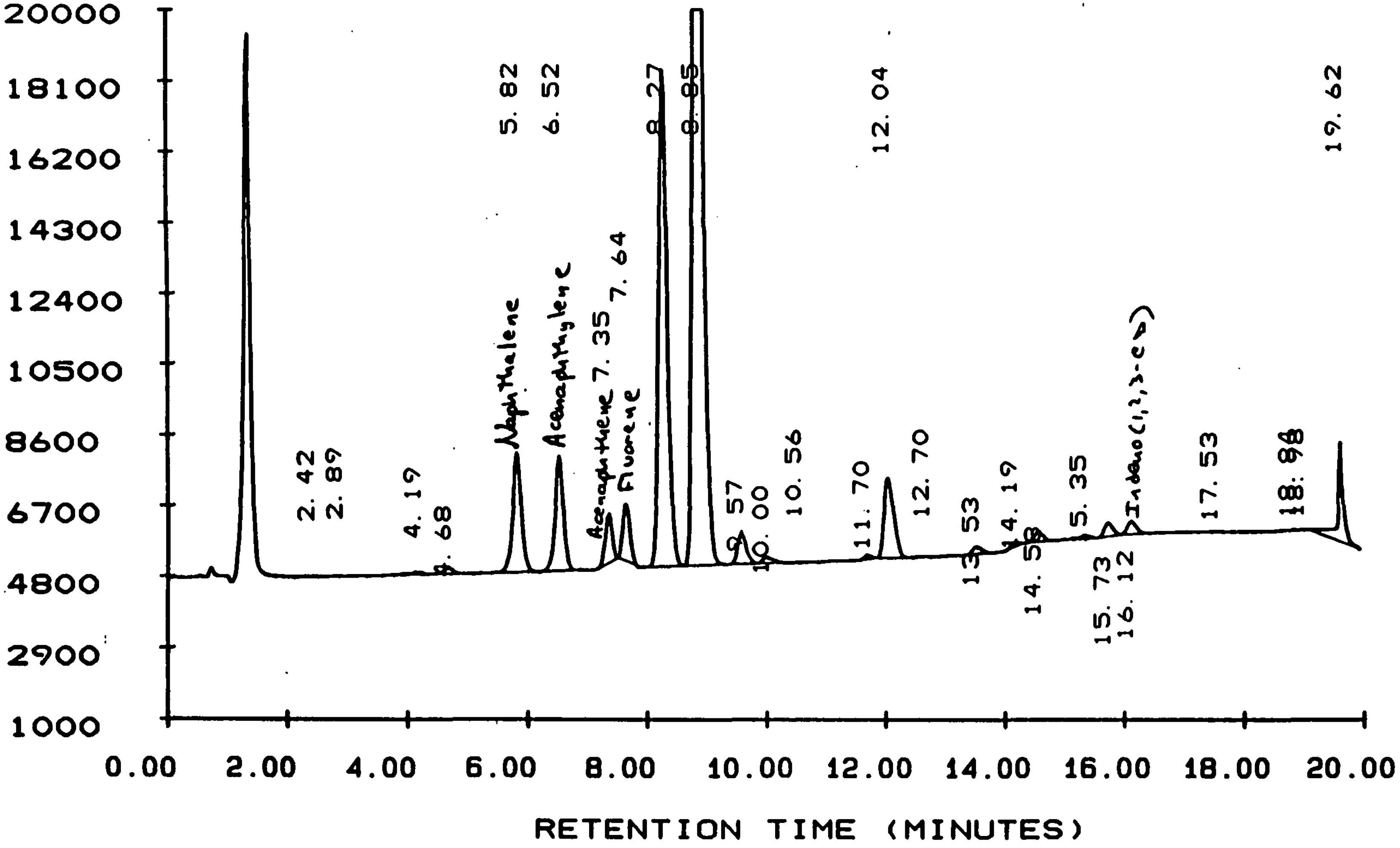
                                                                  HEIGHT
  PK       PEAK      PEAK   BL    RT   GR COMPONENT                CONC
  NO       AREA     HEIGHT   MINUTES #   NAME                     NG/UL
  001      3837       373   V   2.361
  002      6355       197   V   2.974
  003      3703       398   V   4.024
  004      1723       284   V   4.218
  005     46895      4276   V   4.730
  006    821358     78272   V   5.881   NAPHTHALENE                9.025
  007    765553     74728   V   6.587   ACENAPHTHYLENE            11.448
  008    281845     32086   V   7.420   ACENAPHTHENE               9.000
  009    320972     36911   V   7.724   FLUORENE                   1.035
  010   3213697    318261   V   8.372
  011   8002587    762621   V   8.976
  012    209537     20129   V   9.719
  013     54400      4131   V  10.157
  014      1718       191   V  10.792
  015      5745       422   V  11.318
  016     25559      2566   V  11.955
  017    625278     52252   V  12.326
  018      3621       365   V  12.939
  019     54967      4659   V  13.887
  020     47874      4229   V  14.541
  021     61696      5694   V  14.954
  022     18092      1829   V  15.882
  023    101146      8635   V  16.274
  024     94061      7377   V  16.755   INDENO(1,2,3-CD)           0.204
  026    395790     16670      19.516


  All compounds were quantitated using method PAH18
  except those which are labeled.
```

STD PAH 2X

SAMPLE NO. : 03179218 .04
TEST NO. :
METHOD NO. : PAHU1 / PAHU1

INSTRUMENT: 18
DATE TIME: 03/17/92 12:03:42
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                    03/17/92 13:57:01

                          MULTILEVEL EXTERNAL STANDARD

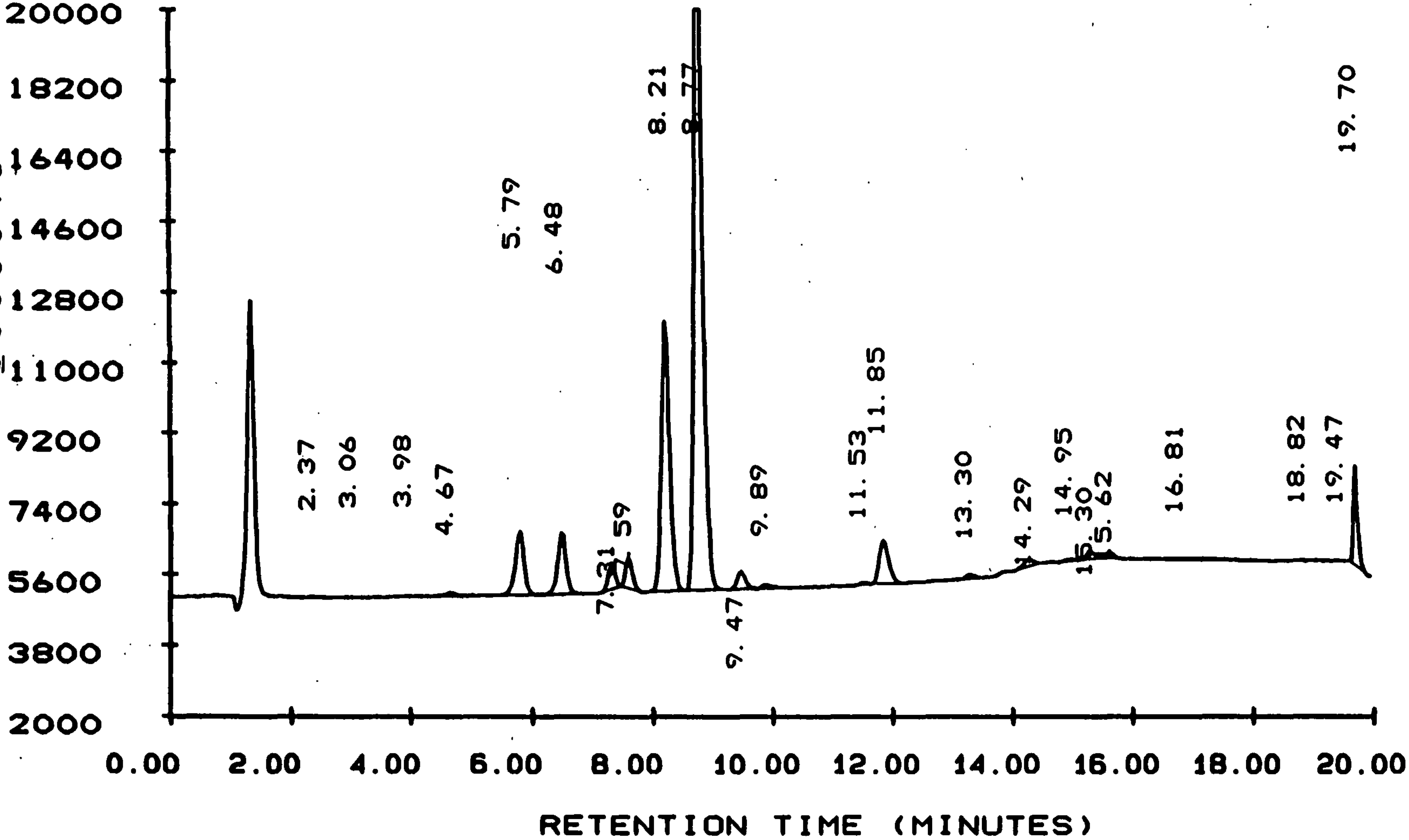
SAMPLE: 03179218     .04                          INST:18 VIAL:F0 SEQ NUMBER:004
TEST :                                   DATE-TIME INJECTED   : 03/17/92 12:03:42
COLLECTION TIME :    19.96               DATE-TIME PROCESSED  : 03/17/92 13:57:01
METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG        SAMP RATE:    0.78
CLIENT ID:                                                  SAMPLE VOL:
CLIENT:                                               COLUMN TYPE: PAH'S BY HPLC UV DET
LAB ID: STD PAH 2X                                       RAW FILE: RAW2:CH242874
SAMPLE WT :                % MOISTURE   :                DILUTION FACTOR :  1.0000

                                                                 HEIGHT
PK        PEAK       PEAK   BL     RT   GR COMPONENT                CONC
NO        AREA     HEIGHT    MINUTES #   NAME                     NG/UL
                             V    0.000
001       3176        205    V    2.417
002       1170        134    V    2.893
003      10713        654    V    4.191
004      18606       1686    V    4.683
005     332336      31413    V    5.820   NAPHTHALENE              3.552
006     302598      30128    V    6.521   ACENAPHTHYLENE           4.597
007     108813      12789    V    7.352   ACENAPHTHENE             3.481
008     126808      14947    V    7.643   FLUORENE                 0.406
009    1298523     130780    V    8.271
010    3311425     323204    V    8.850
011      85018       8252    V    9.571
012      20087       1638    V    9.996
013       1037        127    V   10.558
014       8083        966    V   11.702
015     244303      21282    V   12.041
016       2271        127    V   12.701
017      22882       1952    V   13.529
018      11674        966    V   14.193
019      27398       2742    V   14.578
020       9686        831    V   15.349
021      38163       3540    V   15.735
022      38201       3379        16.118   INDENO(1,2,3-CD)         0.096
023       9003        209    V   17.534
024       1882        130    V   18.836
025        678        115    V   18.981
026     177540      21298        19.621


All compounds were quantitated using method PAH18
except those which are labeled.
```

STD PAH 1X

SAMPLE NO. : 03179218 .05
INSTRUMENT: 18
TEST NO. :
DATE TIME: 03/17/92 12:28:02
METHOD NO. : PAHU1 / PAH18
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                03/17/92 13:57:14

                              MULTILEVEL EXTERNAL STANDARD

   SAMPLE: 03179218    .05                         INST:18 VIAL:F0 SEQ NUMBER:005
   TEST :                                 DATE-TIME INJECTED   : 03/17/92 12:28:02
   COLLECTION TIME :   19.96              DATE-TIME PROCESSED  : 03/17/92 13:57:14
   METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG          SAMP RATE:   0.78
   CLIENT ID:                                                     SAMPLE VOL:
   CLIENT:                                                  COLUMN TYPE: PAH'S BY HPLC UV DET
   LAB ID: STD PAH 1X                                       RAW FILE: RAW2:CH242885
   SAMPLE WT :                   % MOISTURE   :             DILUTION FACTOR :  1.0000

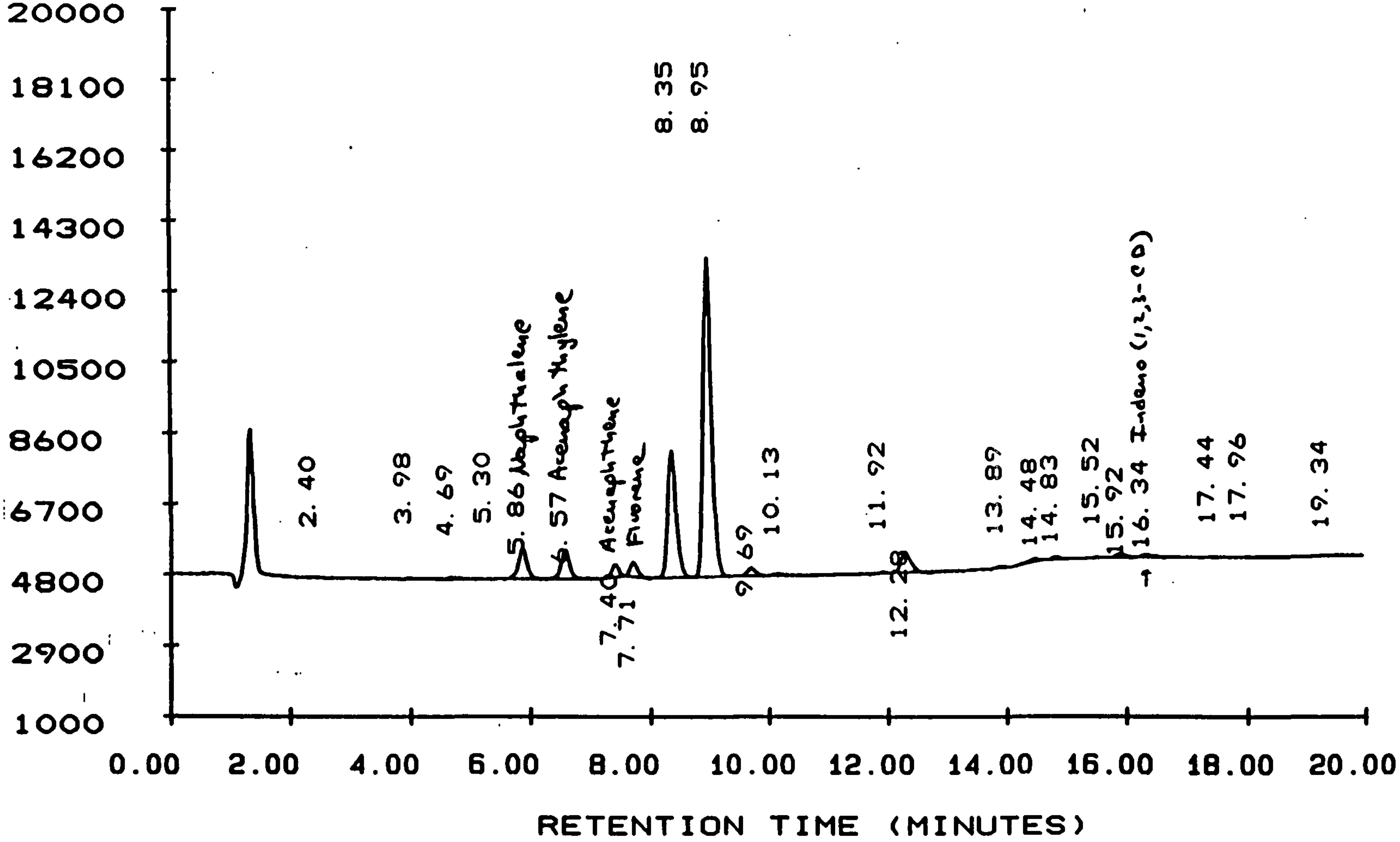
                                                                  HEIGHT
   PK        PEAK       PEAK    BL    RT    GR COMPONENT            CONC
   NO        AREA     HEIGHT    MINUTES  #   NAME                 NG/UL
   001       3659        268   V    2.374
   002       5104        178   V    3.061
   003       2458        161   V    3.975
   005      10748        885   V    4.666
   006     167984      15805   V    5.787    NAPHTHALENE           1.729
   007     153116      15183   V    6.482    ACENAPHTHYLENE        2.301
   008      54627       6475   V    7.311    ACENAPHTHENE          1.675
   009      64372       7522   V    7.590    FLUORENE              0.193
   010     662475      67399   V    8.206
   011    1703721     168387   V    8.766
   012      44820       4266   V    9.472
   013      11385        909   V    9.886
   014       4775        506   V   11.534
   015     127513      11010   V   11.853
   016      10445       1031   V   13.300
   017      19532       1717   V   14.286
   018       4406        452   V   14.953
   019      19650       1957   V   15.298
   020      15142       1548   V   15.619    INDENO(1,2,3-CD)      0.047
   021      26836        355   V   16.808
   022       2383        128   V   18.823
   024       1186        126   V   19.467
   025     115418      20622       19.704


   All compounds were quantitated using method PAH18
   except those which are labeled.
```

STD PAH 0. 5X

SAMPLE NO. : 03179218 . 06
TEST NO. :
METHOD NO. : PAHU1 / PAHU1

INSTRUMENT: 18
DATE TIME: 03/17/92 12: 52: 23
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0. 00
END TIME: 20. 00

```
Roy F. Weston, Inc. - Lionville Laboratory                      03/17/92 13:57:29

                          MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179218    .06                           INST:18 VIAL:F0 SEQ NUMBER:006
TEST :                                    DATE-TIME INJECTED  : 03/17/92 12:52:23
COLLECTION TIME :   19.96                 DATE-TIME PROCESSED : 03/17/92 13:57:29
METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG          SAMP RATE:    0.78
CLIENT ID:                                                     SAMPLE VOL:
CLIENT:                                             COLUMN TYPE: PAH'S BY HPLC UV DET
LAB ID: STD PAH 0.5X                                       RAW FILE: RAW2:CH242900
SAMPLE WT :                 % MOISTURE  :                DILUTION FACTOR :  1.0000

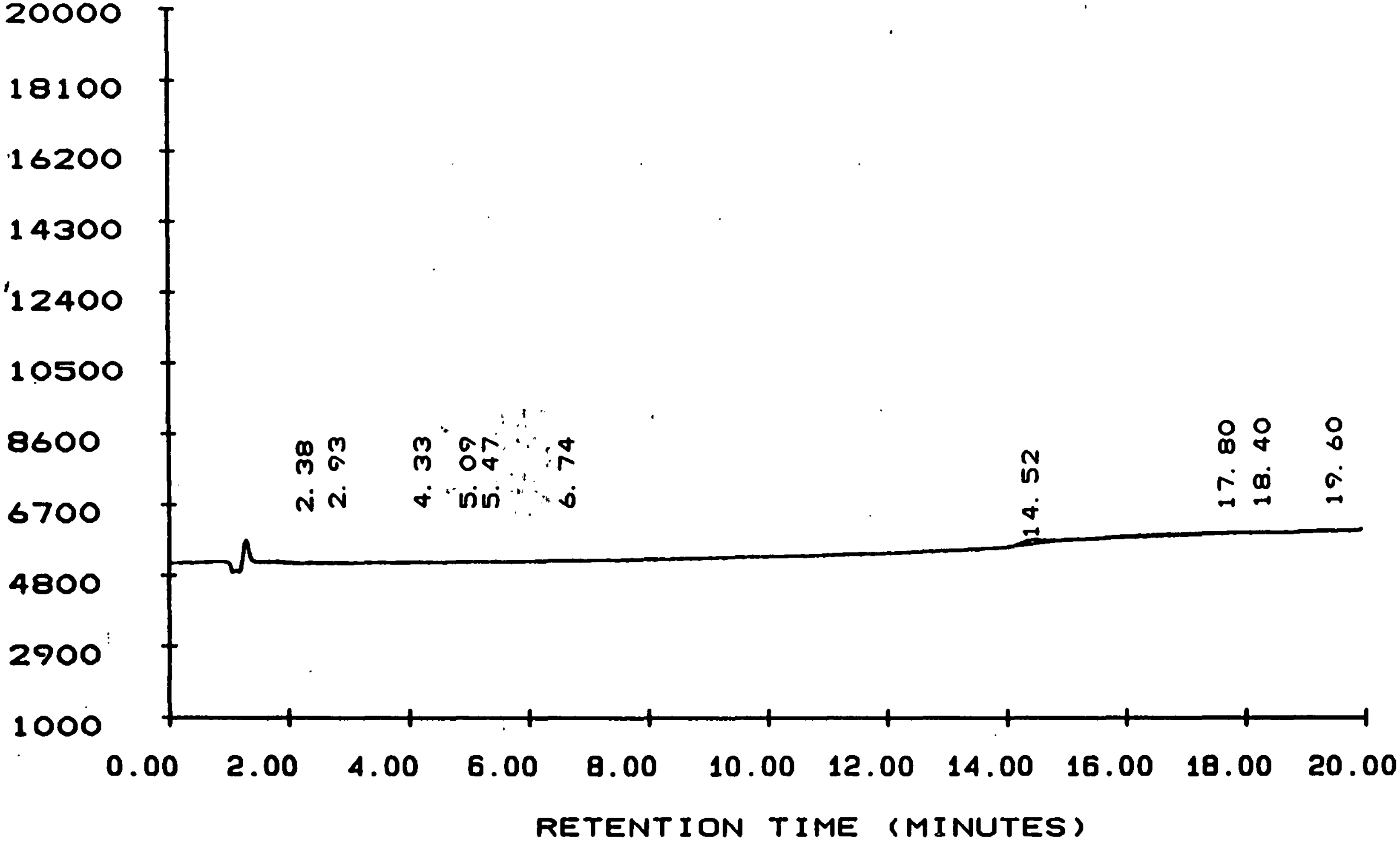
                                                              HEIGHT
PK       PEAK      PEAK   BL    RT   GR COMPONENT              CONC
NO       AREA     HEIGHT      MINUTES #   NAME                 NG/UL
                           V    0.000
001      2004       225    V    2.396
002      2484       129    V    3.976
003      6454       459    V    4.688
004       527       113    V    5.304
005     83198      7948    V    5.862    NAPHTHALENE           0.811
006     79610      7694    V    6.570    ACENAPHTHYLENE        1.151
007     29670      3379    V    7.405    ACENAPHTHENE          0.790
008     34826      3983    V    7.707    FLUORENE              0.092
009    334332     33628    V    8.350
010    852722     84324    V    8.952
011     21482      2081    V    9.689
012      5906       490    V   10.133
013      3177       329    V   11.919
014     69279      5648    V   12.279
015      5412       451    V   13.894
016     10669       791    V   14.485
017      6840       700    V   14.826
018      1947       218    V   15.515
019     11761      1021    V   15.916
020     12179       829        16.338    INDENO(1,2,3-CD)      0.027
022      2497       167    V   17.439
023       677       110    V   17.957
025      5377       290        19.338


All compounds were quantitated using method PAH18
except those which are labeled.
```

OX

SAMPLE NO. : 03179218 .08
TEST NO. :
METHOD NO. : PAHU1 / PAHU1

INSTRUMENT: 18
DATE TIME: 03/17/92 13:41:01
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                03/17/92 14:40:43

                              MULTILEVEL EXTERNAL STANDARD

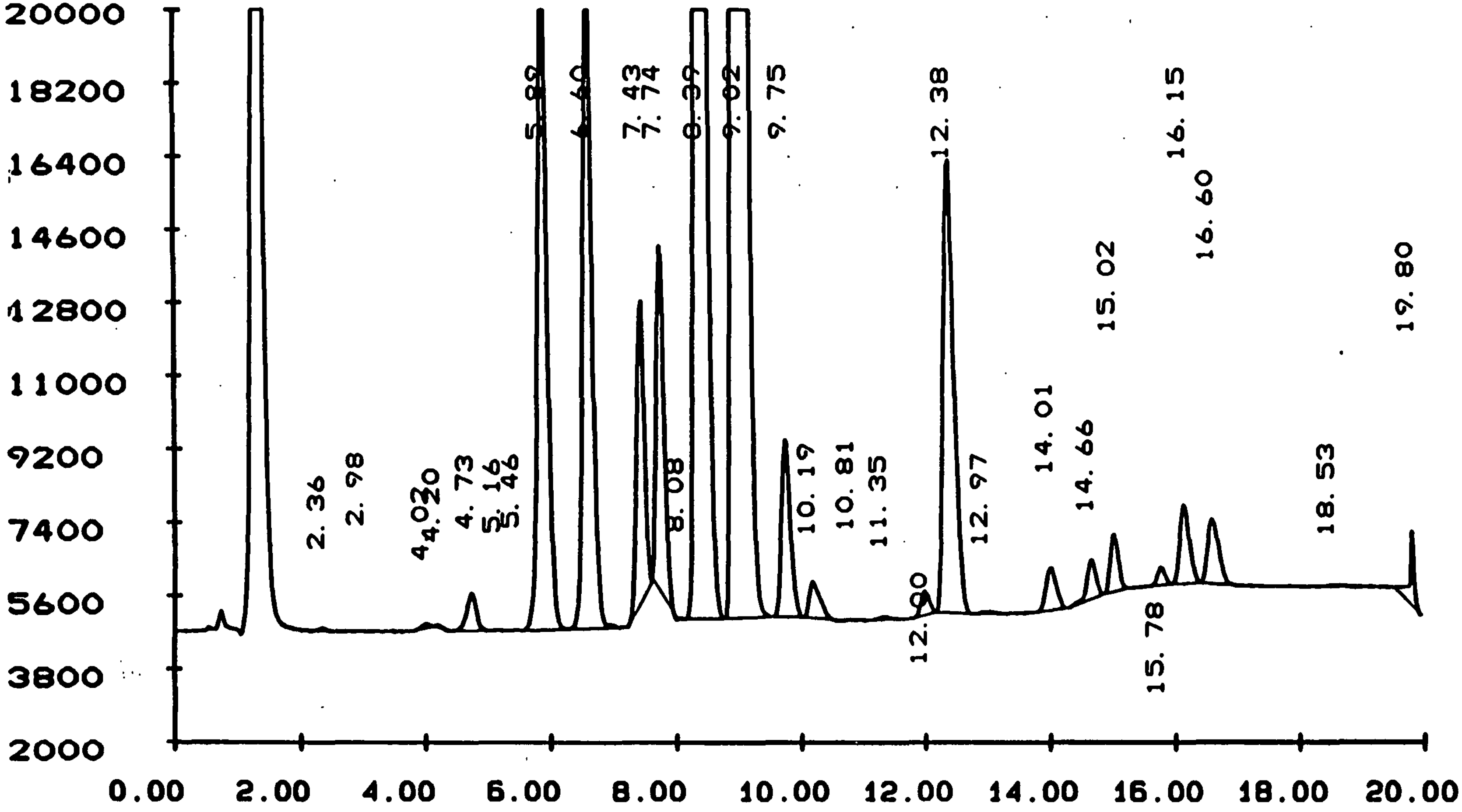
  SAMPLE: 03179218     .08                           INST:18 VIAL:F0 SEQ NUMBER:008
  TEST :                                    DATE-TIME INJECTED   : 03/17/92 13:41:01
  COLLECTION TIME :    19.96                DATE-TIME PROCESSED  : 03/17/92 14:40:43
  METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG          SAMP RATE:     0.78
  CLIENT ID:                                                        SAMPLE VOL:
  CLIENT:                                                 COLUMN TYPE: PAH'S BY HPLC
  LAB ID: 0X                                                 RAW FILE: RAW2:CH242932
  SAMPLE WT :               % MOISTURE   :                DILUTION FACTOR :   1.0000

                                                                  HEIGHT
  PK      PEAK      PEAK  BL    RT   GR COMPONENT                   CONC
  NO      AREA     HEIGHT   MINUTES  #    NAME                      NG/UL
  ==================================================================================
  001      2695      203  V    2.383
  002       698      104  V    2.928
  004      1790      100  V    4.333
  005       492      104  V    5.090
  006      1351      114  V    5.473
                               5.880 M NAPHTHALENE
                               6.590 M ACENAPHTHYLENE
  007      3841      106  V    6.744
                               7.430 M ACENAPHTHENE
                               7.740 M FLUORENE
  009     27928      961  V   14.517
                              16.700 M INDENO(1,2,3-CD)
  010     55110      184  V   17.801
  011      3048      125  V   18.400
  012      4805      102      19.602
```

STD PAH 10X

SAMPLE NO. : 03179218 . 18
TEST NO. :
METHOD NO. : PAHU1 / PAH18

INSTRUMENT: 18
DATE TIME: 03/17/92 17:55:17
PAGE NO. : 01



RETENTION TIME (MINUTES)

Y MAXIMUM: 20000.
Y MINIMUM: 2000.

START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                    03/18/92 13:19:02

                       MULTILEVEL EXTERNAL STANDARD

  SAMPLE: 03179218    .18                    INST:18 VIAL:F0 SEQ NUMBER:018
  TEST :                                DATE-TIME INJECTED   : 03/17/92 17:55:17
  COLLECTION TIME :   19.96             DATE-TIME PROCESSED  : 03/18/92 13:19:02
  METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG         SAMP RATE:   0.78
  CLIENT ID:                                                  SAMPLE VOL:
  CLIENT:                                         COLUMN TYPE: PAH'S BY HPLC UV DET
  LAB ID: STD PAH 10X                                    RAW FILE: RAW2:CH243142
  SAMPLE WT :                % MOISTURE  :               DILUTION FACTOR :  1.0000

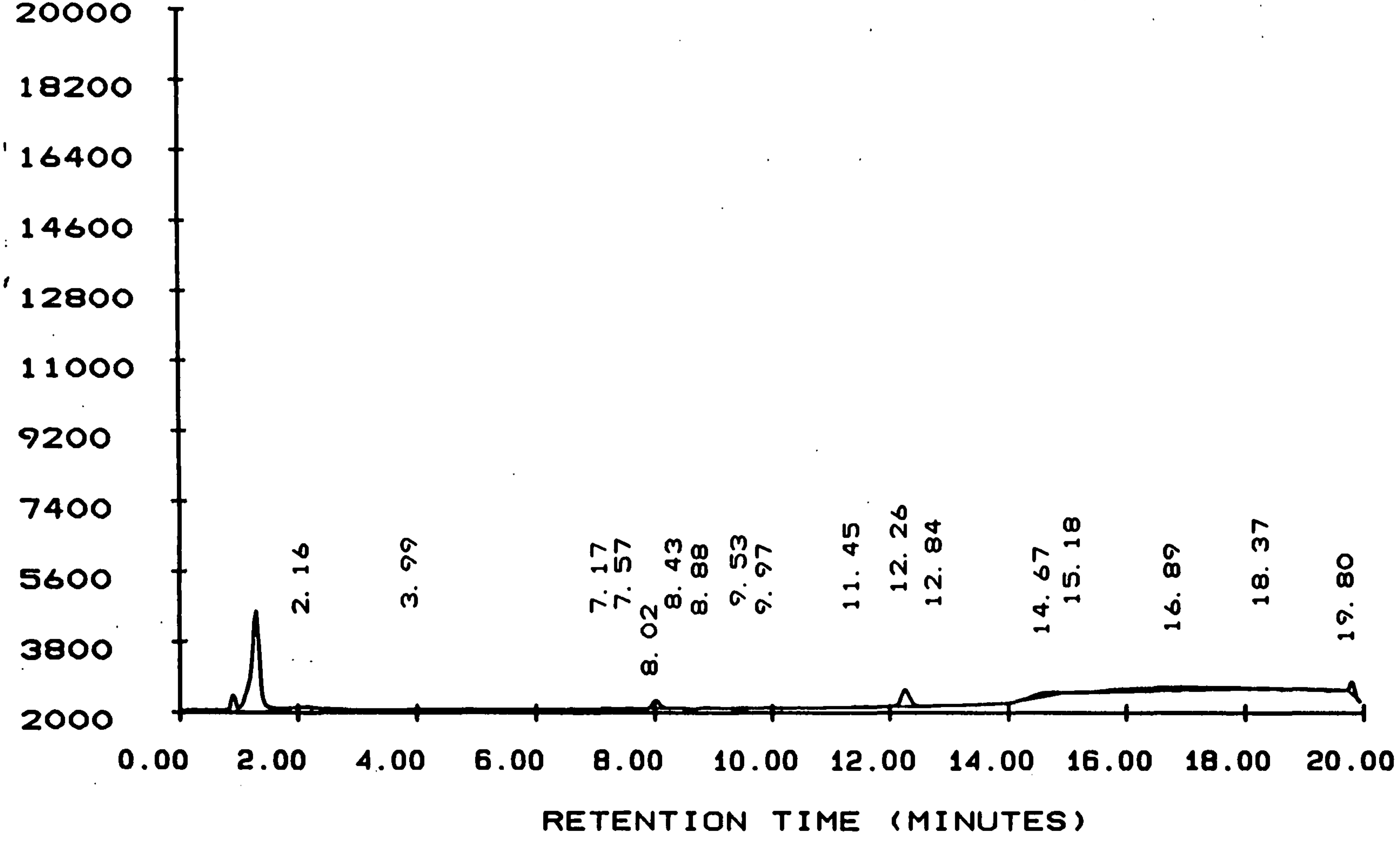
                                                                HEIGHT
  PK       PEAK      PEAK   BL    RT   GR COMPONENT              CONC
  NO       AREA     HEIGHT   MINUTES #    NAME                   NG/UL
  001      5974        683   V   2.362
  002      1169        113   V   2.983
  004      8285        955   V   4.023
  005      3000        507   V   4.205
  006     99822       9081   V   4.735
  007      2065        267   V   5.165                                        Rec %
  008       899        147   V   5.459
  009   1717019     166402   V   5.885    NAPHTHALENE            19.319     107.3
  010   1597659     161647   V   6.598    ACENAPHTHYLENE         24.798     107.8
  011    626222      72033   V   7.431    ACENAPHTHENE           20.425     113.5
  012    720787      83072   V   7.739    FLUORENE                2.358     112.3
  013      1242        247   V   8.078
  014   6694971     684417   V   8.391
  015   3726067     050783   V   9.024
  016    439885      42765   V   9.747
  017    113268       8889   V  10.187
  018      3090        245   V  10.810
  019      8982        675   V  11.349
  020     56053       5713   V  12.000
  021   1317025     110554   V  12.377
  022      8177        623   V  12.974
  023    123813      10401   V  14.013
  024     97683       9455   V  14.663
  025    130259      13863   V  15.022
  026     40154       4270   V  15.777
  027    212339      19050   V  16.147
  028    195151      15608   V  16.598    INDENO(1,2,3-CD)        0.425     98.8
  029      9870        382   V  18.533
  031     99762      13832      19.801
                                                                        3/18/92

  All compounds were quantitated using method PAH18
  except those which are labeled.
```

RAW QC DATA

92LLC030-MB1

SAMPLE NO. : 03179217 .09
INSTRUMENT: 17
TEST NO. :
DATE TIME: 03/17/92 14:05:20
METHOD NO. : PAHF1 / PAHF1
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                    03/17/92 14:40:07

                           MULTILEVEL EXTERNAL STANDARD

 SAMPLE: 03179217    .09                         INST:17 VIAL:F0 SEQ NUMBER:009
 TEST :                                  DATE-TIME INJECTED  : 03/17/92 14:05:20
 COLLECTION TIME :   19.96               DATE-TIME PROCESSED : 03/17/92 14:40:07
 METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG         SAMP RATE:   0.78
 CLIENT ID:                                                    SAMPLE VOL:
 CLIENT:                                             COLUMN TYPE: PAH BY HPLC FL
 LAB ID: 92LLC030-MB1                                    RAW FILE: RAW2:CH242950
 SAMPLE WT :               % MOISTURE  :               DILUTION FACTOR :  1.0000

                                                                HEIGHT
 PK       PEAK      PEAK   BL    RT   GR COMPONENT               CONC
 NO       AREA     HEIGHT   MINUTES #    NAME                    NG/UL
 ==========================================================================
 001      4429       320   V    2.158
 003      2372       165   V    3.993
 008      2960       284   V    7.170
 009      2160       201   V    7.575
 010     21545      2191   V    8.020
 011      1760       190   V    8.426 M PHENANTHRENE            0.168
 012      5627       339   V    8.875 M ANTHRACENE              0.184
 013      1927       102   V    9.530 M FLUORANTHRENE           0.057
 014      4203       326   V    9.966 M PYRENE                  0.089
 016      4889       261   V   11.455
                               11.930 M BENZO(A)ANTHRACENE
 017     46794      4259   V   12.262 M CHRYSENE                0.091
 018      1981       174   V   12.843
                               13.870 M BENZO(B)FLUORANTHREN
                               14.470 M BENZO(K)FLUORANTHREN
 019     26771       888   V   14.673 M BENZO(A)PYRENE
 020       822       121   V   15.184
                               15.740 M DIBENZO(A,H)
                               16.160 M BENZO(GHI)
 021     89475       817   V   16.890
 022      7736       160   V   18.373
 023     17325      3001       19.796
```

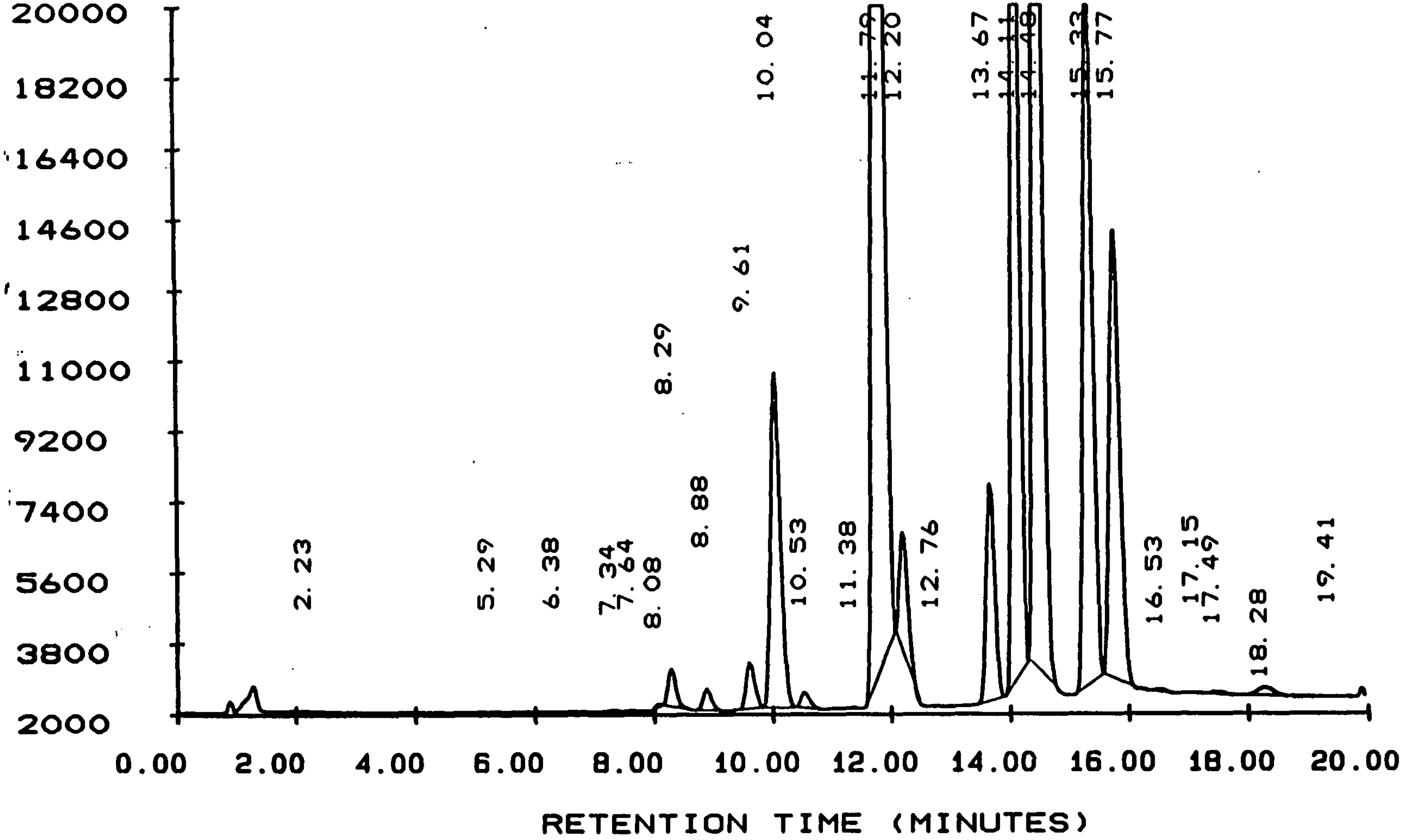
All < 0.5 X

PH 3/18/92

92LLC030-MB1S

SAMPLE NO. : 03179217 .10
TEST NO. :
METHOD NO. : PAHF1 / PAHF1

INSTRUMENT: 17
DATE TIME: 03/17/92 14:29:40
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                    03/17/92 15:02:12

                               MULTILEVEL EXTERNAL STANDARD

  SAMPLE: 03179217     .10                          INST:17 VIAL:F0 SEQ NUMBER:010
  TEST :                                    DATE-TIME INJECTED   : 03/17/92 14:29:40
  COLLECTION TIME :   19.96                 DATE-TIME PROCESSED : 03/17/92 15:02:12
  METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG          SAMP RATE:   0.78
  CLIENT ID:                                                     SAMPLE VOL:
  CLIENT:                                               COLUMN TYPE: PAH BY HPLC FLUORESE
  LAB ID: 92LLC030-MB1MS                                  RAW FILE: RAW2:CH242972
  SAMPLE WT :                 % MOISTURE   :               DILUTION FACTOR :  1.0000

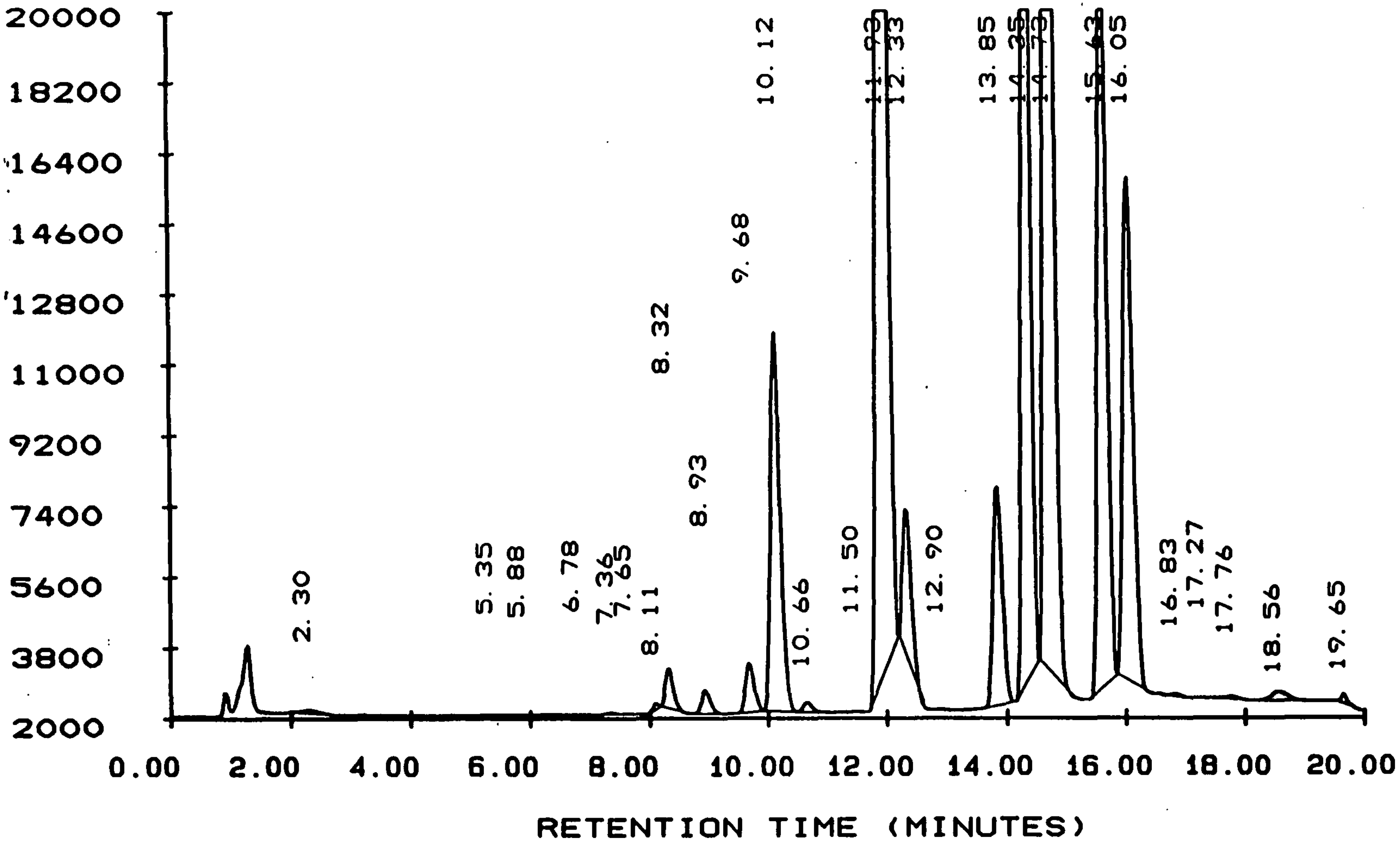
                                                                     HEIGHT
  PK        PEAK       PEAK   BL    RT   GR COMPONENT                 CONC
  NO        AREA      HEIGHT   MINUTES  #   NAME                     NG/UL
                                V   0.000
                                V   0.000
  001       3008        161   V   2.228
  003       3035        147   V   5.294
  004       3328        129   V   6.378
  005       3676        311   V   7.336
  006       1580        162   V   7.638
  007       4290        636   V   8.083                                          Rec %
  008      89459       9391   V   8.288  PHENANTHRENE              0.429         85.8
  009      57498       5480   V   8.881  ANTHRACENE                0.477         95.4
  010     118728      11479   V   9.607  FLUORANTHRENE             0.550         110.0
  011     956774      84317   V  10.036  PYRENE                    1.996
  012      42458       3921   V  10.533
  013       5811        221   V  11.378
  014    4890461     389154   T  11.793  BENZO(A)ANTHRACENE        0.443         88.6
  015     560138      44031   T  12.196  CHRYSENE                  0.502         100.4
  016       3648        297   V  12.761
  017     551626      54179   V  13.668  BENZO(B)FLUORANTHREN      0.556         111.2
  018    2581121     284736   V  14.113  BENZO(K)FLUORANTHREN      0.656         131.2
  019    3653559     348357      14.482  BENZO(A)PYRENE            0.497         99.4
  020    1951623     180414   V  15.332  DIBENZO(A,H)              0.482         96.4
  021    1389855     113857      15.768  BENZO(GHI)                0.379         75.8
  022       6278        635   V  16.527
  023       1776        122   V  17.148
  024       9096        621   V  17.487                                        PH 3/18/92
  025      41023       2093   V  18.280
  026       1758        108      19.411


  All compounds were quantitated using method PAH17
  except those which are labeled.
```

92LLC030-MB1T

SAMPLE NO. : 03179217 .11
TEST NO. :
METHOD NO. : PAHF1 / PAHF1

INSTRUMENT: 17
DATE TIME: 03/17/92 14:54:00
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

START TIME: 0.00
END TIME: 20.00

```
Roy F. Weston, Inc. - Lionville Laboratory                03/17/92 15:20:51

                         MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217     .11                     INST:17 VIAL:F0 SEQ NUMBER:011
TEST :                               DATE-TIME INJECTED  : 03/17/92 14:54:00
COLLECTION TIME :   19.96            DATE-TIME PROCESSED : 03/17/92 15:20:51
METHOD:  PAHF1 / PAH17   REV #: 00061  ANALYST: HUANG       SAMP RATE:   0.78
CLIENT ID:                                                SAMPLE VOL:
CLIENT:                                       COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: 92LLC030-MB1MSD                                RAW FILE: RAW2:CH242994
SAMPLE WT :                 % MOISTURE  :         DILUTION FACTOR :  1.0000

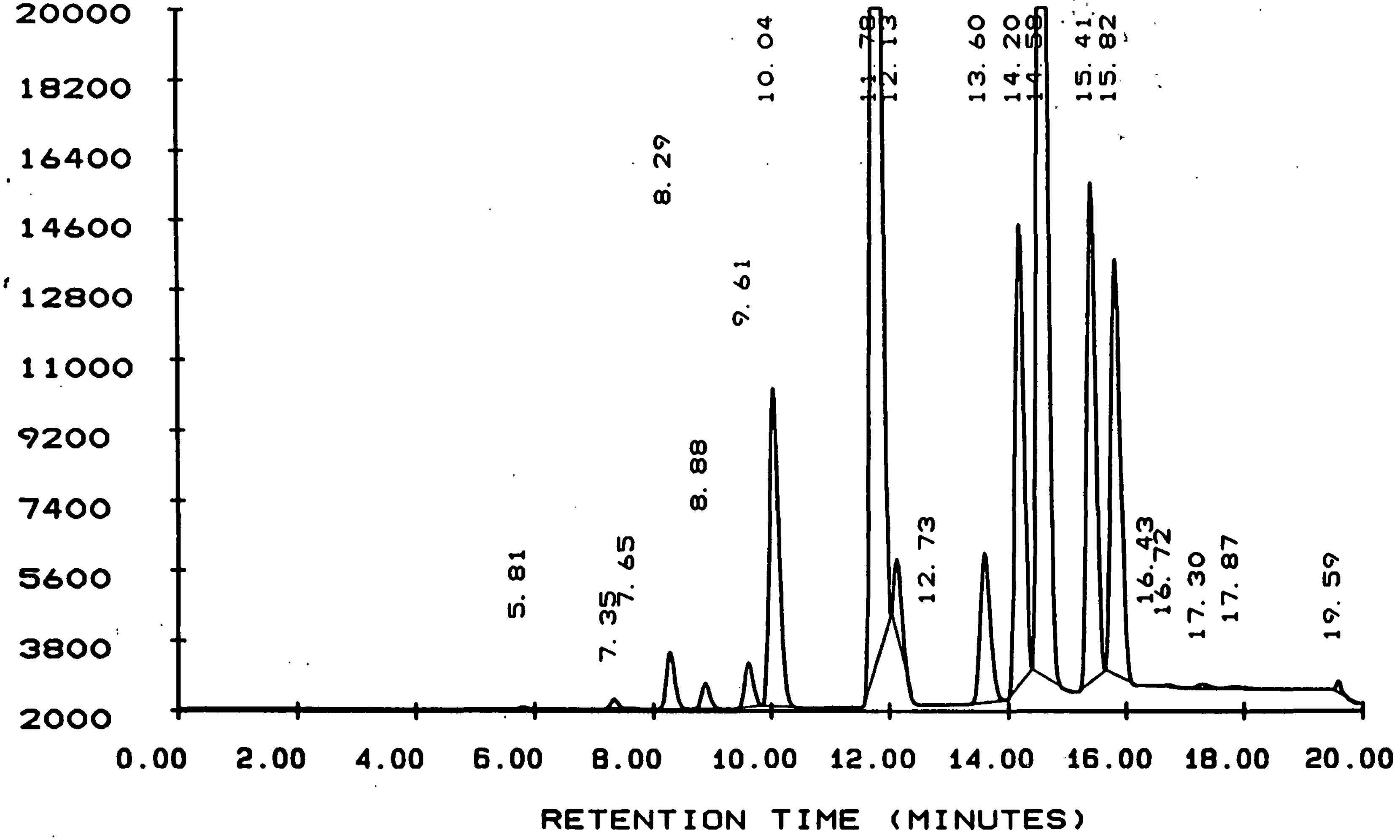
                                                            HEIGHT
PK       PEAK       PEAK   BL    RT   GR COMPONENT           CONC
NO       AREA     HEIGHT      MINUTES #   NAME               NG/UL
001     16704        861   V    2.300
004      2342        157   V    5.351
005      3075        233   V    5.879
007      1298        113   V    6.776
008      4547        417   V    7.360
009      1958        226   V    7.655                                   Rec %
010      8683       1209   V    8.113
011     91945      10010   V    8.324  PHENANTHRENE         0.446      89.2
012     65459       6131   V    8.933  ANTHRACENE           0.515      103.0
013    128880      12300   V    9.683  FLUORANTHRENE        0.586      117.2
014   1090667      95336   V   10.123  PYRENE               2.246
015     27029       2516   V   10.661
016      5198        266   T   11.496
017   5100339     430096   T   11.933  BENZO(A)ANTHRACENE   0.490      98.0
018    645603      50708   T   12.329  CHRYSENE             0.572      114.4
019      4288        364   V   12.905
020    596542      54948   V   13.849  BENZO(B)FLUORANTHREN 0.564      112.8
021   2769325     301169   V   14.352  BENZO(K)FLUORANTHREN 0.694      138.8
022   4230493     397217   V   14.728  BENZO(A)PYRENE       0.567      113.4
023   2115745     201803   V   15.627  DIBENZO(A,H)         0.539      107.8
024   1511859     127266   V   16.048  BENZO(GHI)           0.424      84.8
025      5756        583   V   16.827
026       880        123   V   17.275
027     12953        757   V   17.762
028     47248       2329   V   18.564
029     16538       2404       19.649
                                                                 3/18/92

All compounds were quantitated using method PAH17
except those which are labeled.
```

CLEANUP BS

SAMPLE NO. : 03179217 . 12
TEST NO. :
METHOD NO. : PAHF1 / PAHF1

INSTRUMENT: 17
DATE TIME: 03/17/92 15: 18: 19
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 2000.

START TIME: 0. 00
END TIME: 20. 00

Roy F. Weston, Inc. - Lionville Laboratory 03/17/92 15:51:57

MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179217 .12 INST:17 VIAL:F0 SEQ NUMBER:012
TEST : DATE-TIME INJECTED : 03/17/92 15:18:19
COLLECTION TIME : 19.96 DATE-TIME PROCESSED : 03/17/92 15:51:57
METHOD: PAHF1 / PAH17 REV #: 00061 ANALYST: HUANG SAMP RATE: 0.78
CLIENT ID: SAMPLE VOL:
CLIENT: COLUMN TYPE: PAH BY HPLC FLUORESE
LAB ID: CLEANUP BS RAW FILE: RAW2:CH243010
SAMPLE WT : % MOISTURE : DILUTION FACTOR : 1.0000

PK NO	PEAK AREA	PEAK HEIGHT	BL	RT MINUTES	GR #	COMPONENT NAME	HEIGHT CONC NG/UL	Rec %
			V	0.000				
002	5350	451	V	5.808				
003	22814	2490	V	7.347				
004	1116	172	V	7.646				
005	145024	14234	V	8.286		PHENANTHRENE	0.566	113.2
006	66361	6382	V	8.880		ANTHRACENE	0.529	105.8
007	113739	11073	V	9.613		FLUORANTHRENE	0.533	106.6
008	917560	80398	V	10.043		PYRENE	1.907	
010	4319545	368092	T	11.778		BENZO(A)ANTHRACENE	0.420	84.0
011	449473	37216	T	12.127		CHRYSENE	0.432	86.4
012	4428	237	V	12.731				
013	454503	37633	V	13.604		BENZO(B)FLUORANTHREN	0.387	77.4
014	1194291	116323		14.198		BENZO(K)FLUORANTHREN	0.268	53.6
015	3670733	345514	V	14.582		BENZO(A)PYRENE	0.493	98.6
016	1333477	126563	V	15.415		DIBENZO(A,H)	0.338	67.6
017	1211623	105517	V	15.823		BENZO(GHI)	0.352	70.4
018	1373	159	V	16.431				
019	3830	459	V	16.716				
020	14465	1065	V	17.304				
021	10844	627	V	17.867				
022	17773	2979		19.593				

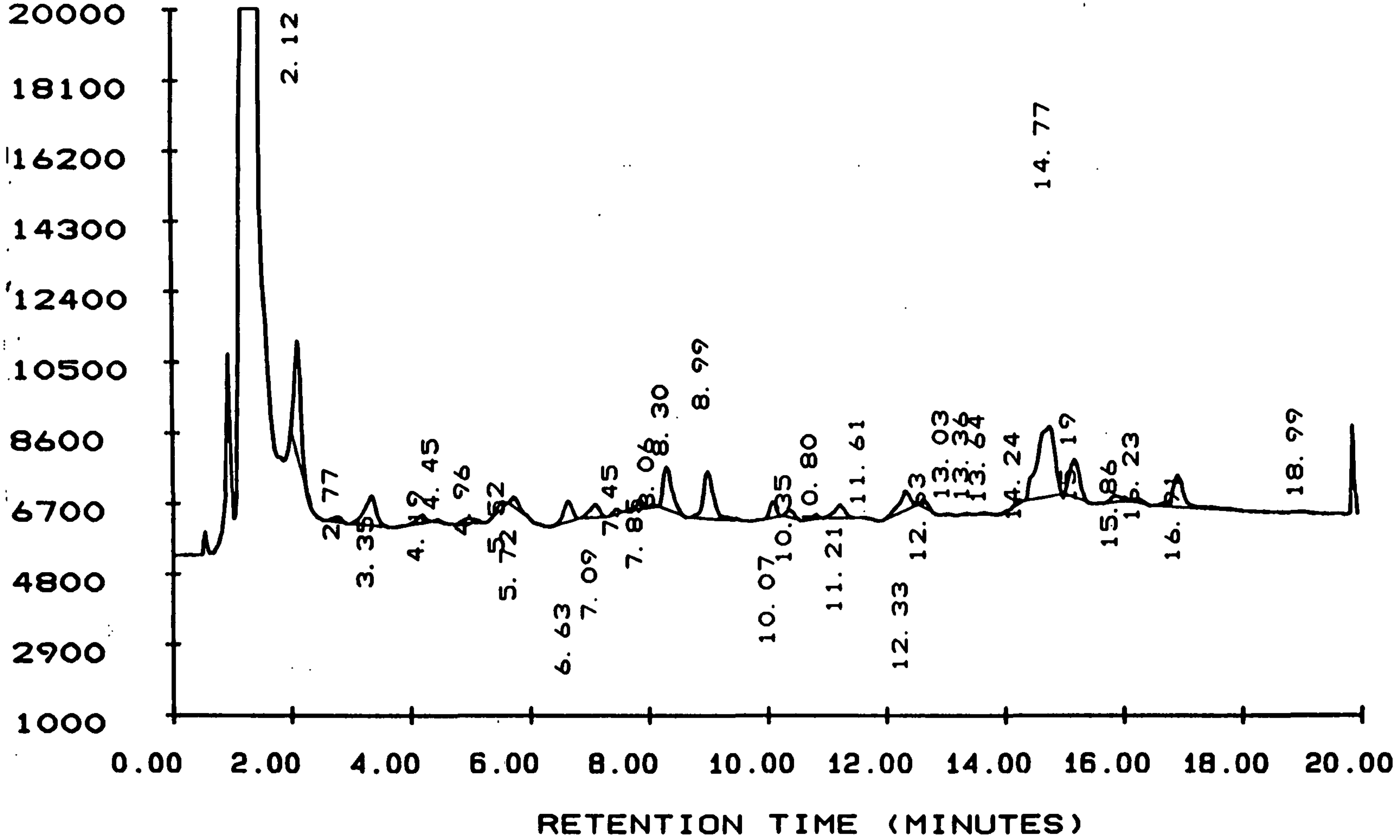
31.8.92

All compounds were quantitated using method PAH17
except those which are labeled.

92LLC030-MB1

SAMPLE NO. : 03179218 .09
TEST NO. :
METHOD NO. : PAHU1 / PAHU1

INSTRUMENT: 18
DATE TIME: 03/17/92 14:05:20
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0.00
END TIME: 20.00

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    Roy F. Weston, Inc. - Lionville Laboratory                03/17/92 14:41:09

                          MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179218     .09                      INST:18 VIAL:F0 SEQ NUMBER:009
TEST :                                DATE-TIME INJECTED   : 03/17/92 14:05:20
COLLECTION TIME :    19.96            DATE-TIME PROCESSED  : 03/17/92 14:41:09
METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG          SAMP RATE:   0.78
CLIENT ID:                                                 SAMPLE VOL:
CLIENT:                                            COLUMN TYPE: PAH'S BY HPLC
LAB ID: 92LLC030-MB1                                  RAW FILE: RAW2:CH242951
SAMPLE WT :                 % MOISTURE   :           DILUTION FACTOR :  1.0000

                                                               HEIGHT
PK        PEAK       PEAK   BL    RT   GR COMPONENT              CONC
NO        AREA      HEIGHT    MINUTES  #   NAME                  NG/UL
==============================================================================
001     233498      31381   V    2.123
002      14297       1390   V    2.771
003     108577       7880   V    3.351
004      22598       1980   V    4.189
005       3375        588   V    4.450
006      20472       1289   V    4.962
007      18282       1748   V    5.524
008      29245       3034   V    5.719
                                 5.880 M NAPHTHALENE
009      58274       5530   V    6.632 M ACENAPHTHYLENE           0.819
010      44557       3557   V    7.093
011      10768       1289   V    7.452 M ACENAPHTHENE             0.192
012      20780       2191   V    7.854 M FLUORENE                 0.040
013       3756        565   V    8.064
014     120391      11425   V    8.304
015     162536      12654   V    8.994
016      35541       4244   V   10.072
017      19860       2063   V   10.355
018      10167        976   V   10.799
019      44398       3390   V   11.206
020       4626        502   V   11.606
021      70782       4950   V   12.327
022      17266       2015   V   12.626
023       2879        353   V   13.027
024       4215        375   V   13.365
025       4650        441   V   13.641
026       8481        914   V   14.240
027     391776      18522   V   14.765
028     119492      10117   V   15.186
029      21531       1676   V   15.862
030      10314        943   V   16.234
031     133629       8529   V   16.913 M INDENO(1,2,3-CD)         0.235
032       3229        310   V   18.993
```
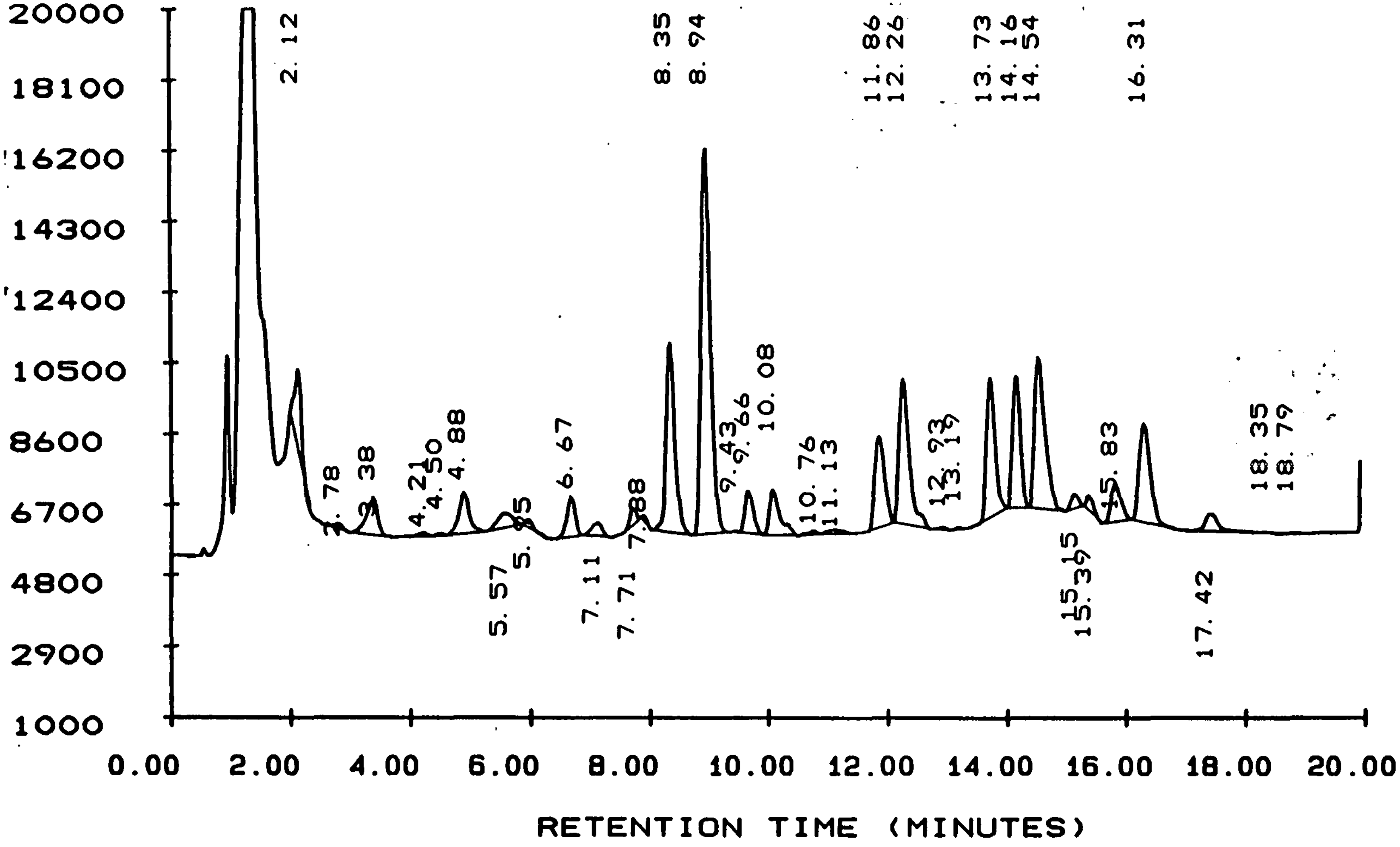

Interference

3/18/92

92LLC030-MB1S

SAMPLE NO. :	03179218 .10	INSTRUMENT:	18
TEST NO. :		DATE TIME:	03/17/92 14:29:40
METHOD NO. :	PAHU1 / PAHU1	PAGE NO. :	01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

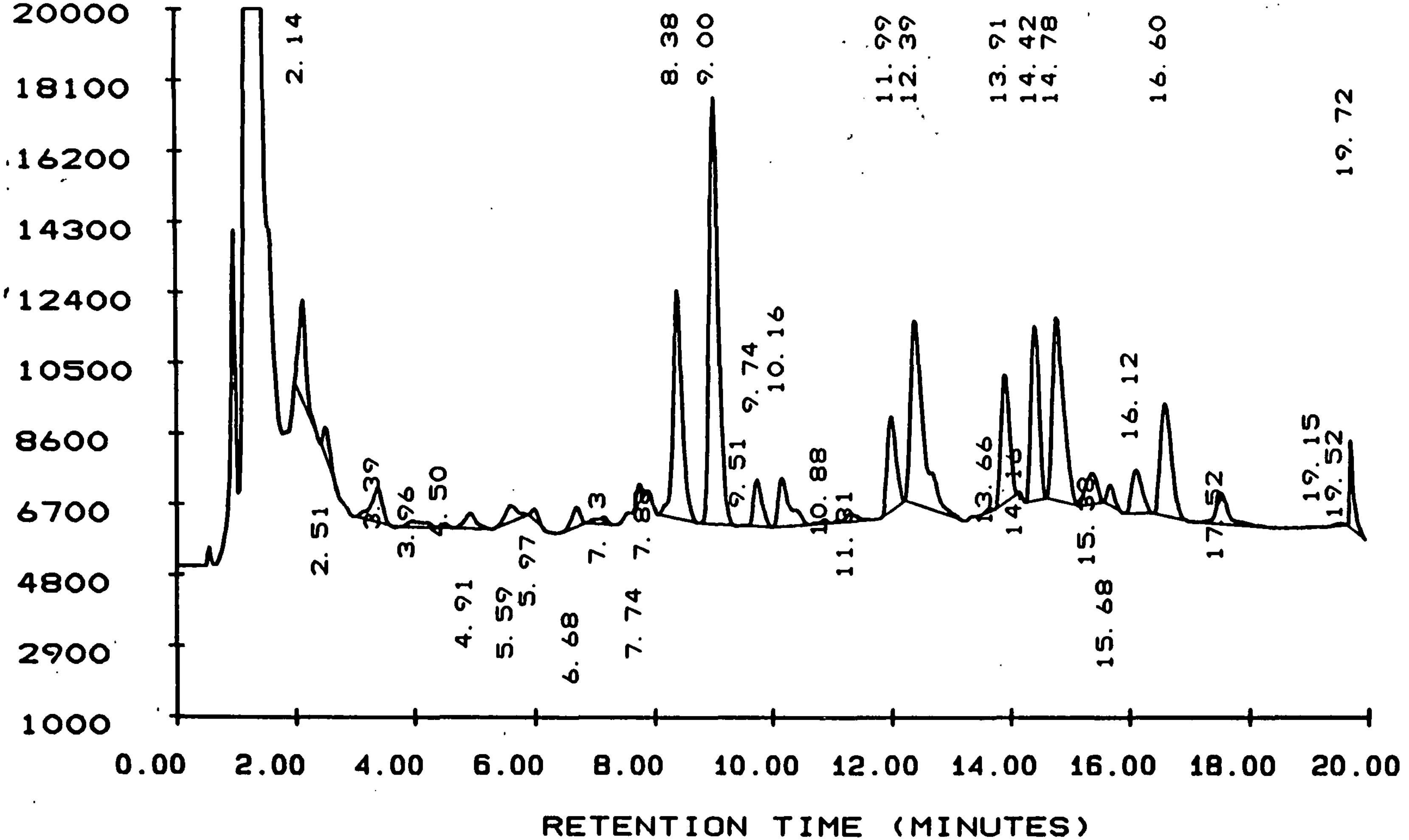
START TIME: 0.00
END TIME: 20.00

```
    Roy F. Weston, Inc. - Lionville Laboratory                    03/17/92 15:02:44

                          MULTILEVEL EXTERNAL STANDARD

  SAMPLE: 03179218    .10                             INST:18 VIAL:F0 SEQ NUMBER:010
  TEST :                                      DATE-TIME INJECTED   : 03/17/92 14:29:40
  COLLECTION TIME :   19.96                   DATE-TIME PROCESSED  : 03/17/92 15:02:44
  METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG           SAMP RATE:    0.78
  CLIENT ID:                                                      SAMPLE VOL:
  CLIENT:                                                  COLUMN TYPE: PAH'S BY HPLC UV DET
  LAB ID: 92LLC030-MB1MS                                          RAW FILE: RAW2:CH242973
  SAMPLE WT :               % MOISTURE  :                  DILUTION FACTOR :   1.0000

                                                                  HEIGHT
  PK       PEAK       PEAK   BL    RT   GR COMPONENT                CONC
  NO       AREA     HEIGHT     MINUTES #   NAME                    NG/UL
                              V    0.000
                              V    0.000
  001    164227      22035    V    2.124
  002     12282       1306    V    2.785
  003    130996       9807    V    3.376
  004     11915       1061    V    4.213                                        Rec %.
  005      4344        581    V    4.500
  006    132250      10793    V    4.878
  007     71215       3966    V    5.566   NAPHTHALENE             0.346
  008     17588       2161    V    5.949   ACENAPHTHYLENE          0.301
  009    106398      10581    V    6.668                                      N/A.
  010     44728       3635         7.114   ACENAPHTHENE            0.863
  011     32242       4068    V    7.709   FLUORENE                0.094
  012      7453       1650    V    7.878
  013    530883      50143    V    8.346
  014   1131181     102782    V    8.945
  015       748        121    V    9.433
  016    112006      11242    V    9.665
  017    155358      12047    V   10.078
  018     10618       1012    V   10.762
  019     19825       1101    V   11.126
  020    275038      24197    V   11.859
  021    478893      38676    V   12.260
  022      5312        560    V   12.931
  023      2670        411    V   13.192
  024    374674      36708    V   13.730
  025    325210      34545    V   14.165
  026    496528      40128    V   14.540
  027     34359       3610    V   15.154
  028     30140       4060    V   15.386
  029    121306       9958    V   15.832
  030    349295      26442        16.309   INDENO(1,2,3-CD)        0.717
  031     73446       4623    V   17.416                           0.482       96.4%
  032       869        143    V   18.349
  033       908        154        18.787


  All compounds were quantitated using method PAH18
  except those which are labeled.
```

92LLC030-MB1T

SAMPLE NO. : 03179218 .11
TEST NO. :
METHOD NO. : PAHU1 / PAHU1

INSTRUMENT: 18
DATE TIME: 03/17/92 14:54:00
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0.00
END TIME: 20.00

Roy F. Weston, Inc. - Lionville Laboratory 03/17/92 15:21:53

MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179218 .11 INST:18 VIAL:F0 SEQ NUMBER:011
TEST : DATE-TIME INJECTED : 03/17/92 14:54:00
COLLECTION TIME : 19.96 DATE-TIME PROCESSED : 03/17/92 15:21:53
METHOD: PAHU1 / PAH18 REV #: 00061 ANALYST: HUANG SAMP RATE: 0.78
CLIENT ID: SAMPLE VOL:
CLIENT: COLUMN TYPE: PAH'S BY HPLC UV DET
LAB ID: 92LLC030-MB1MSD RAW FILE: RAW2:CH242995
SAMPLE WT : % MOISTURE : DILUTION FACTOR : 1.0000

PK NO	PEAK AREA	PEAK HEIGHT	BL	RT MINUTES	GR #	COMPONENT NAME	HEIGHT CONC NG/UL	
			V	0.000				
001	222998	27022	V	2.136				
002	58266	7968	V	2.509				
003	122525	9214	V	3.393				
004	32706	1588	V	3.964				
005	7503	1040	V	4.497				
006	54491	4019	V	4.909				Rec %
007	63069	4287	V	5.588				
008	22180	2892	V	5.973		NAPHTHALENE	0.221	
009	49817	4963	V	6.680		ACENAPHTHYLENE	0.732	NA
010	23582	1770		7.133		ACENAPHTHENE	0.330	
011	29710	4303	V	7.743		FLUORENE	0.101	
012	6254	1648	V	7.882				
013	637904	60308	V	8.379				
014	1223651	113344	V	8.996				
015	1248	240	V	9.505				
016	124954	12346	V	9.741				
017	180951	12814	V	10.164				
018	9774	1043	V	10.879				
019	32917	2150	V	11.311				
020	260535	24686	V	11.991				
021	739347	48986	V	12.395				
022	6658	745	V	13.659				
023	339460	33867	V	13.908				
024	4678	1063	V	14.156				
025	448762	46813	V	14.418				
026	640545	48750	V	14.782				
027	111905	8196	V	15.379				
028	45222	5452	V	15.684				
029	142810	11563	V	16.115				
030	389540	30189	V	16.601		INDENO(1,2,3-CD)	0.818	
031	130477	8359	V	17.521			0.583	116.6
032	2498	164	V	19.145				
033	7102	583	V	19.523				
034	112141	18635		19.716				

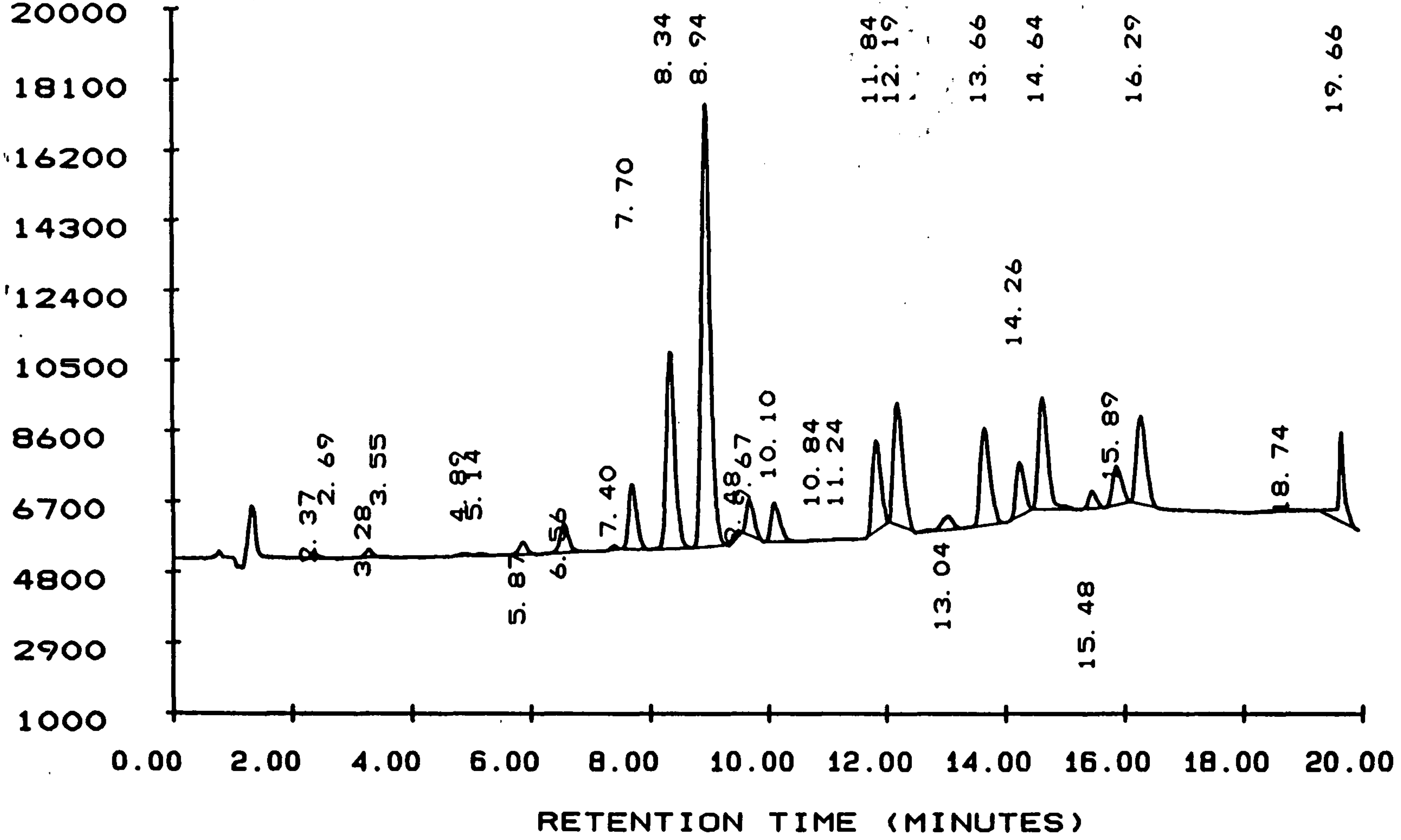
RH 3/18/92

All compounds were quantitated using method PAH18
except those which are labeled.

CLEANUP BS

SAMPLE NO. : 03179218 .12
TEST NO. :
METHOD NO. : PAHU1 / PAHU1

INSTRUMENT: 18
DATE TIME: 03/17/92 15:18:19
PAGE NO. : 01



Y MAXIMUM: 20000.
Y MINIMUM: 1000.

START TIME: 0.00
END TIME: 20.00

```
                         MULTILEVEL EXTERNAL STANDARD

SAMPLE: 03179218    .12                      INST:18 VIAL:F0 SEQ NUMBER:012
TEST :                                 DATE-TIME INJECTED  : 03/17/92 15:18:19
COLLECTION TIME :   19.96              DATE-TIME PROCESSED : 03/17/92 15:53:02
METHOD:  PAHU1 / PAH18   REV #: 00061  ANALYST: HUANG         SAMP RATE:   0.78
CLIENT ID:                                                 SAMPLE VOL:
CLIENT:                                         COLUMN TYPE: PAH'S BY HPLC UV DET
LAB ID: CLEANUP BS                                    RAW FILE: RAW2:CH243011
SAMPLE WT :                 % MOISTURE  :          DILUTION FACTOR :  1.0000

                                                             HEIGHT
PK        PEAK       PEAK  BL    RT   GR COMPONENT             CONC
NO        AREA     HEIGHT     MINUTES #   NAME                 NG/UL
                            V   0.000
001      12776       1670   V   2.372
002        617        122   V   2.688
003      20227       2060   V   3.283
004       1139        144   V   3.553
006       6631        593   V   4.887
007       4629        517   V   5.140
008      37270       3337   V   5.870    NAPHTHALENE           0.273
009      80615       7668   V   6.555    ACENAPHTHYLENE        1.147
010       7241        971   V   7.401    ACENAPHTHENE          0.101
011     162766      17125   V   7.703    FLUORENE              0.468
012     523633      51940   V   8.341
013    1205825     117674   V   8.936
014       7415       1133   V   9.484
015      88769       9650   V   9.669
016     118669      10314   V  10.097
017       3162        208   V  10.840
018       3601        215   V  11.243
019     236328      23561   V  11.836
020     366733      32627   V  12.189
021      53410       3538   V  13.043
022     300314      25746   V  13.664
023     140868      13868   V  14.261
024     332045      29650   V  14.644
025      44785       4582   V  15.476
026     113330      10268   V  15.886
027     270050      23375      16.287    INDENO(1,2,3-CD)      0.634
028      15464        381   V  18.743
029     160034      20176      19.660

All compounds were quantitated using method PAH18
except those which are labeled.
```

Rec %

W/A

126.8

PH 3/18/92

SAMPLE EXTRACTION RECORD

Sheet no.: 1

Extract. Date: 03/04/92
Extraction Batch No: 92LLC030
Analyst: GS
Method: N/A

Test: OLCS
Cleanup Date: 03/12/92
Analyst: PH
Client: KERR MCGEE

LIMS Report Date: 04/09/92
Solvent: DCM
Adsorbent: ALUMIN

Sample No:	Client Name Client ID	pH	Initial WT/VOL	Surr. Mult.	Spike Mult.	Final VOL	Final VOL	Split Mult.	GPC Y/N	% Solids	C/D FACTOR
9202L422-	KERR MCGEE										
001 C	SITE SOIL		10.2			1		1.0	N	66.46	147.5
002 C	SEDIMENT-BKGND		10.0			1		1.0	N	56.01	178.5
003 C	SOIL-BKGND		9.99			1		1.0	N	43.68	229.2
92LLC030-MB1 C			10			1		1.0	N		100.0
92LLC030-MB1 CS			10		1.0	1		1.0	N		100.0
92LLC030-MB1 CT			10		1.0	1		1.0	N		100.0

Comments: BLANK AND BLANK SPIKES WERE NOT CLEANED
Surrogate:
Spike:

Extracts Transferred	Relinquished By	Date Time	Received By	Date Time	Reason for Transfer

END OF DATA PACKAGE