COMPARATIVE EXTRACTION TECHNIQUES
FOR
ENVIRONMENTAL POLLUTANTS

By

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(ABSTRACT)

By addressing new sample preparation techniques, the U.S. Environmental Protection Agency (EPA) has recently implemented research programs to reduce or abolish laboratory pollution. In the work reported here, EPA Method 8270, established for Priority Pollutant Organics, will be evaluated by both Soxhlet and microwave-assisted extraction (MAE) for two classifications of compounds.

The common procedure for sample preparation of solids is Soxhlet extraction. This is a lengthy operation and uses abundant solvent volumes. With ever changing times, the new technology of MAE is surfacing. This technique uses far less solvent and sample preparation times are greatly reduced. The work reported here compares the recoveries of phenolic and polynuclear aromatic compounds for both Soxhlet and closed-vessel MAE.

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INTRODUCTION

By promoting new sample preparation techniques, the U.S. Environmental Protection Agency (EPA) has recently implemented research programs to reduce or abolish laboratory pollution. Sample preparation is as inherently responsible for accuracy and precision of any method as is instrumental analysis. These EPA research programs will concentrate on improving recoveries of target analytes and reducing sample preparation costs[1]. The common technique for the analysis of solids, as established by the EPA, is Soxhlet extraction. With ever changing technology, the new technique of microwave-assisted extraction (MAE) is surfacing. The work reported here compares the recoveries of phenolic and polynuclear aromatic compounds for both Soxhlet and closed-vessel MAE.

The traditional sample preparation method for analyses of soils and sediments is Soxhlet extraction. This extraction process dissolves the components of interest from the sample matrix.[2], by insuring intimate contact of the solid sample with the extraction solvent. In practice, the sample is mixed with anhydrous sodium sulfate, placed in an extraction thimble, and extracted

with a solvent for 16 - 24 hours. The extract is then dried with sodium sulfate, concentrated, and analyzed via each respective technique.[3]

There are several advantages to using the Soxhlet extraction procedure. It is an extraction technique that has been in practice for some time; environmentalists are comfortable with its intricacies; it costs little to purchase the glassware; sample size is limited only by the capacity of the thimble; it is a simple one-step technique. After the extraction process, the concentration step follows immediately; there is no suspended solids filtration step. Once the Soxhlet apparatus is set-up, extraction can run its course unattended.

Unfortunately, there are several disadvantages to using the Soxhlet technique. The Soxhlet extraction method is time and energy consuming, lasting between 4 - 24 hours. Due to the long extraction process, degradation of the components can occur.[4] While start-up costs are small, operating costs are high. This situation is due, in part, to the large amount of solvent that must be used, normally between 100 - 500 mLs per extraction. Due to the large volumes workers are exposed to solvent vapors, which could lead to possible health related illnesses. Soxhlet extraction is also restricted to solids. Extraction temperatures are limited by solvent boiling points. In conductive heating, vaporization at the liquid surface causes a thermal gradient to be established by

convection currents and only a small portion of the liquid is at the temperature of the flask (see figure 1)[5].

Microwave-assisted extraction (MAE) is a new method of extracting soluble analytes into a solvent from a wide range of materials. This extraction process can be understood by a basic physical principle, namely the absorption of microwave energy varies with the chemical nature of the species being irradiated (see figure 2). The parameter generally used as a measure of this physical property is the dielectric constant. In general, the higher the dielectric constant, the higher the absorption of microwave energy.[6] A dielectric is an electrically non-conducting material, also known as an insulator. Some dielectric materials contain permanent dipoles (see table 1). The rotation of the dipoles in an alternating field causes friction, which produces heat. Microwave energy is non-ionizing radiation that causes molecular motion by migrating ions and rotating dipoles, but does not cause changes in molecular structure. Microwaves are electromagnetic waves composed of an electric and magnetic field (see figure 3). They have a frequency range of 300 - 300,000 MHz with four frequencies used for industrial and scientific purposes, the most common being 2450 MHz which is used in all domestic microwave ovens.[5] Use of microwave energy to enhance extraction of organic compounds from solid matrices such as soil, seeds, foods, and feeds was reported by Ganzler and Salgo in 1986 and 1987.[4,7] These researchers used a conventional, household

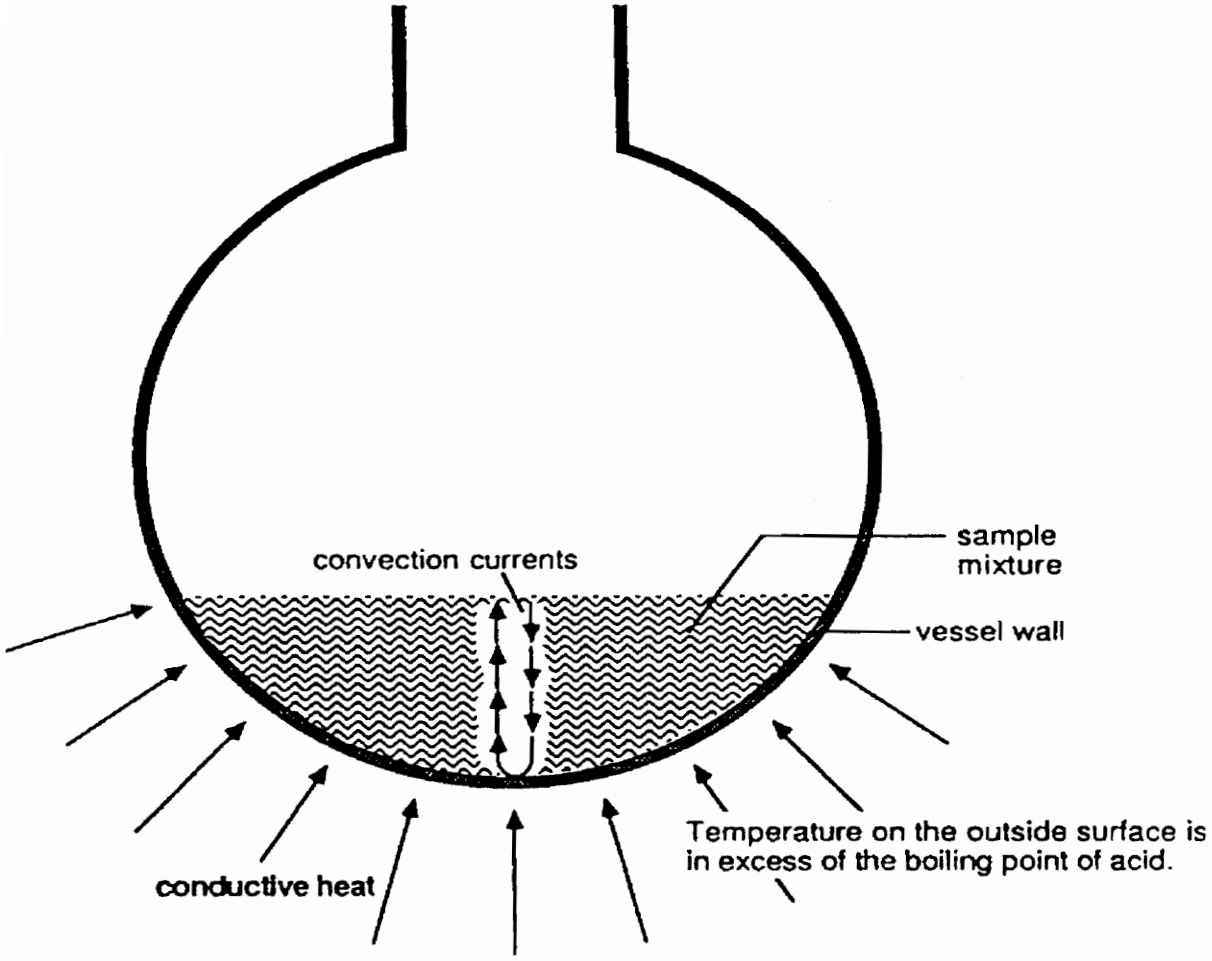


FIGURE 1: Diagram of sample heating by conduction[8]

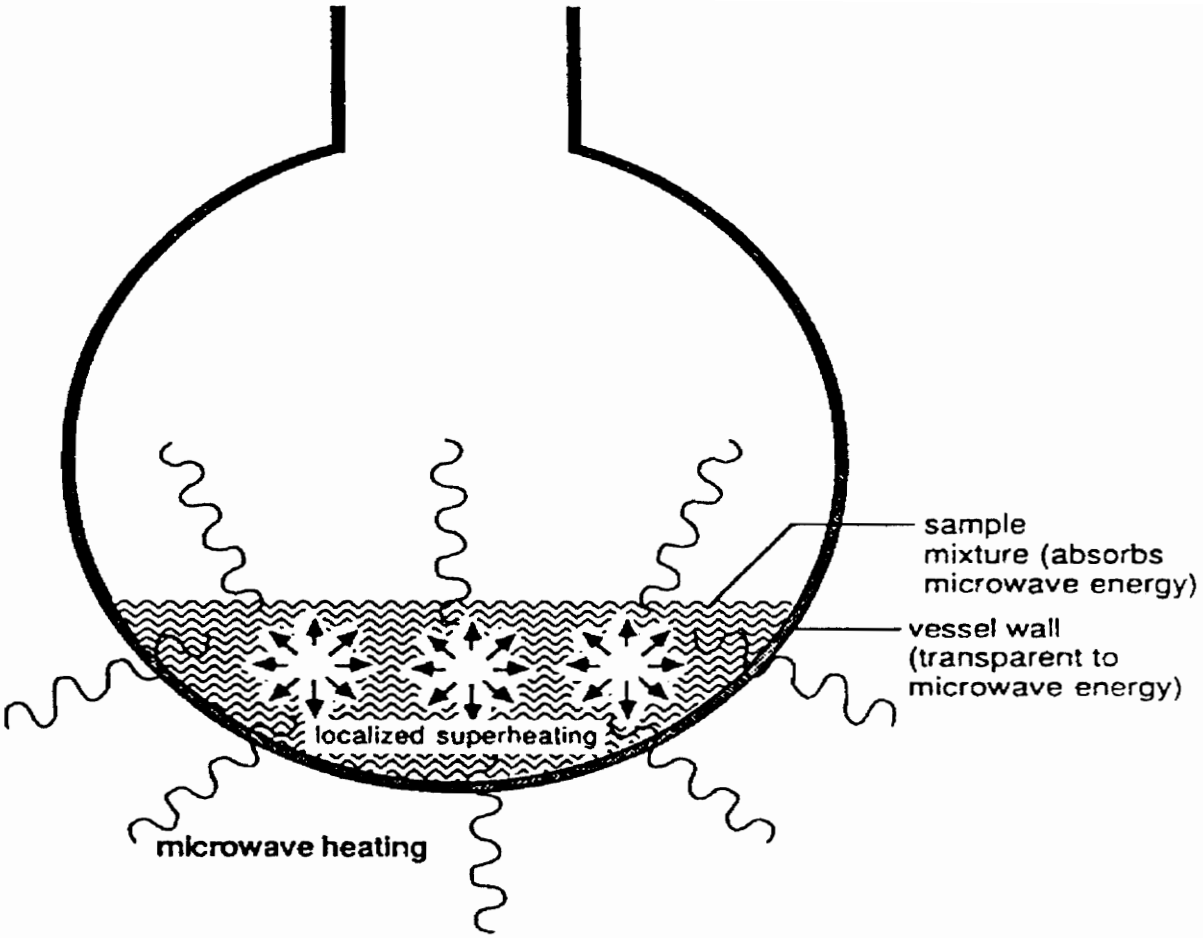
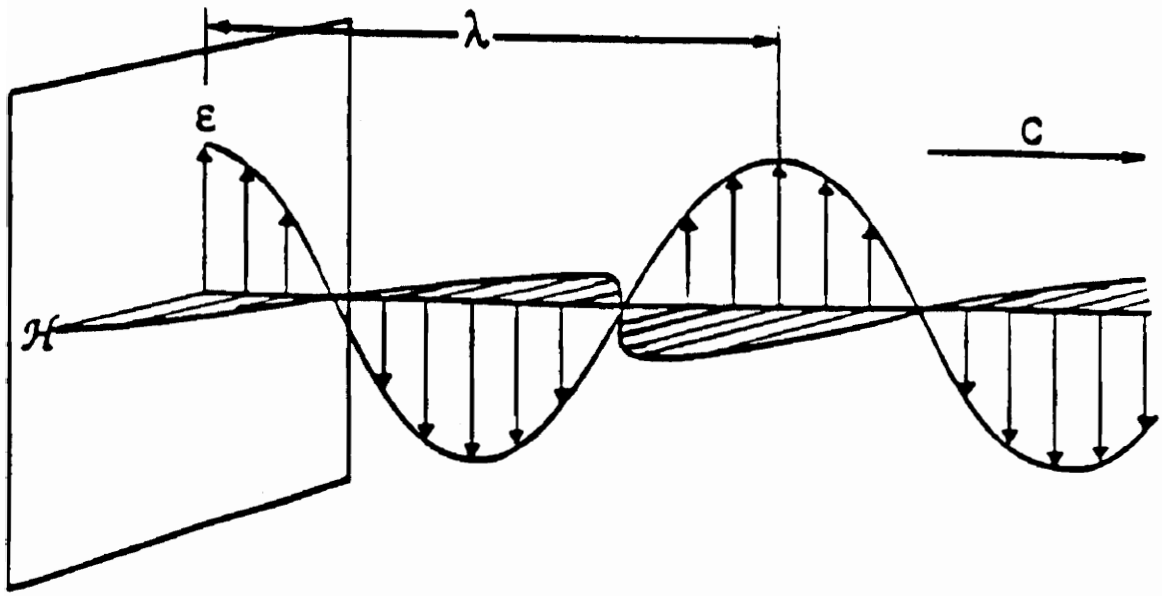


FIGURE 2: Schematic of sample heating by microwave energy[8]

TABLE 1: Dipole Moments and Dielectric Constants for Organic Solvents used in this work[9]

Solvent	Dipole Moment (debyes)	Dielectric Constant
Acetone	2.88	20.770[A]
Chloroform	1.01	4.806[B]
Hexane	0.00	1.890[B]

[A] 25 °C
[B] 20 °C



ε = electric field
ℋ = magnetic field
λ = wavelength (12.2 cm for 2450 MHz)
c = speed of light (300,000 km/s)

FIGURE 3: A Microwave[10]

microwave oven to irradiate solvent/sample suspensions for 30 s up to seven times each and compared this to Soxhlet. They reported that the MAE method was more efficient than Soxhlet extraction for polar compounds.[5] Recently, Onuska and Terry [11] used MAE to extract organochlorine pesticides from sediment samples; they reported quantitative recoveries and no compound breakdown due to sample exposure to microwaves. Extraction of essential and other oils from biological materials such as plant and fish tissue by exposure to microwave energy was recently described in a patent application.[12] In another U.S. patent,[13] extraction of natural products from mint, sea parsley, cedar foliage,and garlic with the solvents hexane, methylene chloride, or ethanol in two or more stages is described. Other researchers have reported use of microwave energy to extract stabilizers from polyolefins.[14]

In organic microwave sample preparation, polar molecules in the solvent and sample orient themselves with the electric field of the radiation. As the field changes, molecular motions result in thermal energy being released. At 2450 MHz, the alignment of the molecules followed by their return to disorder occurs 4.9×10^9 times per second, which results in rapid heating.[5] There is a potential for enhancing analytical capabilities such as increased selectivity and higher sensitivity, due to the increased solvating power of the solvent. Similar or better

linearity and reproducibility factors can exist when compared to Soxhlet[7] since temperature and pressure are controllable.

MAE is gaining in importance for solid matrices because it offers greatly reduced use of organic solvents, reduced extraction times, and increased sample throughput by the use of multivessel systems that allows simultaneous MAE of multiple samples.[15]

Microwaves are generated by a magnetron and are fed into a cavity such as a microwave oven. The inside walls of this cavity are metallic surfaces that completely reflect the microwaves. The heating of a sample with microwave energy depends, in part, upon the dissipation factor (tan δ). The dissipation factor is a ratio of the sample's dielectric loss, so called the loss factor ε'', to its dielectric constant ε', thus : $\tan \varepsilon = \varepsilon''/\varepsilon'$. The dielectric constant is a measure of the sample's ability to absorb the microwave energy as it passes through. The loss factor measures the sample's ability to dissipate that energy. As a sample intercepts the microwaves, energy in the wave is absorbed according to the dissipation factor of the solution.[1]

The polarizability of the solvent molecules obviously depends upon the nature of the solvent and its relative permittivity. Therefore the greater the relative permittivity, the more thermal energy is released, the more rapid the heating. Non-polar solvents, such as hexane and toluene, with low relative permittivities are not affected by microwave energy and therefore require polar additives if they are to be used as solvents in microwave extraction.[5]

Microwaves irradiate all of the sample simultaneously without heating the vessel. Therefore, with microwave heating, the sample reaches its boiling point very rapidly. Also, because the solvent is in a sealed system, it is capable of "reaching" a far greater boiling point than at atmospheric pressure. Polar solvents, such as acetone and dichloromethane, are heated to approximately 100 °C above their normal atmospheric boiling point. It is these higher extraction solvent temperatures and pressures, combined with rapid heating, which increase extraction efficiency and therefore reduce extraction time.[5] Low solvent volumes and thus low exposure rates make this technique environmentally attractive. High throughput is achieved because multiple samples can be run simultaneously.

MAE suffers inherent drawbacks such as the requirement for relatively high pressures and temperatures, and the relatively high capital cost associated with MAE equipment. Another minor limitation is the necessity of a filtration step

when extracting solids. While it is a new technique, the EPA has judged microwave-assisted extraction to be a comparable technique to the conventional extraction procedures.

In some of the previously referenced papers, there was not a fully satisfactory comparison between Soxhlet extraction and MAE. For example in Lopez-Avila and Young [16] the final extract from the MAE process was concentrated to ~5 mL using nitrogen blowdown evaporation. Onuska and Terry [1] used a rotary evaporator to concentrate the MAE extract to 3 mL, followed by further concentration using a gentle stream of nitrogen applied to the extract while suspended in a water bath at 40 °C. Traditionally, the Soxhlet extraction has a concentration step which uses a Kuderna-Danish apparatus to bring the extract to a volume of 1 mL. In this work, both Soxhlet and MAE will undergo the same preparation step prior to GC/MS injection. Doing this will enable, for the first time, a fully satisfactory comparison.

EXPERIMENTAL

Gas Chromatograph/Mass Spectrometer

The gas chromatograph used in this research was a Hewlett Packard (Avondale, PA) model 5890 Series II coupled to a Hewlett Packard (HP) 5971 Mass Selective Detector (MSD). The unit was equipped with an electronic pressure controlled split/splitless capillary injector, and connected to a HP Vectra 386-20 MHz computer with version G1034C ChemStation software.

The HP 5971 Mass Selective Detector (MSD) was a bench-top mass spectrometer which had an electron impact (EI) source, a fused-silica quadrapole mass filter with hyperbolic geometry, and a continuous dynode electron multiplier. The X-Ray lens directs the ion beam into the horn of the electron muliplier. Ions entering the horn strike the sides of it, liberating secondary electrons. In turn, these cascade through the horn, freeing more electrons as they go. From these electrons a logarithmic preamplifier produces a dynamic reponse range of more than six decades. The mass filter consisted of four hyperbolic rods coated on the inside with a conductive material. A combined dc and RF voltage is applied to the two pairs of opposite segments

which are connected together. The magnitude of the RF voltage applied to the mass filter controls the mass-to-charge ratio of ions that pass through the mass filter and reach the detector. The ratio of dc-to-RF voltage determines the resolution. The unit had the capability of monitoring or scanning mass to charge ratios from 1.2 to 650 AMU with a resolution of 0.5 ± 0.05 AMU. The electron impact source was operated with an electron energy of 70eV. This source had two filaments to permit continuous use in the event of one filament failure. Proper alignment was crucial to achieve the maximum number of electrons in the source and therefore the optimum sensitivity. The electron multiplier voltage was adjustable from 0 to 3000 V.

The MSD used a vacuum pumping system consisting of a vapor diffusion pump backed by a two-stage mechanical pump. Overall system pumping speed was around 60 liter/s for helium. A foreline trap was present to prevent vapor oil flash-back into the MS manifold. Prior to any analyses, the vapor diffusion pump and the two-stage mechanical pump oils were changed. In addition, the source was cleaned, the filaments properly aligned, and the foreline trap adsorption pellets replaced.

The interface connecting the gas chromatograph to the mass spectrometer was a capillary direct interface. This type of interface permitted the fused silica capillary column to be inserted through the GC oven wall directly

into the EI source chamber. This interface controlled the temperature of the transfer line portion of the capillary column to ensure that condensation did not occur. The temperature was set and controlled by the GC or by the ChemStation through the GC. The transfer line also heated the MSD source. It does this by direct thermal transfer of heat from the transfer line to the MSD source, source supporting housing, and mass filter (see figure 4B). As a result, the transfer line is normally kept at a temperature between 260 - 290 °C to maintain a source temperature of 180 °C (see figure 4A).

As a proper routine procedure, Perfluorotributylamine (PFTBA) was used as the calibration compound for all tuning procedures. Autotune is an automated tuning program for general purpose MSD operation. The program uses the PFTBA ions at m/z 69,219, and 502. Autotune provides acceptable measurements of ion abundances, peak widths, mass assignments, and relative ion abundances over the entire MSD mass range (see figure 5). The GC/MS conditions are given in Table 2.

TABLE 2: GC/MS Conditions

Injector Parameters	Inj. Temp: 275 ^{0}C Splitless for 0.15 min Flow Rate: 1 mL/min
Oven Parameters	Initial Temp: 70 ^{0}C Hold Time: 2 min Ramp Rate: 10 ^{0}C /min Final Temp: 150 ^{0}C Hold Time: 2 min Ramp Rate: 10 ^{0}C /min Final Temp: 300 ^{0}C Hold Time: 2 min
Column	HP-5MS (Crosslinked 5% Ph Me Silicone) (30m x 0.25mm x 0.25 µm)
Transfer Line Temperature	300 ^{0}C
Electron Energy	70 eV
EM Voltage	1800 V
Solvent Delay	3.5 min

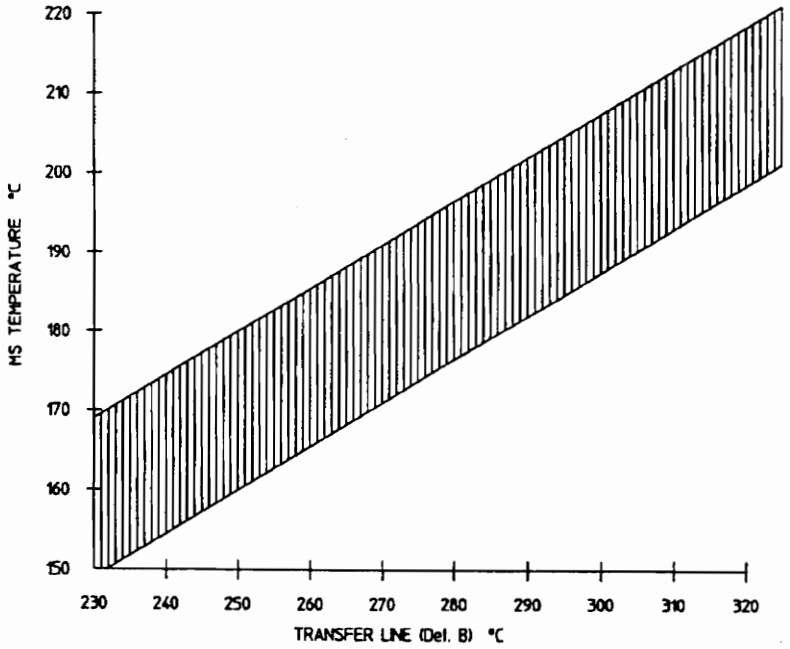


FIGURE 4A: MS Temperature vs. GC/MSD Interface Temperature[17]

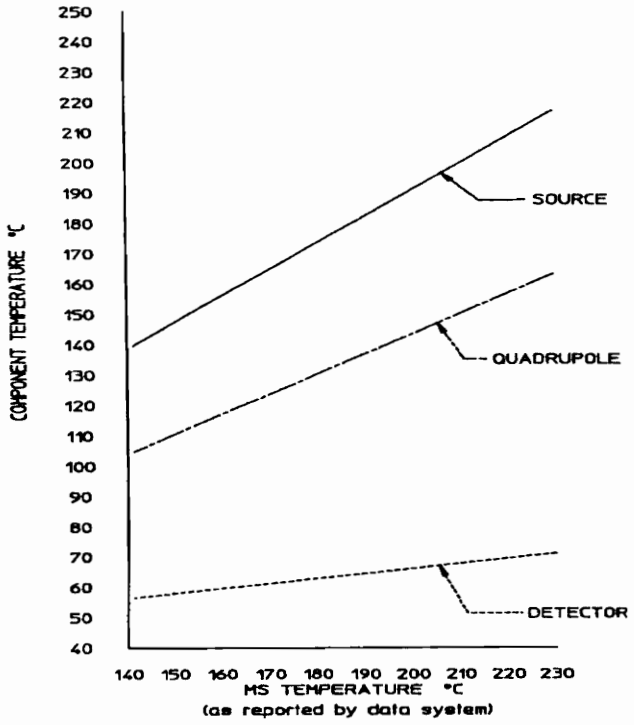


FIGURE 4B: Component Temperatures vs. MS Temperature[17]

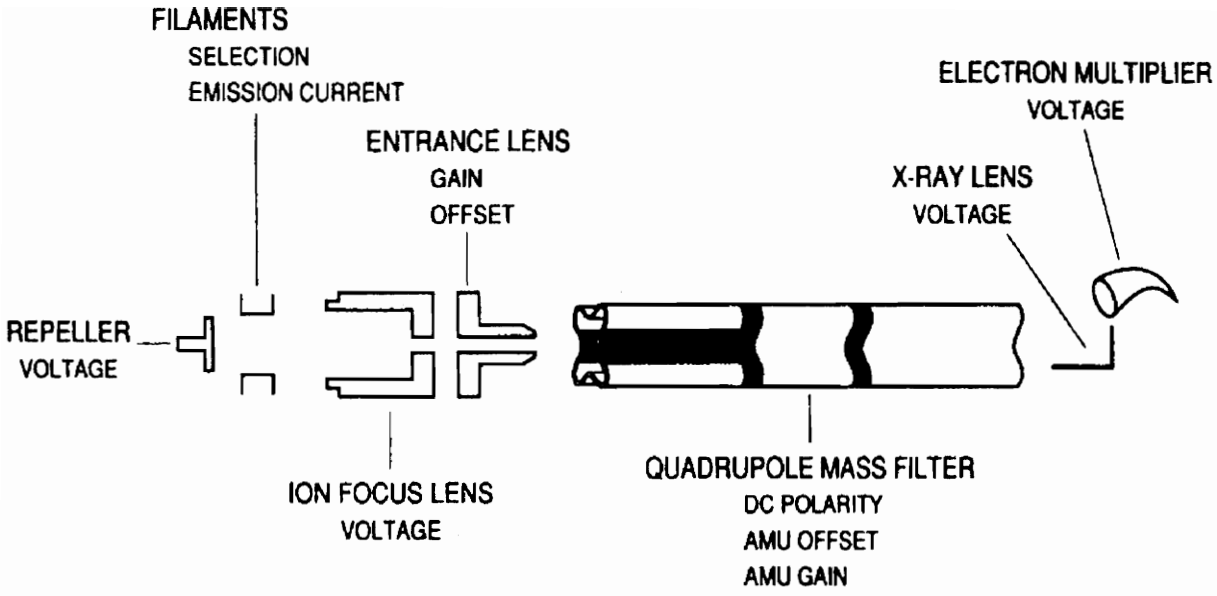


FIGURE 5: Parts Affected by Tuning[17]

Analytical Balance

All weighings were done on an electronic Mettler (Highstown, NJ) model AE250 analytical balance. It had a range from 0 to 205 g with a readability of 0.001 g. In addition, this balance was equipped with a built-in 100 g calibration weight and had user selectable integration time of 6 seconds and stability selection (1, [great sensitivity which caused a long pause before data was readable]). All sand samples were accurately weighed into the Teflon® liner of the extraction vessel, or beakers.

Chemicals, Solutions, and Supplies

Analytical reference standards of 11 phenols and 16 polynuclear aromatic hydrocarbons [see table 3 for the compound lists] were purchased as composite solutions, at a concentration of 2 mg/mL per compound, from Supelco, Inc. (Bellefonte, PA). The purity of these compounds was higher than 97%. The working calibration standards were prepared by serial dilution of the composite stock solutions. The solvents used in this work were HPLC grade hexane *OPTIMA*® and chloroform purchased from Fisher Scientific (Fair Lawn, NJ), as well as HPLC grade acetone from EM Science (Gibbstown, NJ). Anhydrous sodium sulfate from Mallinckrodt Chemical Inc. (Paris, KY) was used as the drying-agent in both the extraction and concentration steps. The liquid chromatograph sample cleanup apparatus, the Kuderna-Danish sample concentrator, and the Soxhlet apparatus were all supplied through Supleco, Inc.

TABLE 3: Compounds Obtained as Solutions in Methylene Chloride

Compounds	Percent Purity (%)	Weight Concentration (µg/L)	% Standard Deviation (**)
Pentachlorophenol	99.0	2000.0	± 10.0
Phenol	99.0	2000.0	± 53.0
2-Chlorophenol	98.9	1999.0	± 46.0
2-Methyl-4,6-Dinitrophenol	99.0	2001.0	± 10.0
2-Nitrophenol	99.0	1999.0	± 27.0
2,4-Dichlorophenol	99.0	2000.0	± 28.0
2,4-Dimethylphenol	99.0	2000.0	± 26.0
2,4-Dinitrophenol	97.9	2000.1	± 15.0
2,4,6-Trichlorophenol	99.0	1999.0	± 19.0
4-Chloro-3-Methylphenol	99.0	2002.0	± 21.0
4-Nitrophenol	99.0	2001.0	± 17.0
Acenaphthene	98.6	2000.2	± 20.7
Acenaphthylene	99.0	2000.0	± 1.7
Anthracene	99.0	2000.0	± 2.8
Benzo (a) anthracene	99.0	2000.2	± 17.6
Benzo (a) pyrene	99.0	2000.2	± 27.4
Benzo (b) fluoranthene	99.0	2000.0	± 12.6
Benzo (g,h,i) perylene	99.0	1999.7	± 21.0
Benzo (k) fluoranthene	99.0	2000.5	± 21.8
Chrysene	99.0	2000.2	± 12.6
Dibenzo (a,h) anthracene	99.0	2000.0	± 2.7
Fluoranthene	98.5	2000.5	± 4.5
Fluorene	99.0	2000.0	± 15.1
Indeno (1,2,3-cd) pyrene	99.0	1999.7	± 21.2
Naphthalene	99.0	2000.0	± 23.0
Phenanthrene	99.0	2000.0	± 3.8
Pyrene	98.6	2000.2	± 15.5

(**) Specified by supplier.

as was the glass wool. In addition, Supleco was the supplier of 100 mL filtration flask and porcelain bruckner funnels. Cellulose extraction thimbles (33mm x 94mm) and glass microfiber filters GF/A (70mm) were purchased through Whatman (Maidstone, England).

Microwave Sample Preparation System

The microwave system used was a CEM Corporation (Matthews, NC) model MES-1000 (see figure 6). It was equipped with an inboard pressure and fiberoptic temperature control system and had a power range from 0-950 ± 50 watts in one percent increments. This instrument has been specifically designed for use with organic solvents: all ignition sources have been eliminated from the microwave cavity. The cavity is Teflon® lined and additional Teflon® has been added to the cavity ceiling. A frequency of 2450 MHz was reached at full power. Temperature and pressure control set points could be programmed in five separate heating stages. Extraction temperatures could be selected from 20 ^{0}C to 200 ^{0}C in 1 ^{0}C increments. The instrument controlled either pressure or temperature, depending on which parameter reached its control set point first. Temperature and pressure data can be sent to an external printer or downloaded to a PC. A control vessel (see figures 7 and 8) was used to measure both temperature and pressure. The extraction vessels are double-walled vessels specifically adapted for use with organic solvents. The outer vessel body and cap were comprised of microwave-transparent Ultem®

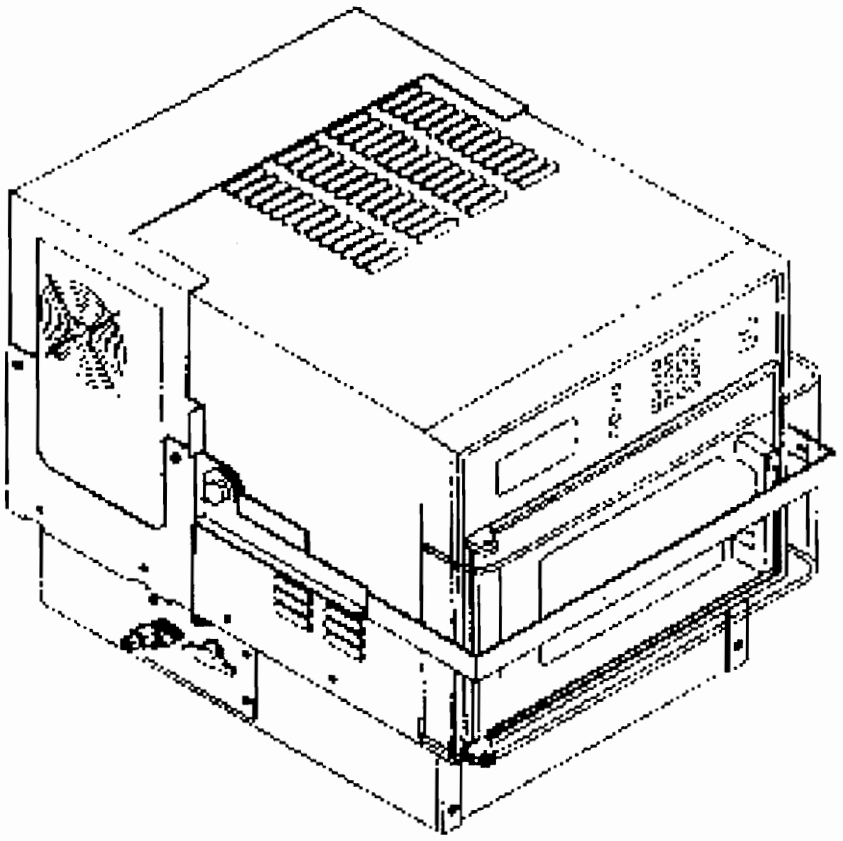


FIGURE 6: MES-1000 Microwave solvent extraction system[18]

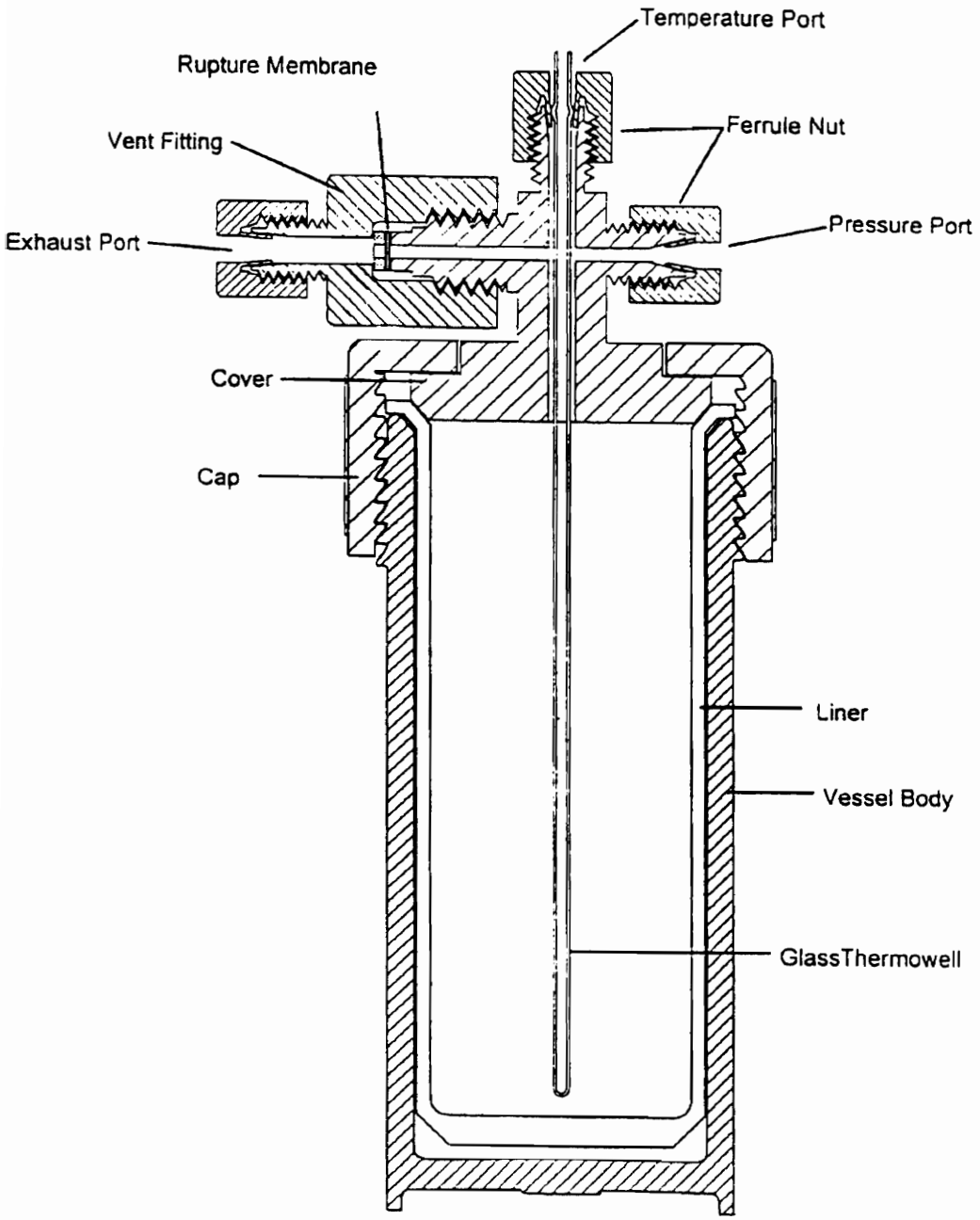


FIGURE 7: Lined Extraction Vessel for Temperature and Pressure Control[18]

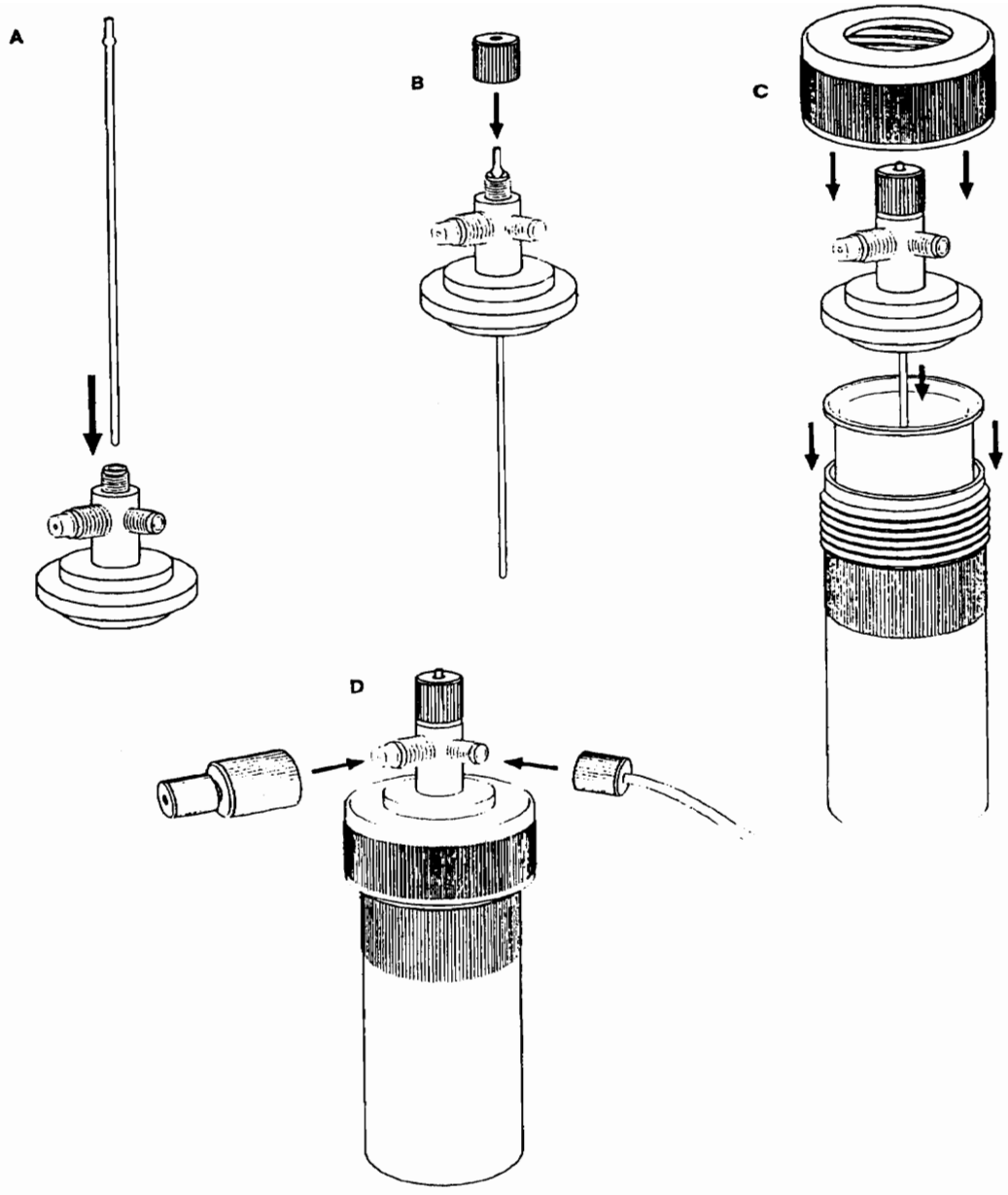


FIGURE 8: Assembly of Lined Extraction Vessel for Temperature and Pressure Control[18]

poly(ether imide). The removable inner liner, the liner cover, and safety rupture membrane were made of Teflon PFA®. Gases could escape through the exhaust port in the event the safety membrane ruptured. The liner cover had Teflon PFA® fittings to allow for pressure tubing connections and insertion of a Pyrex tube through the cap to the bottom of the vessel. This Pyrex tube housed the fiberoptic probe (see figure 9) and protected it form solvent exposure. The standard lined extraction vessels (See Figures 10 and 11) did not have temperature and pressure ports but only a rupture membrane and vent stem. These vessels were rated for a maximum operating temperature of 200 ^{0}C, a maximum operating pressure of 200 psi, and a 100 mL volume. Thus, a maximum of 12 samples could be extracted simultaneously.

Safety features of the microwave system are intended to prevent ignition of flammable and explosive solvents (see figure 12). These safety features include a solvent vapor detector in the system air exhaust; it turns off the microwave magnetron if solvent vapors are detected. An exhaust blower continually moves air through the cavity. In case of exhaust blower failure or blockage, an airflow switch turns off the magnetron. All extraction vessels are connected to a sealed-center collection vessel so that in the event of a safety membrane rupture, solvent vapors will be contained. These vapors are then directed from the cavity through a venturi exhaust tube and routed to the external exhaust. The venturi in the cavity exhaust creates a slight vacuum,

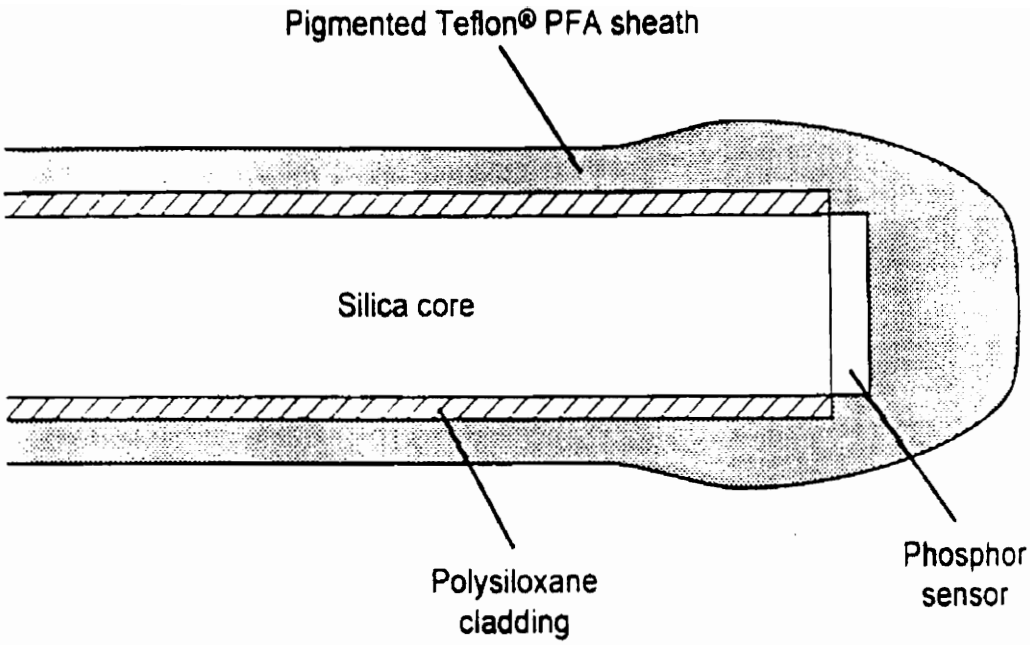


FIGURE 9: Fiber Optic Thermometry Probe[18]

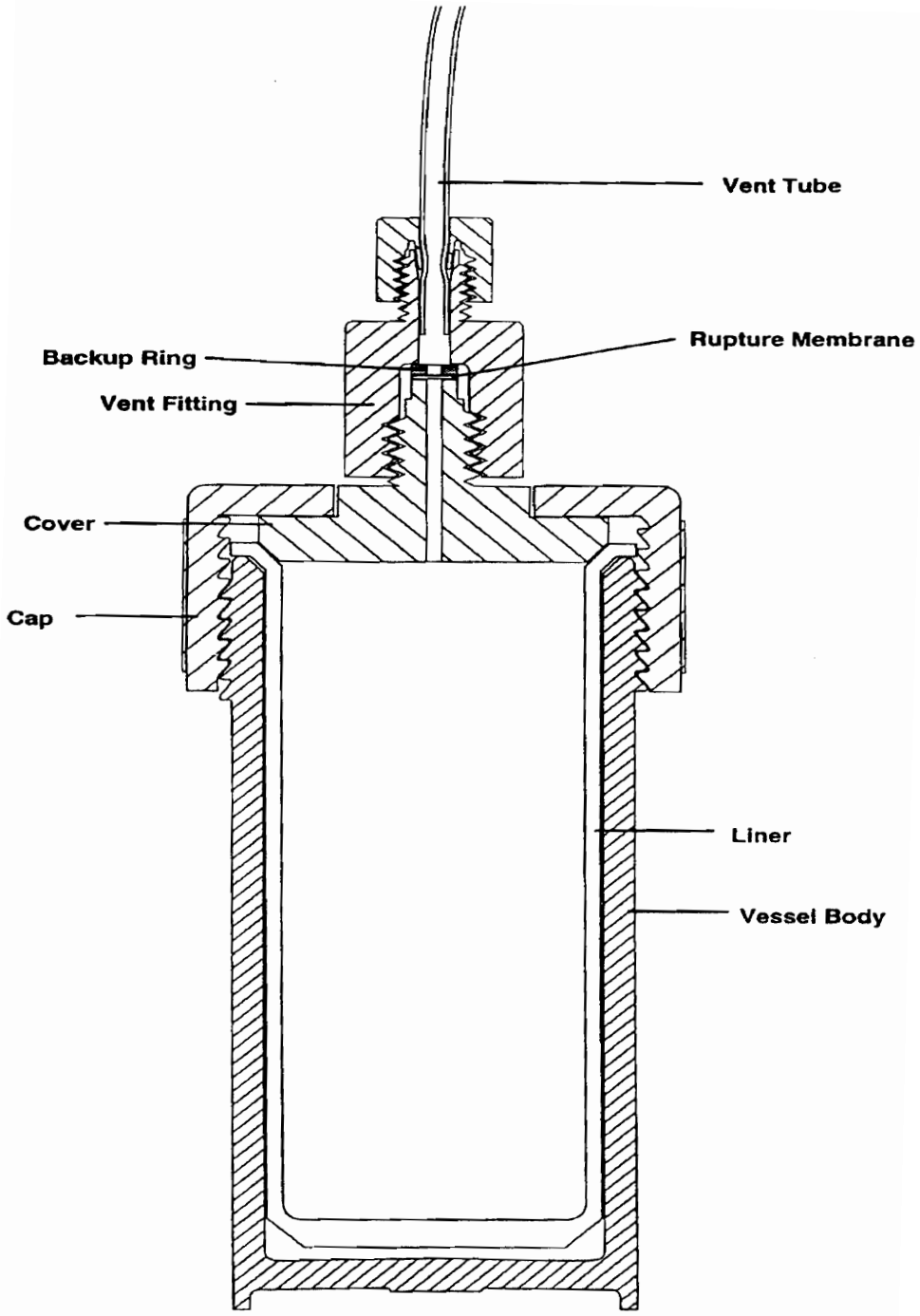


FIGURE 10: Lined Extraction Vessel[18]

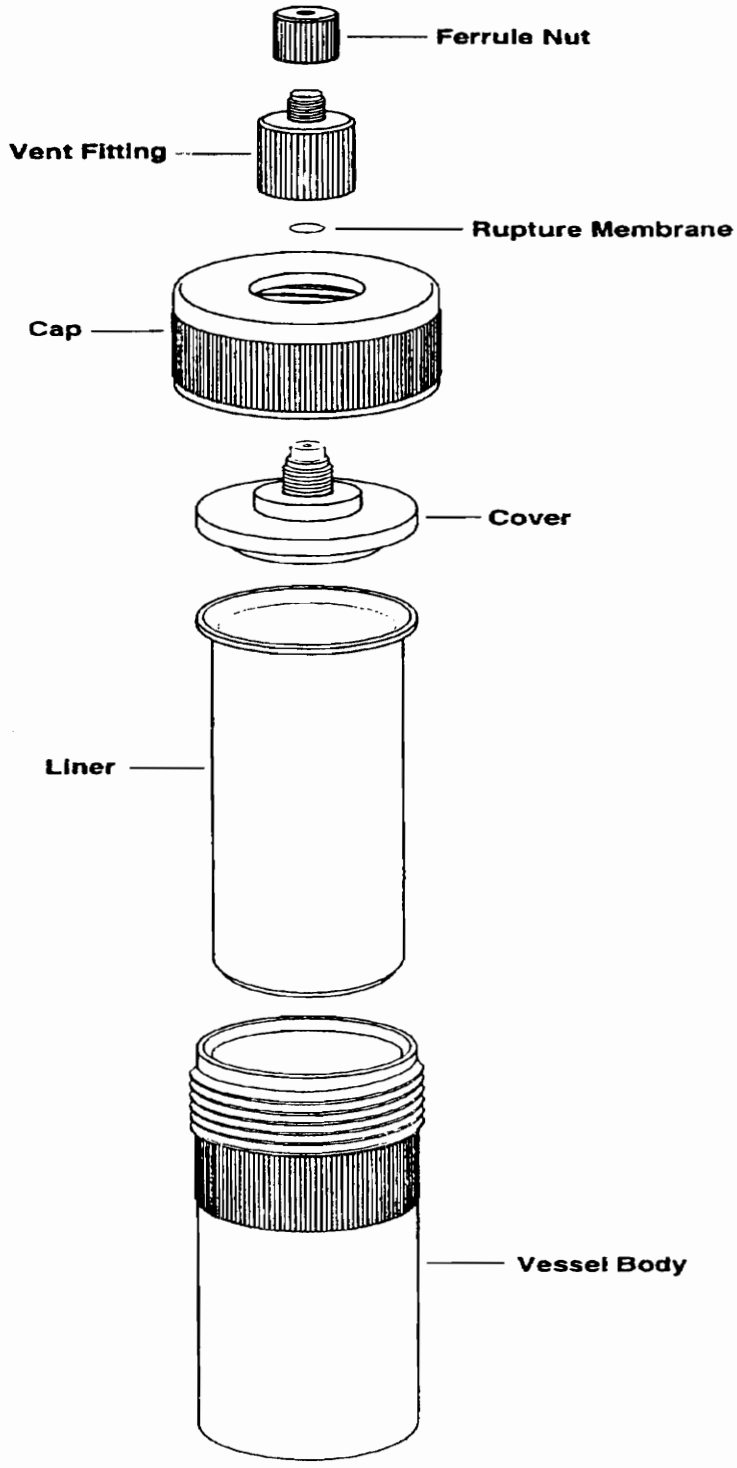


FIGURE 11: Lined Extraction Vessel (Exploded View) [18]

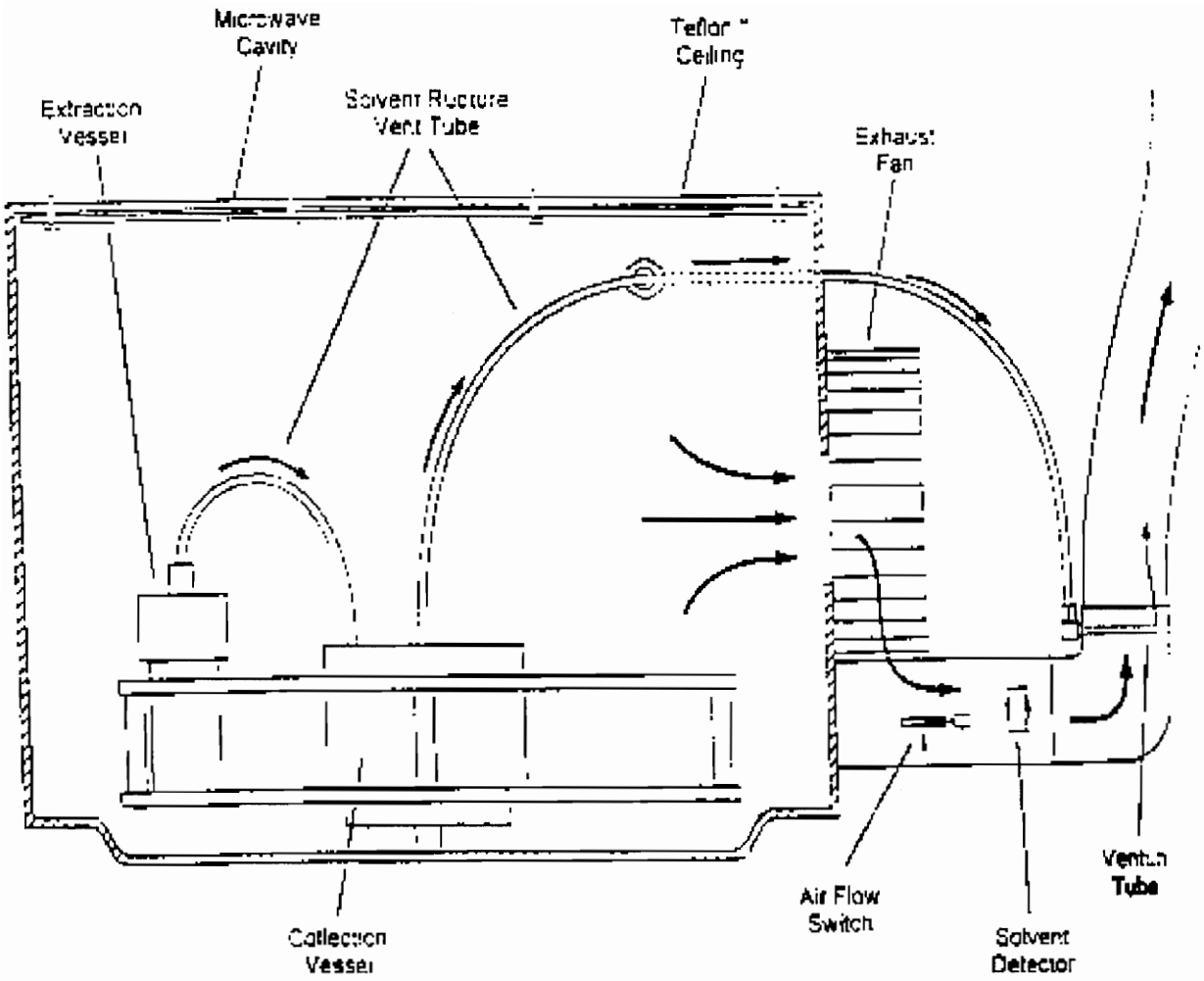


FIGURE 12: Safety Features of microwave solvent extraction system[10]

which helps to remove solvent vapors.

Consumables

The carrier gas used with the GC/MS system was Airco (Murray Hill, NJ) grade 5.0 (99.999%) Helium and was purified using a Supelco, Inc. heated gas purifier prior to entering the GC system. The consumables used by the GC/MS included LB-2 thermogreen® septa and M-2A graphite/vespel (40%/60%) ferrules both purchased from Supelco, Inc. A Hamilton (Reno, NV) model #701 10 µL syringe purchased from Supelco, Inc. was used for automatic injection into the GC/MS.

Standard Calibration

All solutions were prepared using standard analytical techniques. Calibration standards at 20, 50, and 100 µg/L were prepared by serial dilution with chloroform of the composite stock solutions. Dilute solutions were prepared directly into HP autosampler vials. These autosampler vials were sealed with Kimble® brand aluminum crimp top seals fitted with PTFE silicone septa obtained from Hewlett-Packard. These solutions were then subsequently analyzed via GC/MS and a reference curve established. A "global" database was setup in the data analysis window of the HP ChemStation software. This database contained all 27 compounds, their relative retention times, and three point calibration curves. A calibration sample of 50 µg/L was run daily to ensure

curve linearity. The daily calibration was plotted against this database as were the blank and spiked samples. Eppendorf Pipettes™, made by Brinkmann Instruments, Inc. (Westbury, NY) and purchased through Fisher Scientfic, were used to measure minute solution transfer. Two different sizes were used, having ranges of 10 to 100 µL and 100 to 1000 µL. Eppendorf dispoable tips, purchased through Fisher, were attached to these pipettes.

Microwave-assisted Extraction Procedure

BLANK

Triplicate sets of 5 g portions of sand and 5 g portions of sodium sulfate (Na_2SO_4) were accurately weighed into the Teflon® liners of the extraction vessels and mixed using a glass stirring rod. A 30 mL volume of either acetone, chloroform, or 1:1 chloroform/hexane was added to these liners. After installing a new rupture membrane, the extraction vessel was closed. The extraction procedure was performed at 145 ^{0}C for 15 min. at 100% power. After extraction, the vessels were allowed to cool to room temperature for ~30 min. before they were opened. The supernatant liquid was filtered through a micro-filtration apparatus. Filtrates were collected in a filtration flask, transfered to a glass bottle, and combined with two 5 mL solvent rinses of the extraction liner, two 5 mL solvent rinses of the filter, a one 5 mL solvent rinse of the filtration flask. These extracts were then concentrated to 1 mL for GC/MS analysis by the K-D method.

SPIKED SAMPLES

Triplicate sets of 5 g portions of sand were accurately weighed into the Teflon® liners of the extraction vessels. Two 25 µL aliquots of the composite standard solution for both the phenols and PAHs were added directly to the 5 g of sand in the extraction liners, and allowed to age for 7 days while stored in a sealed extraction vessel. After seven days, 5 g samples of sodium sulfate were added to each liner and stirred with a glass stirring rod. A 30 mL volume of either acetone, chloroform, or 1:1 chloroform/hexane was added to these liners. After installing a new rupture membrane, the extraction vessel was closed. The extraction procedure was performed at 145 ^{0}C for 15 min. at 100% power. After extraction, the vessels were allowed to cool to room temperature for ~30 min. before they were opened. The supernatant liquid was filtered through a micro-filtration apparatus. Filtrates were collected in a filtration flask, transfered to a glass bottle, and combined with three 5 mL solvent rinses of the extraction liner, sample and filtration flask. These extracts were then concentrated to 1 mL for GC/MS analysis.

Soxhlet Extraction Procedure (EPA Method 3540B)

BLANK

Duplicate sets of 5 g portions of sand and 5 g portions of sodium sulfate were weighed in into a 100 mL beaker. (The EPA method states 10 g portions of

each, but these weights were reduced to match those of the microwave extraction vessel). These portions were then stirred using a glass stirring rod, added to a solvent pre-rinsed cellulose thimble, and placed inside the Soxhlet apparatus (see figure 13). A few boiling chips were added to the round-bottom flask as was 200 mL of the appropriate solvents (acetone, chloroform, or 1:1 chloroform/hexane). The entire apparatus was assembled, the water turned on, and the sample extracted for 24 hours at 4-6 cycles per hour. After 24 hours had elapsed, the extract was cooled and removed. It was then concentrated to 1 mL for GC/MS analysis.

SPIKED SAMPLES

Duplicate sets of 5 g portions of sand were weighed in duplicate into a 100 mL beaker. These samples were then transferred to a pre-rinsed cellulose thimble, two 25 µL aliquots of the composite standard solution for both phenols and PAHs were added directly to the sand in the cellulose extraction thimbles and the extraction vessels were sealed for 7 days. After seven days, 5 g portions of sodium sulfate were added, mixed using a glass stirring rod, and placed inside the Soxhlet apparatus. A few boiling chips were added to the round-bottom flask, as was 200 mL of the appropriate solvent. The entire hours apparatus was assembled, the water turned on, and the sample extracted for 24 at 4-6 cycles per hour. After 24 hours had elapsed, the extract was cooled and removed. It was then concentrated to 1 mL for GC/MS analysis.

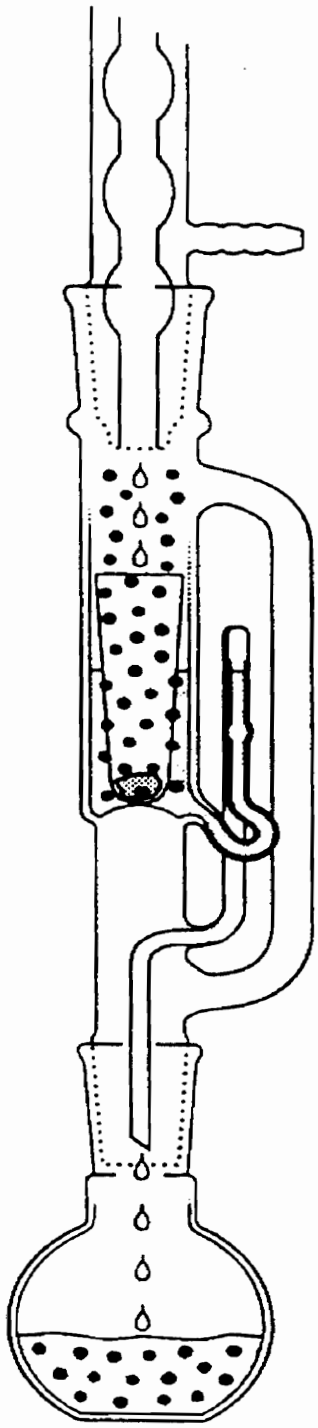


FIGURE 13: Soxhlet Apparatus[9]

Concentration Procedure

To the extract from either extraction step a small amount of sodium sulfate was added to remove water. The extract was poured into a liquid chromatography clean-up column containing a plug of glass wool and more sodium sulfate. The extract dripped into a Kuderna-Danish concentrator minus its 3-ball Snyder column. After all of the extract passed through the clean-up column, a 50 mL wash with the appropriate solvent was added to the collection flask, and this wash was added to the column. When all of the solution had finished dripping, the clean-up column was removed and the 3-ball Snyder column re-attached. The K-D apparatus was placed in boiling water until the solution had a final volume of approximately 1 mL. The K-D was allowed to cool for ~ 15 minutes before removing the 3-ball column. Three washes of the inside of the K-D were made. Finally the K-D apparatus was retrofitted with its micro setup. The K-D was placed in the boiling water until a volume of 1 mL was reached. The K-D was removed and allowed to cool. The solution housed in the receiving vessel was drawn up and put into an autosampler vial, labeled accordingly, and put in the refrigerator until analysis.

GC/MS Analysis

A HP 7673 Autosampler was used to inject the standards, blanks, and spikes. For the standards, three replicates were analyzed for each calibration point. The area counts for these three were averaged and added into the

"global" database. Blanks and spikes were analyzed in triplicate and plotted versus the calibration curve. A 50 ng/µL standard was analyzed in triplicate with each batch of either spikes of blanks.

RESULTS AND DISCUSSION

Optimizing GC conditions

The phenolic and polynuclear aromatic compounds are present on the EPA's Priority Pollutant Organics list and, therefore, have severe environmental ramifications. Methods for analyzing these two compound groups have already been developed by the EPA. Following the guidelines for method 8270B in SW-846, only slight modifications were made. The injector port was raised 25 ^{0}C after observing that the peak shapes and area counts were sharper and larger for phenols at this higher temperature. In addition, the initial oven temperature was elevated and the ramp rate changed to shorten analysis time. Generally in capillary GC, 95% of the sample is transfered to the column in the early stages of splittless injection. A splittless time of 0.15 minutes was chosen to minimize the possiblity of thermal decomposition in the hot injector port. The transfer line, which also helps resolve peak shape, was set at 300 ^{0}C to prevent condensation. Prior to establishing calibration curves, the sand was analyzed using both Soxhlet and MAE techniques to provide background interference information (see figures 14A&B - 16). This helped to establish the solvent choice that had the least interference pattern. By comparing these

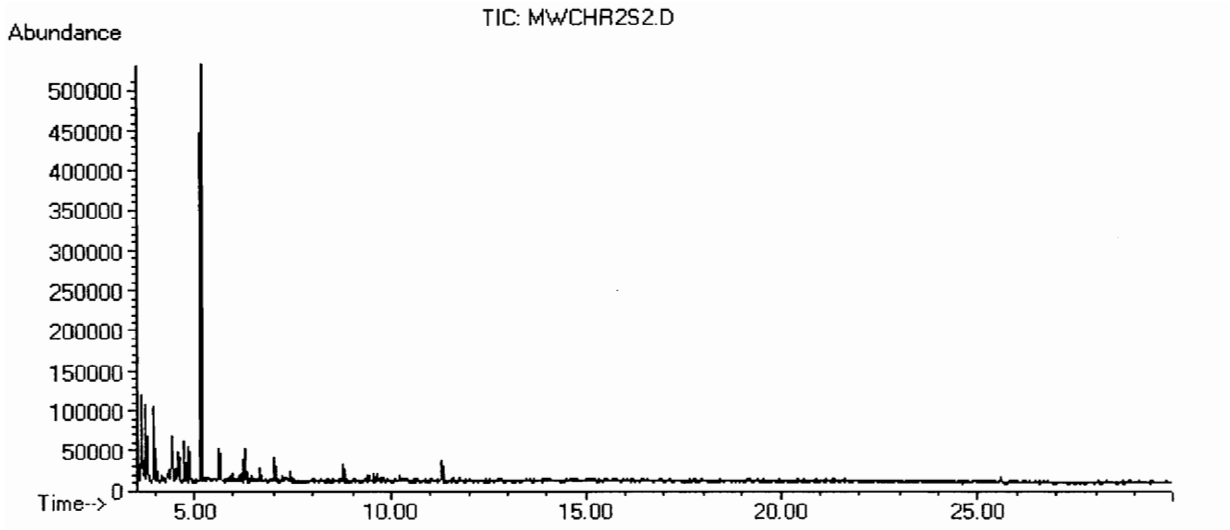


FIGURE 14A: Microwave Background for Chloroform/Hexane

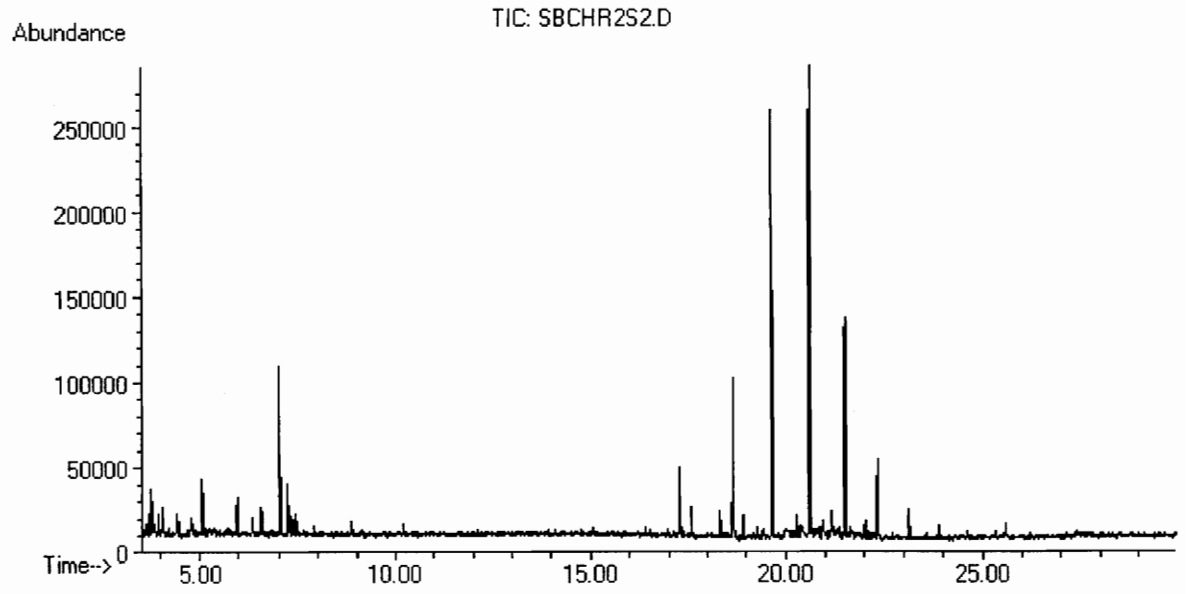


FIGURE 14B: Soxhlet Background for Chloroform/Hexane

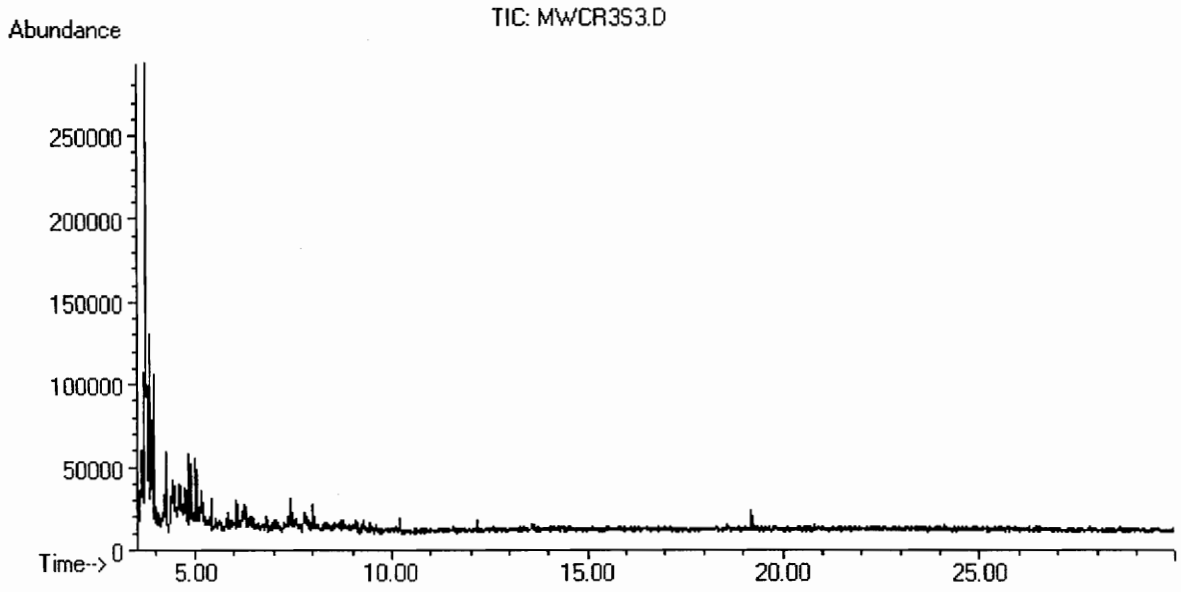


FIGURE 15A: Microwave Background for Chloroform

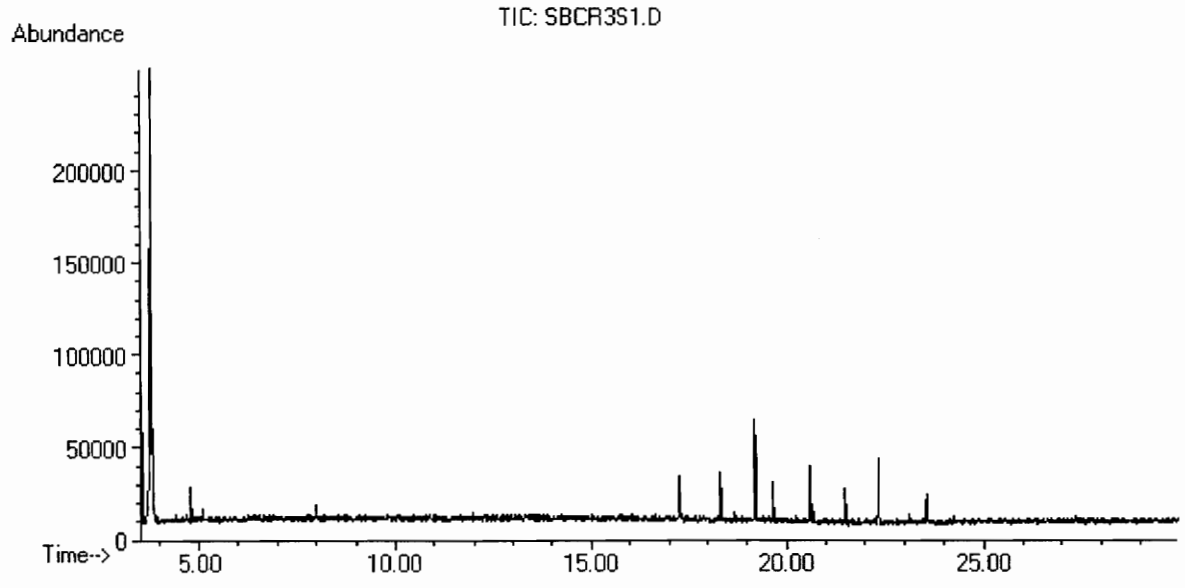


FIGURE 15B: Soxhlet Background for Chloroform

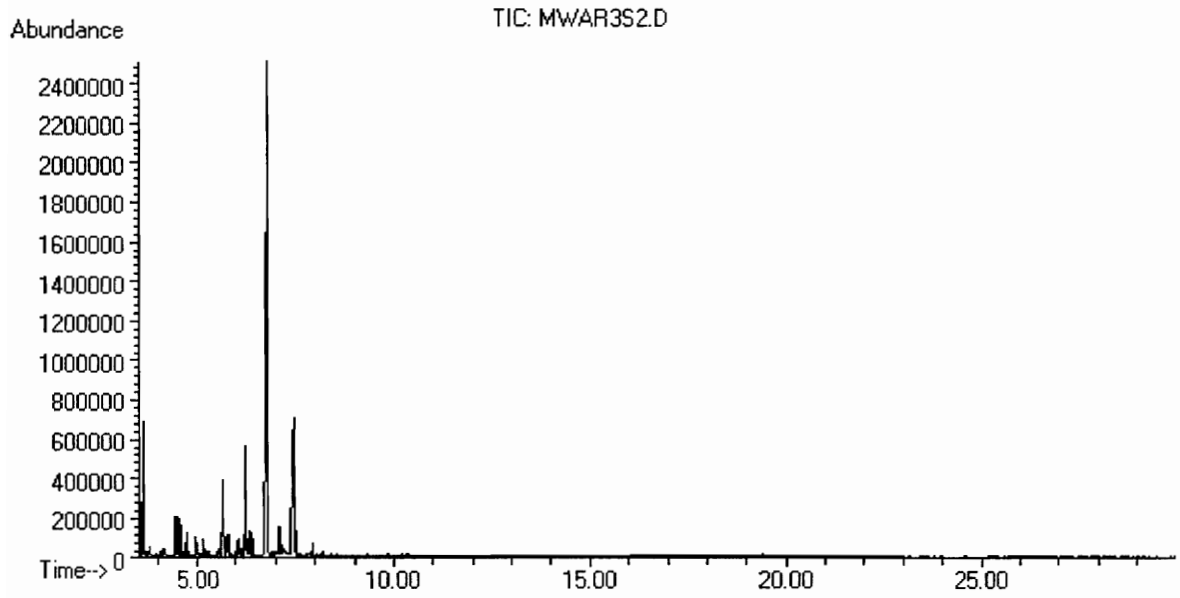


FIGURE 16: Microwave Background for Acetone

chromatograms, it is evident that chloroform is the best solvent of choice for both extraction methods. Acetone was not analyzed via Soxhlet because of the large interferring peak (2-Methyl-2-nonen-4-one) that co-eluted with peak number 4, 2,4-Dimethylphenol. By executing the GC/MS conditions developed, full chromatographic separation of all 27 compounds was achieved in under 30 minutes (see figure 17 and table 4).

Mass Spectrometer Detector

Standard operating procedure followed in SW-846 protocol requires mass spectrometers to operate continously with an electron energy of 70 eV. The electron multiplier voltage was the only allowed variable and was maintained at 1800 V in this work. The instrument was always tuned with PFTBA prior to analysis. Via GC/MS, identity was established for each of the peaks (see appendix A).

Calibration

Standards of phenols and PAHs were prepared by diluting each 2 mg/mL composite solution in chloroform to 20, 50, and 100ppm (see figures 18-20). These three solutions were then injected three times each into the GC, alternating each concentration level to incorporate as much error as possible (see tables 5-7). The chromatograms were integrated in the chemstation and the area counts were averaged in MS Excel and added into the "global"

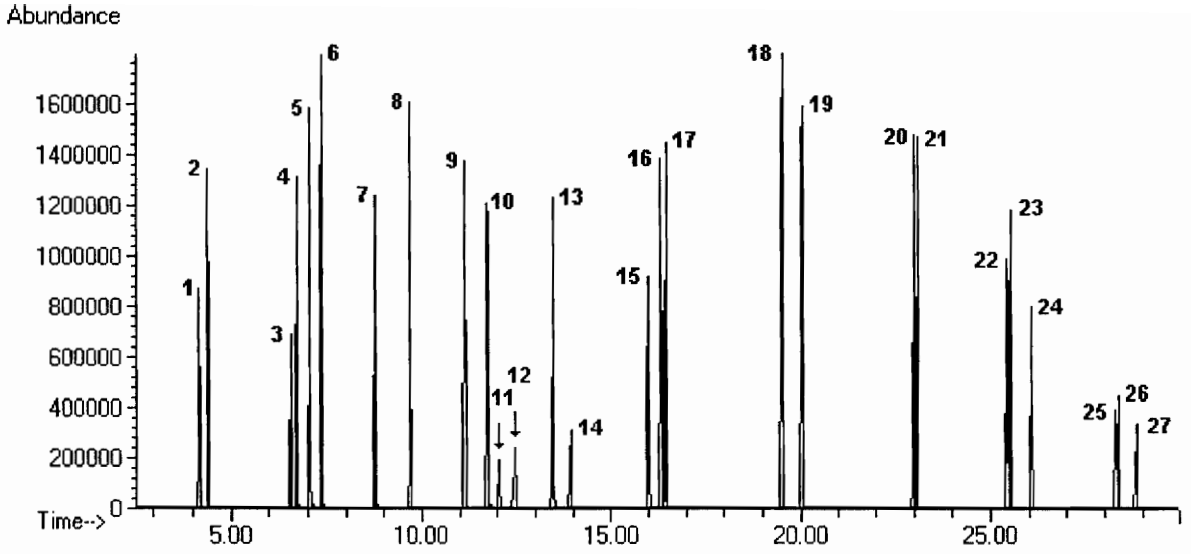


FIGURE 17: Typical Standard Chromatogram

TABLE 4: Average Retention Times over all Standard Levels

Peak Number	Compound Name	Average Retention Time (minutes)	Peak Number	Compound Name	Average Retention Time (minutes)
1	Phenol	4.175	15	Pentachlorophenol	15.981
2	2-Chlorophenol	4.399	16	Phenanthrene	16.323
3	2-Nitrophenol	6.556	17	Anthracene	16.451
4	2,4-Dimethylphenol	6.712	18	Fluoranthene	19.490
5	2,4-Dichlorophenol	7.051	19	Pyrene	20.022
6	Naphthalene	7.354	20	Chrysene	22.997
7	4-Chloro-3-methylphenol	8.754	21	Benzo (a) anthracene	23.099
8	2,4,6-Trichlorophenol	9.685	22	Benzo (b) fluoranthene	25.421
9	Acenaphthylene	11.128	23	Benzo (k) fluoranthene	25.483
10	Acenaphthene	11.726	24	Benzo (a) pyrene	26.062
11	2,4-Dinitrophenol	12.013	25	Indeno (1,2,3-cd) pyrene	28.270
12	4-Nitrophenol	12.441	26	Dibenzo (a,h) anthracene	28.343
13	Fluorene	13.464	27	Benzo (g,h,i) perylene	28.824
14	2-Methyl-4,6-dinitrophenol	13.909			

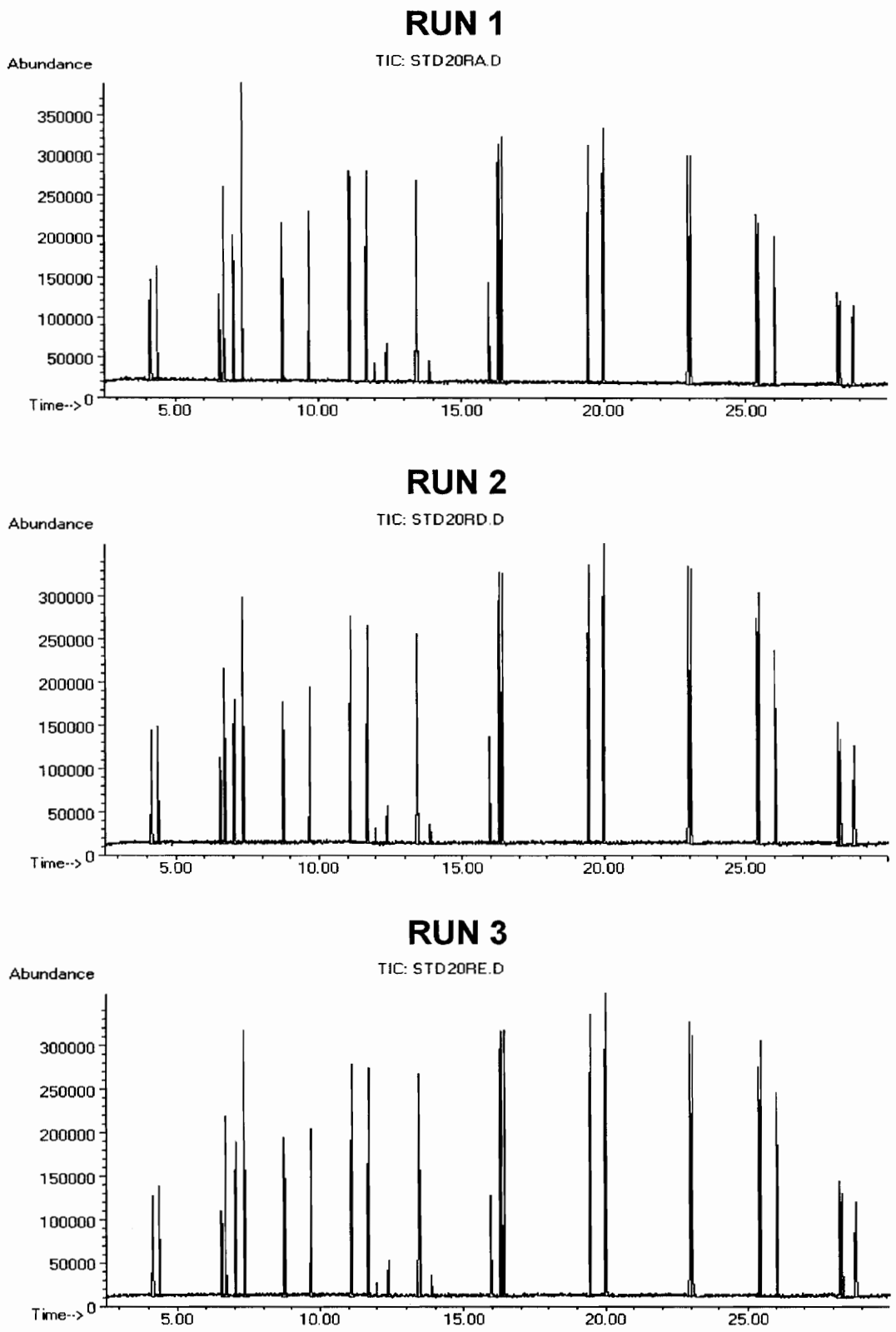


FIGURE 18: Triplicate Analyses of the 20 ng/µL Standard
(Elution order corresponds to Figure 17)

TABLE 5: Area Counts for the triplicate 20 ng/µL Standards

No.	Retention Time (min.)	Compound Names				Mean	S.D.	% RSD
1	4.164	Phenol	2461163	2118489	2089427	*2223026*	*206744*	**9.3**
2	4.394	2-Chlorophenol	1870229	2171067	2186547	*2075948*	*178326*	**8.6**
3	6.546	2-Nitrophenol	1753892	1642618	1566920	*1654477*	*94048*	**5.7**
4	6.698	2,4-Dimethylphenol	3578790	3125468	3304354	*3336204*	*228333*	**6.8**
5	7.040	2,4-Dichlorophenol	2824469	2560639	2693112	*2692740*	*131915*	**4.9**
6	7.343	Naphthalene	5428259	4591944	5211323	*5077175*	*433996*	**8.5**
7	8.740	4-Chloro-3-methylphenol	2774620	2526882	2789707	*2697070*	*147580*	**5.5**
8	9.675	2,4,6-Trichlorophenol	3106270	2642010	3037286	*2928522*	*250513*	**8.6**
9	11.113	Acenaphthylene	5367461	4995288	5372678	*5245142*	*216396*	**4.1**
10	11.707	Acenaphthene	5740483	5248508	5610440	*5533144*	*254933*	**4.6**
11	11.984	2,4-Dinitrophenol	210919	208994	176887	*198933*	*19117*	**9.6**
12	12.395	4-Nitrophenol	916885	1024908	1100471	*1014088*	*92270*	**9.1**
13	13.443	Fluorene	5462746	5290776	5664280	*5472601*	*186947*	**3.4**
14	13.879	2-Methyl-4,6-dinitrophenol	411821	407792	347256	*388956*	*36170*	**9.3**
15	15.965	Pentachlorophenol	2342073	2240602	2172873	*2251849*	*85159*	**3.8**
16	16.301	Phenanthrene	5720544	5833797	5784450	*5779597*	*56782*	**1.0**
17	16.427	Anthracene	5587144	5579577	5539616	*5568779*	*25538*	**0.5**
18	19.472	Fluoranthene	5668987	6379586	6352945	*6133839*	*402794*	**6.6**
19	20.003	Pyrene	5636173	6273777	6230495	*6046815*	*356284*	**5.9**
20	22.980	Chrysene	5126091	5620766	5691179	*5479345*	*307946*	**5.6**
21	23.076	Benzo (a) anthracene	4811395	5390418	5255966	*5152593*	*303037*	**5.9**
22	25.403	Benzo (b) fluoranthene	4363601	5032668	5190411	*4862227*	*438966*	**9.0**
23	25.460	Benzo (k) fluoranthene	4098573	4789514	4901210	*4596432*	*434761*	**9.5**
24	26.042	Benzo (a) pyrene	3480791	4012580	4099964	*3864445*	*335115*	**8.7**
25	28.247	Indeno (1,2,3-cd) pyrene	3600488	3360868	3319156	*3426837*	*151825*	**4.4**
26	28.320	Dibenzo (a,h) anthracene	3249037	2985370	2866331	*3033579*	*195855*	**6.5**
27	28.794	Benzo (g,h,i) perylene	2597913	3153388	2975534	*2908945*	*283661*	**9.8**
		Data File:	**STD20RA**	**STD20RD**	**STD20RE**			

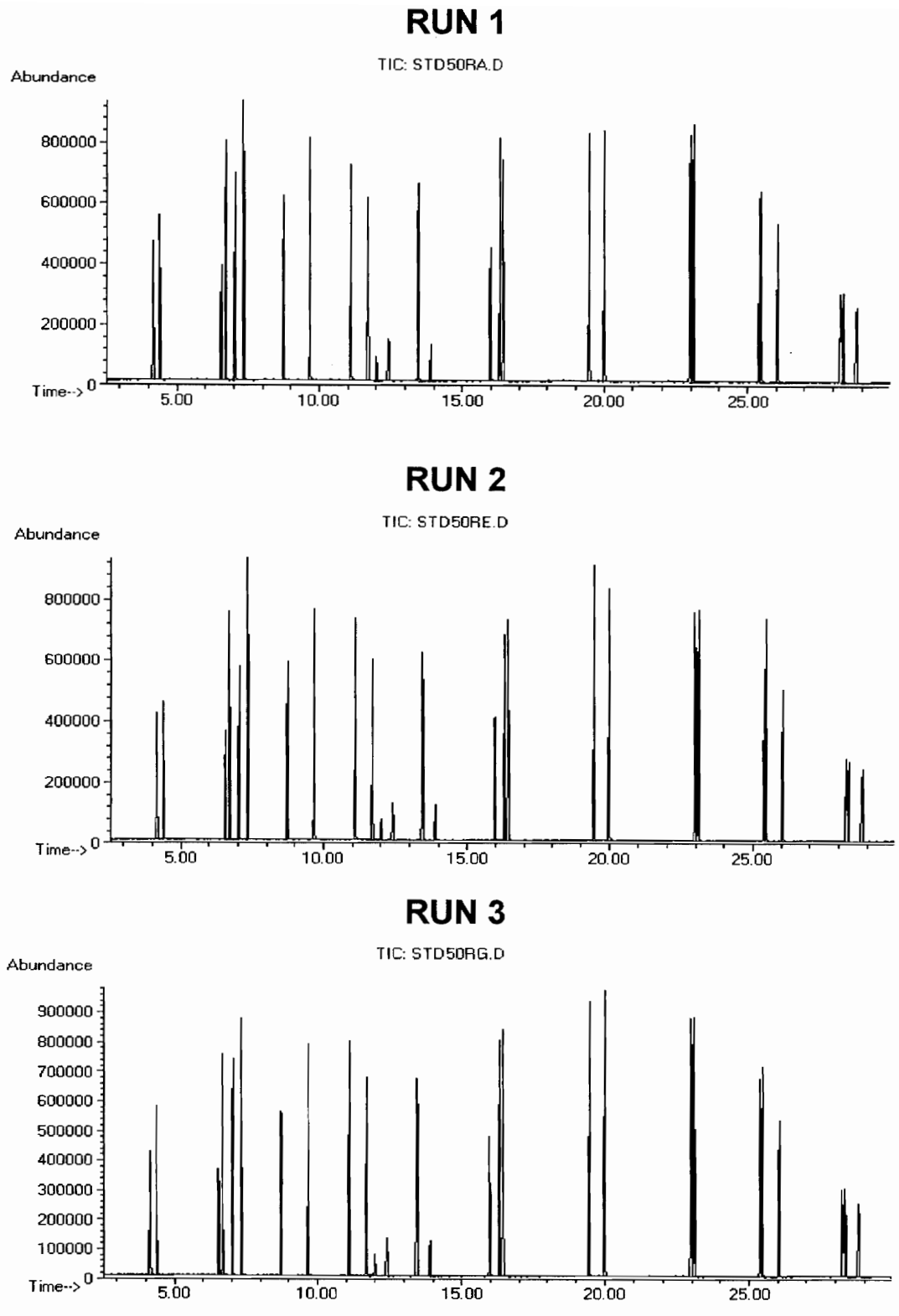


FIGURE 19: Triplicate Analyses of the 50 ng/µL Standard
(Elution order corresponds to Figure 17)

TABLE 6: Area Counts for the triplicate 50 ng/µL Standards

No.	Retention Time (min.)	Compound Names				Mean	S.D.	% RSD
1	4.175	Phenol	8037791	7006424	7576860	*7540358*	*516651*	**6.9**
2	4.399	2-Chlorophenol	8984715	8036604	8838918	*8620079*	*510536*	**5.9**
3	6.554	2-Nitrophenol	6280751	5723602	5959030	*5987794*	*279686*	**4.7**
4	6.710	2,4-Dimethylphenol	12270178	11607267	12063470	*11980305*	*339190*	**2.8**
5	7.049	2,4-Dichlorophenol	11036534	9860647	11001216	*10632799*	*668936*	**6.3**
6	7.351	Naphthalene	14783377	13380918	14842399	*14335565*	*827275*	**5.8**
7	8.751	4-Chloro-3-methylphenol	10227534	9425074	10373565	*10008724*	*510703*	**5.1**
8	9.682	2,4,6-Trichlorophenol	11867110	11110398	12309148	*11762219*	*606219*	**5.2**
9	11.124	Acenaphthylene	14729631	14099063	15953741	*14927478*	*943035*	**6.3**
10	11.722	Acenaphthene	15265710	14682949	16026844	*15325168*	*673918*	**4.4**
11	12.008	2,4-Dinitrophenol	1611232	1412707	1424397	*1482779*	*111397*	**7.5**
12	12.439	4-Nitrophenol	4294234	3879878	4137270	*4103794*	*209197*	**5.1**
13	13.459	Fluorene	14948698	14090607	15463342	*14834216*	*693491*	**4.7**
14	13.903	2-Methyl-4,6-dinitrophenol	2658379	2499589	2952107	*2703358*	*229588*	**8.5**
15	15.978	Pentachlorophenol	10003210	9352647	10109094	*9821650*	*409605*	**4.2**
16	16.319	Phenanthrene	16310673	15710028	17069549	*16363417*	*681293*	**4.2**
17	16.447	Anthracene	15772086	15049851	16096362	*15639433*	*535718*	**3.4**
18	19.486	Fluoranthene	17559849	17172655	18830881	*17854462*	*867482*	**4.9**
19	20.018	Pyrene	17443005	17304486	19031965	*17926485*	*959875*	**5.4**
20	22.994	Chrysene	16450345	16241240	17810166	*16833917*	*851897*	**5.1**
21	23.097	Benzo (a) anthracene	15627049	15164644	16786707	*15859467*	*835635*	**5.3**
22	25.419	Benzo (b) fluoranthene	13697779	13863128	15092804	*14217904*	*762183*	**5.4**
23	25.482	Benzo (k) fluoranthene	13256956	13114185	14892774	*13754638*	*988236*	**7.2**
24	26.060	Benzo (a) pyrene	10977258	10933938	11946313	*11285836*	*572400*	**5.1**
25	28.267	Indeno (1,2,3-cd) pyrene	8295830	7889085	8639425	*8274780*	*375613*	**4.5**
26	28.340	Dibenzo (a,h) anthracene	7096811	6542935	7314342	*6984696*	*397737*	**5.7**
27	28.822	Benzo (g,h,i) perylene	7478071	6867291	7391214	*7245525*	*330427*	**4.6**
		Data File:	**STD50RA**	**STD50RE**	**STD50RG**			

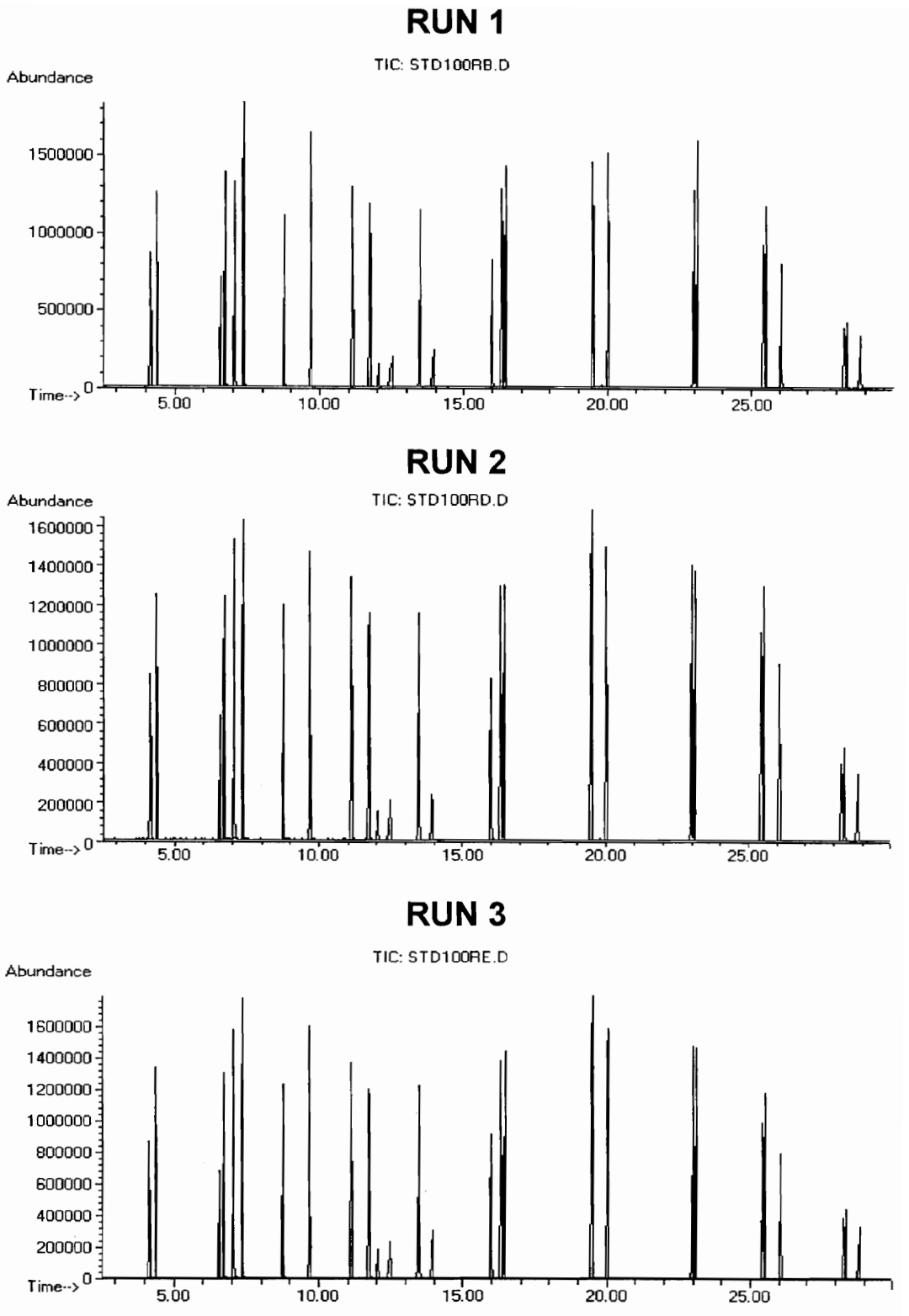


FIGURE 20: Triplicate Analyses of the 100 ng/µL Standard
(Elution order corresponds to Figure 17)

TABLE 7: Area Counts for the triplicate 100 ng/µL Standards

No.	Retention Time (min.)	Compound Names				Mean	S.D.	% RSD
1	4.186	Phenol	15790379	15191404	15743923	*15575235*	*333218*	**2.1**
2	4.405	2-Chlorophenol	19051618	18466990	19705716	*19074775*	*619688*	**3.2**
3	6.568	2-Nitrophenol	13034440	12755771	13692466	*13160892*	*480980*	**3.7**
4	6.727	2,4-Dimethylphenol	25333747	25395960	27045165	*25924957*	*970627*	**3.7**
5	7.065	2,4-Dichlorophenol	24058438	22875771	25699124	*24211111*	*1417855*	**5.9**
6	7.367	Naphthalene	30799267	31018981	33402317	*31740188*	*1443632*	**4.5**
7	8.770	4-Chloro-3-methylphenol	22357308	21910574	24308075	*22858652*	*1274956*	**5.6**
8	9.697	2,4,6-Trichlorophenol	26963943	26274784	28521721	*27253483*	*1151111*	**4.2**
9	11.147	Acenaphthylene	31267837	30265070	33968845	*31833917*	*1915678*	**6.0**
10	11.749	Acenaphthene	31224455	30537464	33658146	*31806688*	*1639790*	**5.2**
11	12.046	2,4-Dinitrophenol	3955048	3726411	3846881	*3842780*	*114374*	**3.0**
12	12.488	4-Nitrophenol	8617098	8073686	9172025	*8620936*	*549180*	**6.4**
13	13.491	Fluorene	30042126	29633836	33242831	*30972931*	*1976363*	**6.4**
14	13.945	2-Methyl-4,6-dinitrophenol	5988736	6200868	6403472	*6197692*	*207386*	**3.3**
15	16.000	Pentachlorophenol	22262185	21675211	24302290	*22746562*	*1378895*	**6.1**
16	16.348	Phenanthrene	32849672	31866645	35632160	*33449492*	*1953104*	**5.8**
17	16.478	Anthracene	31794008	31487879	34880941	*32720943*	*1876865*	**5.7**
18	19.511	Fluoranthene	36291406	36120302	39734906	*37382205*	*2039294*	**5.5**
19	20.045	Pyrene	35109889	35898341	38971289	*36659840*	*2040224*	**5.6**
20	23.018	Chrysene	33418338	33163643	33317829	*33299937*	*128287*	**0.4**
21	23.125	Benzo (a) anthracene	31277298	32760547	32749589	*32262478*	*853208*	**2.6**
22	25.440	Benzo (b) fluoranthene	28003553	29382236	28189607	*28525132*	*748081*	**2.6**
23	25.508	Benzo (k) fluoranthene	28358580	28180801	28799880	*28446420*	*318750*	**1.1**
24	26.083	Benzo (a) pyrene	25003121	24452430	25449791	*24968447*	*499584*	**2.0**
25	28.295	Indeno (1,2,3-cd) pyrene	16977137	17024388	18339732	*17447086*	*773415*	**4.4**
26	28.369	Dibenzo (a,h) anthracene	12299212	12491539	12903020	*12564590*	*308461*	**2.5**
27	28.856	Benzo (g,h,i) perylene	14730717	14436858	13127509	*14098361*	*853524*	**6.1**
		Data File:	**STD100RB**	**STD100RD**	**STD100RE**			

database. Appendix A contains the original chromatograms, area counts, mass spectrums, and calibration curves. Even though these curves contain the correlation coefficients, these have been included in Table 8 for each respectable compound.

Optimizing the Extraction Procedures

Optimal extraction for the Soxhlet method was not a consideration, since EPA guidelines were followed. Referring to Lopez-Avila and Young[16], good recoveries were observed when the microwave operated at 145 ^{0}C for 15 min, using 100% power, and 30 mL of solvent. For this reason, the previous setpoints were used for MAE.

Spiked Sand

After the aging process, extraction in each respective technique was conducted and the extracts were subsequently analyzed. Figures 21 - 25 depict typical chromatograms for each extraction procedure with the appropriate solvent. Upon viewing the percent recovery tables (see tables 9 - 13), it can be concluded that MAE of the spiked sand using chloroform as the solvent yields the best recovery.

TABLE 8: Correlation Coefficients
(Area Counts vs. Concentration)

COMPOUNDS	CORRELATION COEFFICIENT
Phenol	0.999652143
2-Chlorophenol	0.999936139
2-Nitrophenol	0.999998361
2,4-Dimethylphenol	0.999962245
2,4-Dichlorophenol	0.999976937
Naphthalene	0.999511708
4-Chloro-3-methylphenol	0.999902877
2,4,6-Trichlorophenol	0.999910580
Acenaphthylene	0.999924981
Acenaphthene	0.999996604
2,4-Dinitrophenol	0.999673876
4-Nitrophenol	0.999372498
Fluorene	0.999960354
2-Methyl-4,6-dinitrophenol	0.999646152
Pentachlorophenol	0.999979644
Phenanthrene	0.999963899
Anthracene	0.999989245
Fluoranthene	0.999999996
Pyrene	0.999890447
Chrysene	0.999290690
Benzo (a) anthracene	0.999743983
Benzo (b) fluoranthene	0.999732801
Benzo (k) fluoranthene	0.999948125
Benzo (a) pyrene	0.999654263
Indeno (1,2,3-cd) pyrene	0.999459459
Dibenzo (a,h) anthracene	0.998987621
Benzo (g,h,i) perylene	0.999898666

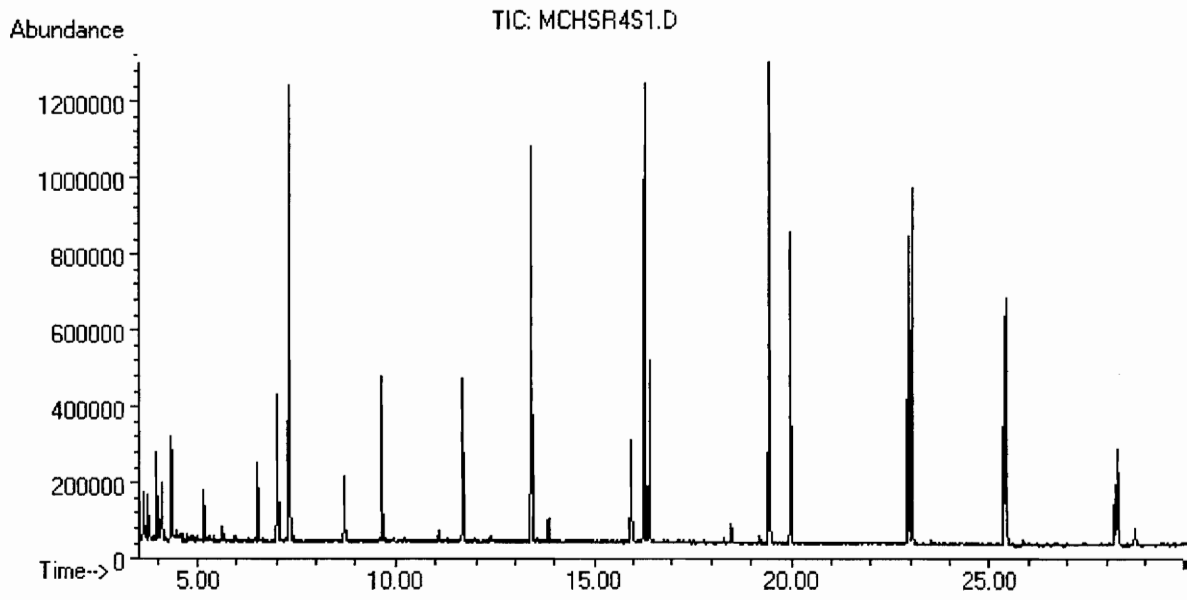


FIGURE 21: Typical chromatogram of a Chloroform/Hexane Microwave Spike

TABLE 9: % Recoveries for Chloroform/Hexane Microwave Spike

COMPOUNDS	% RECOVERY	% RECOVERY	AVG. % RECOVERY
Phenol	13.37	16.14	14.76
2-Chlorophenol	19.64	26.66	23.15
2-Nitrophenol	9.17	13.70	11.43
2,4-Dimethylphenol	ND	ND	ND
2,4-Dichlorophenol	17.06	24.15	20.60
Naphthalene	138.33	126.64	132.48
4-Chloro-3-methylphenol	4.85	10.24	7.55
2,4,6-Trichlorophenol	10.54	17.39	13.96
Acenaphthylene	4.77	3.08	3.93
Acenaphthene	75.14	50.51	62.83
2,4-Dinitrophenol	ND	ND	ND
4-Nitrophenol	ND	ND	ND
Fluorene	138.11	116.45	127.28
2-Methyl-4,6-dinitrophenol	ND	ND	ND
Pentachlorophenol	ND	7.54	7.54
Phenanthrene	141.53	116.73	129.13
Anthracene	71.85	48.53	60.19
Fluoranthene	109.05	90.70	99.88
Pyrene	81.12	63.56	72.34
Chrysene	71.53	59.85	65.69
Benzo (a) anthracene	80.21	67.63	73.92
Benzo (b) fluoranthene	60.89	55.40	58.15
Benzo (k) fluoranthene	56.56	51.53	54.05
Benzo (a) pyrene	ND	ND	ND
Indeno (1,2,3-cd) pyrene	26.90	23.46	25.18
Dibenzo (a,h) anthracene	59.86	53.74	56.80
Benzo (g,h,i) perylene	21.23	75.23	48.23

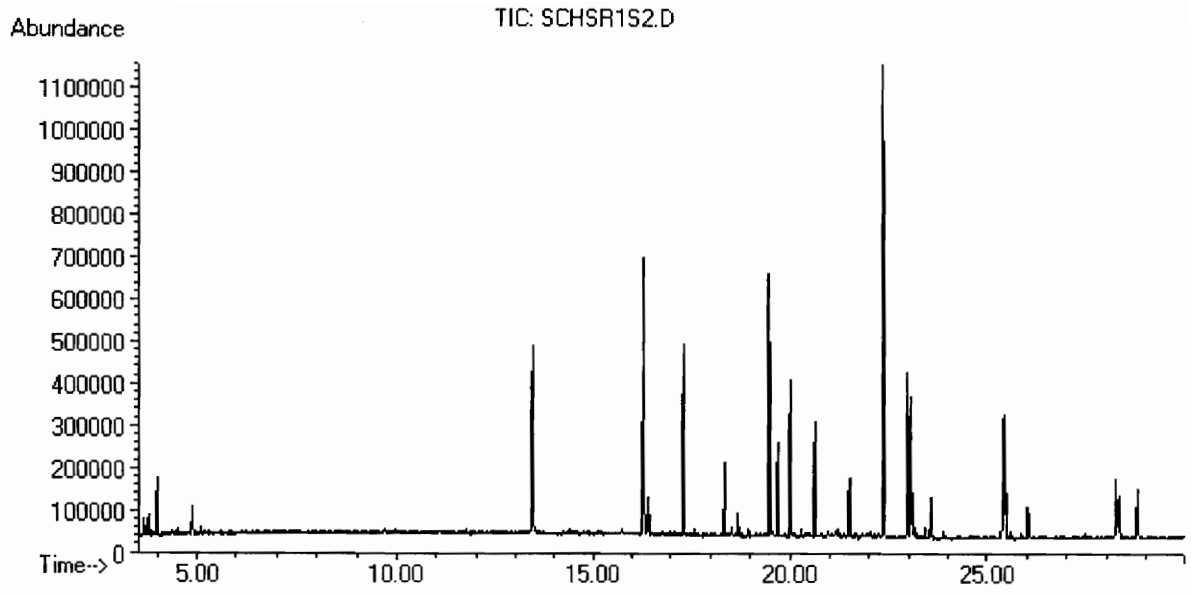


FIGURE 22: Typical chromatogram of a Chloroform/Hexane Soxhlet Spike

TABLE 10: % Recoveries for Chloroform/Hexane Soxhlet Spike

COMPOUNDS	% RECOVERY	% RECOVERY	AVG. % RECOVERY
Phenol	ND	ND	ND
2-Chlorophenol	ND	ND	ND
2-Nitrophenol	ND	ND	ND
2,4-Dimethylphenol	ND	ND	ND
2,4-Dichlorophenol	ND	ND	ND
Naphthalene	ND	ND	ND
4-Chloro-3-methylphenol	ND	ND	ND
2,4,6-Trichlorophenol	ND	ND	ND
Acenaphthylene	ND	ND	ND
Acenaphthene	ND	ND	ND
2,4-Dinitrophenol	ND	ND	ND
4-Nitrophenol	ND	ND	ND
Fluorene	60.12	58.37	59.25
2-Methyl-4,6-dinitrophenol	ND	ND	ND
Pentachlorophenol	ND	ND	ND
Phenanthrene	72.22	71.31	71.77
Anthracene	10.66	15.30	12.98
Fluoranthene	58.70	59.92	59.31
Pyrene	36.28	38.79	37.54
Chrysene	39.66	39.05	39.35
Benzo (a) anthracene	33.54	32.91	33.22
Benzo (b) fluoranthene	38.11	36.05	37.08
Benzo (k) fluoranthene	33.31	29.91	31.61
Benzo (a) pyrene	11.41	14.74	13.08
Indeno (1,2,3-cd) pyrene	33.91	31.03	32.47
Dibenzo (a,h) anthracene	37.27	32.69	34.98
Benzo (g,h,i) perylene	37.63	36.79	37.21

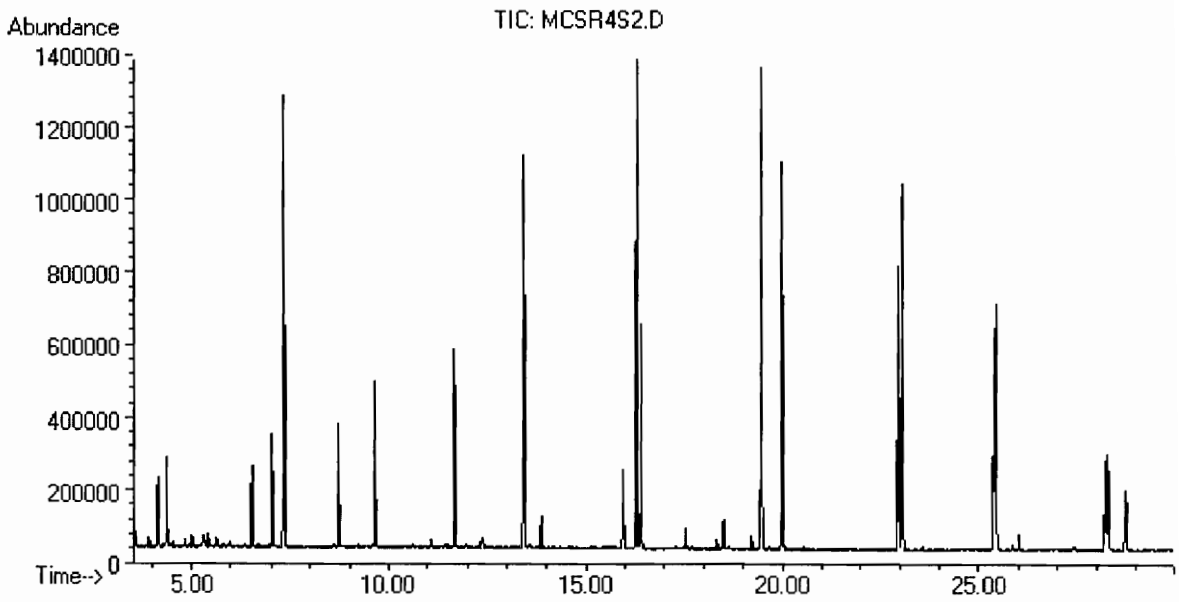


FIGURE 23: Typical chromatogram of a Chloroform Microwave Spike

TABLE 11: % Recoveries for Chloroform Microwave Spike

COMPOUNDS	% RECOVERY	% RECOVERY	AVG. % RECOVERY
Phenol	46.58	51.13	48.85
2-Chlorophenol	41.59	50.51	46.05
2-Nitrophenol	43.05	56.97	50.01
2,4-Dimethylphenol	ND	ND	ND
2,4-Dichlorophenol	37.17	49.03	43.10
Naphthalene	143.49	136.36	139.93
4-Chloro-3-methylphenol	36.86	48.47	42.67
2,4,6-Trichlorophenol	39.29	52.78	46.03
Acenaphthylene	3.88	3.90	3.89
Acenaphthene	80.70	73.48	77.09
2,4-Dinitrophenol	ND	ND	ND
4-Nitrophenol	3.60	22.00	12.80
Fluorene	162.97	156.97	159.97
2-Methyl-4,6-dinitrophenol	32.65	53.05	42.85
Pentachlorophenol	23.20	36.34	29.77
Phenanthrene	164.30	160.56	162.43
Anthracene	76.02	67.31	71.67
Fluoranthene	133.40	133.38	133.39
Pyrene	107.93	103.04	105.48
Chrysene	103.11	99.23	101.17
Benzo (a) anthracene	112.27	115.43	113.85
Benzo (b) fluoranthene	95.93	98.50	97.21
Benzo (k) fluoranthene	93.12	94.51	93.81
Benzo (a) pyrene	12.24	7.98	10.11
Indeno (1,2,3-cd) pyrene	79.23	80.55	79.89
Dibenzo (a,h) anthracene	112.49	115.63	114.06
Benzo (g,h,i) perylene	74.28	69.07	71.67

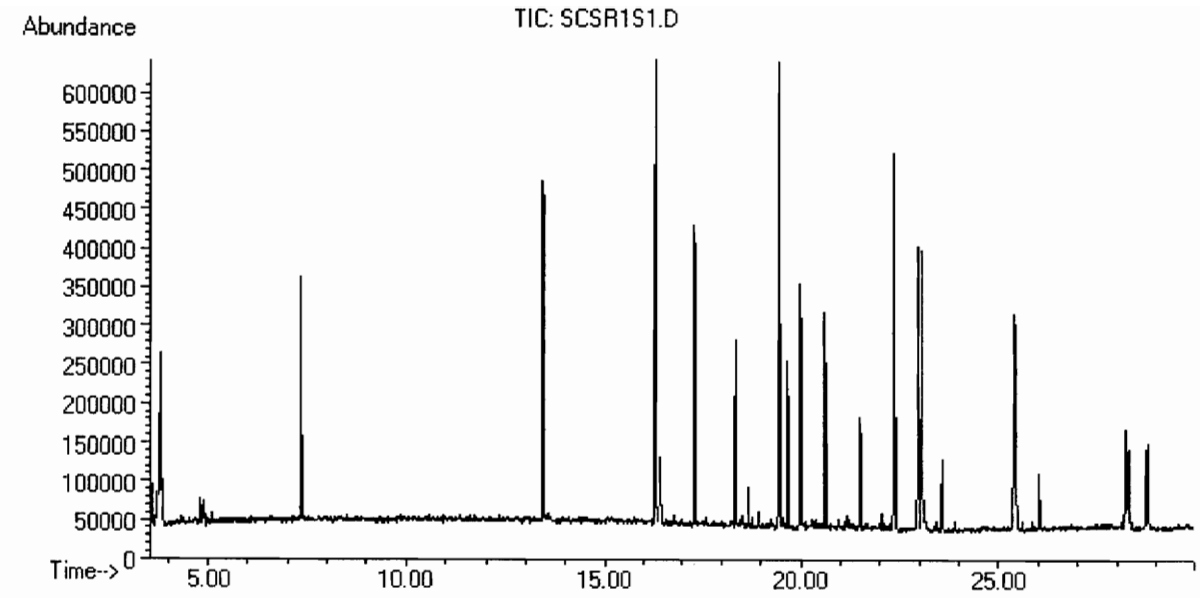


FIGURE 24: Typical chromatogram of a Chloroform Soxhlet Spike

TABLE 12: % Recoveries for Chloroform Soxhlet Spike

COMPOUNDS	% RECOVERY	% RECOVERY	AVG. % RECOVERY
Phenol	ND	ND	ND
2-Chlorophenol	ND	ND	ND
2-Nitrophenol	ND	ND	ND
2,4-Dimethylphenol	ND	ND	ND
2,4-Dichlorophenol	ND	ND	ND
Naphthalene	36.30	33.82	35.06
4-Chloro-3-methylphenol	ND	ND	ND
2,4,6-Trichlorophenol	ND	ND	ND
Acenaphthylene	ND	ND	ND
Acenaphthene	ND	ND	ND
2,4-Dinitrophenol	ND	ND	ND
4-Nitrophenol	ND	ND	ND
Fluorene	86.24	61.55	73.90
2-Methyl-4,6-dinitrophenol	ND	ND	ND
Pentachlorophenol	ND	ND	ND
Phenanthrene	87.55	71.93	79.74
Anthracene	11.19	12.03	11.61
Fluoranthene	65.13	59.78	62.46
Pyrene	35.24	31.65	33.45
Chrysene	44.53	39.82	42.17
Benzo (a) anthracene	44.61	40.64	42.63
Benzo (b) fluoranthene	43.47	37.08	40.28
Benzo (k) fluoranthene	40.65	34.36	37.51
Benzo (a) pyrene	7.81	11.65	9.73
Indeno (1,2,3-cd) pyrene	39.59	33.17	36.38
Dibenzo (a,h) anthracene	47.02	43.74	45.38
Benzo (g,h,i) perylene	41.78	41.41	41.59

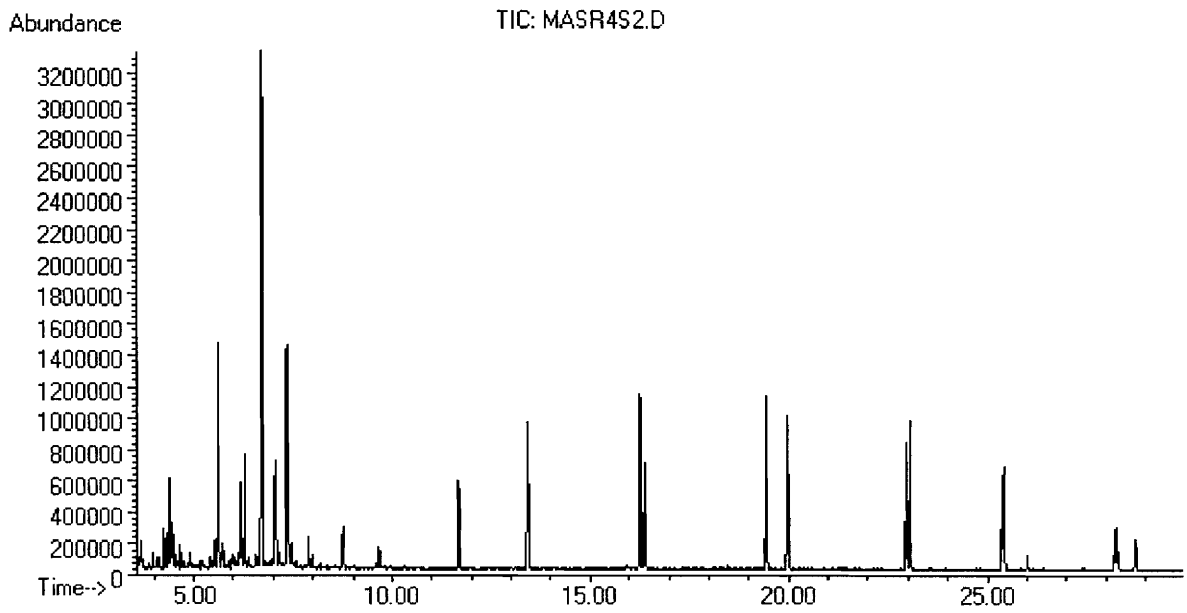


FIGURE 25: Typical chromatogram of an Acetone Microwave Spike

TABLE 13: % Recoveries for Acetone Microwave Spike

COMPOUNDS	% RECOVERY	% RECOVERY	AVG. % RECOVERY
Phenol	ND	ND	ND
2-Chlorophenol	102.25	81.55	91.90
2-Nitrophenol	ND	ND	ND
2,4-Dimethylphenol	ND	ND	ND
2,4-Dichlorophenol	165.05	129.35	147.20
Naphthalene	195.82	271.11	233.47
4-Chloro-3-methylphenol	59.76	43.95	51.86
2,4,6-Trichlorophenol	32.30	ND	32.30
Acenaphthylene	ND	ND	ND
Acenaphthene	67.51	74.62	71.06
2,4-Dinitrophenol	ND	ND	ND
4-Nitrophenol	ND	ND	ND
Fluorene	153.00	137.54	145.27
2-Methyl-4,6-dinitrophenol	ND	ND	ND
Pentachlorophenol	ND	ND	ND
Phenanthrene	155.02	140.76	147.89
Anthracene	55.43	75.80	65.62
Fluoranthene	129.81	117.22	123.52
Pyrene	101.08	99.35	100.22
Chrysene	104.15	96.52	100.33
Benzo (a) anthracene	109.85	102.50	106.17
Benzo (b) fluoranthene	94.25	87.60	90.92
Benzo (k) fluoranthene	94.35	88.11	91.23
Benzo (a) pyrene	ND	ND	ND
Indeno (1,2,3-cd) pyrene	78.95	74.58	76.77
Dibenzo (a,h) anthracene	115.34	106.79	111.06
Benzo (g,h,i) perylene	72.05	72.78	72.41

SUMMARY

A full comparison between Soxhlet and MAE was conducted. Conclusions drawn from this are as follows:

(1) Following the EPA's approved Soxhlet extraction (Method 3540B in SW-846), the sample size was reduced to match the capacity of the microwave. A comparison was conducted, wherein MAE and Soxhlet both went through the same concentration step prior to GC/MS analysis. Better results were achieved in the MAE for both the non-polar and polar compounds regardless of solvent choice. Soxhlet seems to be more discriminatory than previously thought. For example, the non-polar compounds (PAHs) were generally recovered, but the polar compounds (phenols) were not detected.

(2) MAE is being actively investigated and may become an EPA approved procedure in the future. Soxhlet uses 100 - 500 mL of solvent and MAE uses only 10 - 30 mL. Therefore, lower waste solvent disposal results and workers are exposed to less solvent.

(3) Soxhlet extraction time can range from 16 - 24 hours. MAE run times are substantially lower, usually under 30 min and multiple samples can be extractedsimultaneously, provided the same solvent and conditions are used. Higher efficiency is acheived with MAE due to the increased solvating power of the polar solvent.

(4) The solvent choices in other papers usually included a 1:1 acetone/hexane mixes. The EPA does offer this solvent selection in its Soxhlet method, as well as methylene chloride. It was shown here that chloroform, similar to $MeCl_2$, is actually better than acetone.

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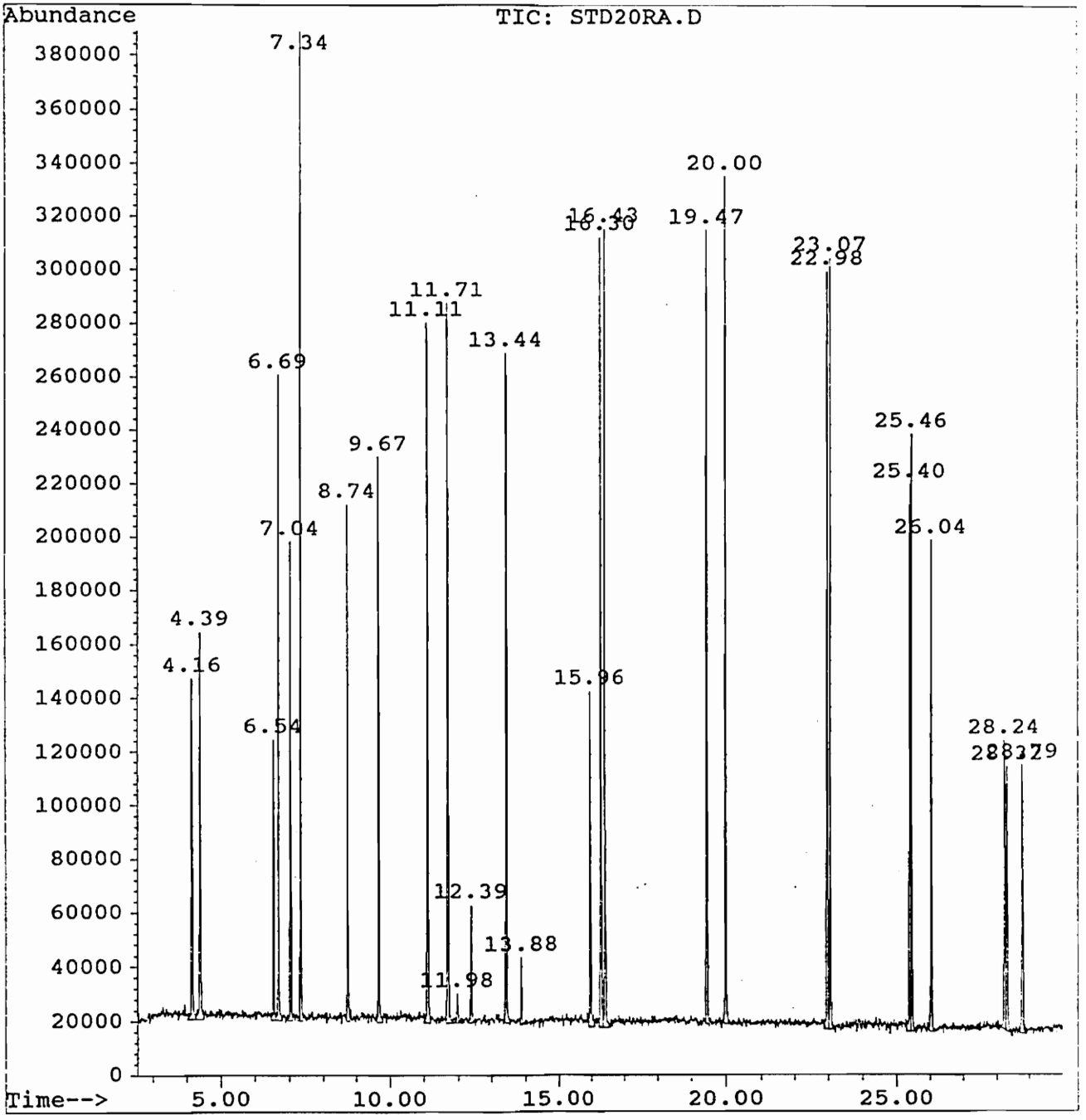
APPENDIX A

Standard Chromatograms, Area Counts,

Calibration Curves, and

Mass Spectrums

File : C:\HPCHEM\1\DATA\STD20RA.D
Operator : Smith
Acquired : 15 Dec 95 8:16 pm using AcqMethod SCOTT
Instrument : 5971 - In
Sample Name: Phenols and PAHs
Misc Info : Chloroform
Vial Number: 1

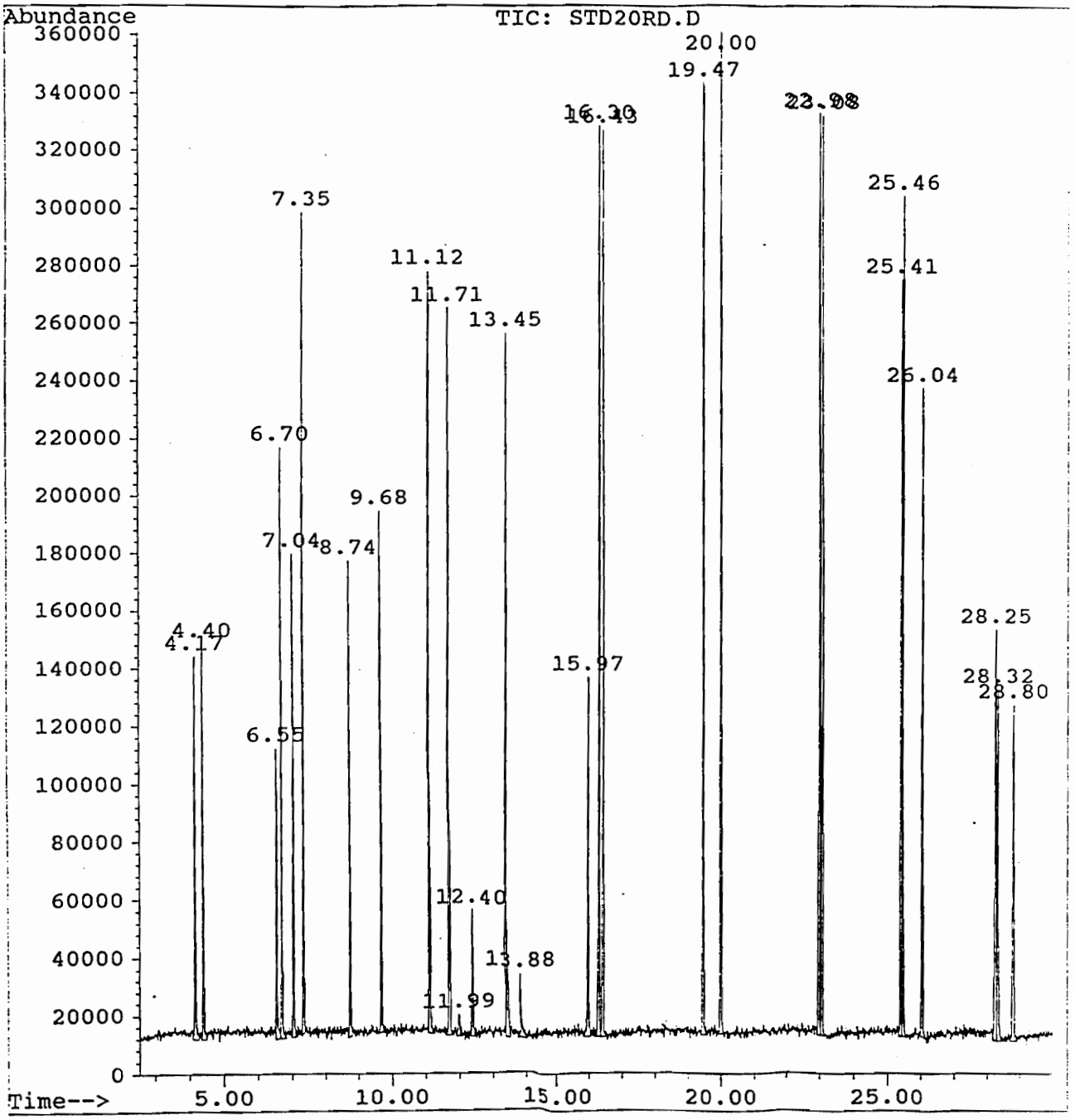


TIC: STD20RA.D

Phenols and PAHs

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	4.157	PV	0.032	2461163	4.045	4.252
2	4.387	VV	0.032	1870229	4.252	4.494
3	6.541	VV	0.028	1753892	6.477	6.584
4	6.692	VV	0.052	3578790	6.584	6.779
5	7.035	VV	0.039	2824469	6.945	7.100
6	7.339	VV	0.029	5428259	7.213	7.387
7	8.738	VV	0.025	2774620	8.692	8.798
8	9.672	VV	0.038	3106270	9.599	9.777
9	11.111	VV	0.033	5367461	11.026	11.179
10	11.706	VV	0.035	5740483	11.641	11.899
11	11.980	VV	0.036	210919	11.939	12.055
12	12.390	VV	0.035	916885	12.289	12.440
13	13.441	VV	0.035	5462746	13.378	13.539
14	13.877	PV	0.029	411821	13.832	13.911
15	15.964	VV	0.032	2342073	15.913	16.095
16	16.300	VV	0.033	5720544	16.236	16.353
17	16.426	VV	0.049	5587144	16.353	16.569
18	19.472	VV	0.031	5668987	19.413	19.532
19	20.002	VV	0.031	5636173	19.940	20.084
20	22.979	VV	0.031	5126091	22.893	23.023
21	23.074	VV	0.030	4811395	23.023	23.147
22	25.401	VV	0.035	4363601	25.286	25.428
23	25.457	VV	0.030	4098573	25.428	25.521
24	26.040	VV	0.032	3480791	25.976	26.124
25	28.243	BV	0.038	3600488	28.122	28.277
26	28.316	VV	0.037	3249037	28.277	28.395
27	28.788	VV	0.041	2597913	28.695	28.908

File : C:\HPCHEM\1\DATA\STD20RD.D
Operator : Smith
Acquired : 16 Dec 95 3:25 am using AcqMethod SCOTT
Instrument : 5971 - In
Sample Name: Phenols and PAHs
Misc Info : Chloroform
Vial Number: 1



TIC: STD20RD.D

Phenols and PAHs

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	4.167	VV	0.042	2118489	4.093	4.266
2	4.396	VV	0.029	2171067	4.341	4.487
3	6.548	PV	0.028	1642618	6.514	6.649
4	6.701	VV	0.027	3125468	6.649	6.813
5	7.043	VV	0.028	2560639	6.973	7.153
6	7.346	PV	0.027	4591944	7.288	7.396
7	8.742	PV	0.037	2526882	8.681	8.799
8	9.677	PV	0.026	2642010	9.586	9.744
9	11.115	VV	0.042	4995288	11.058	11.202
10	11.709	VV	0.036	5248508	11.588	11.780
11	11.987	PV	0.044	208994	11.904	12.046
12	12.398	VV	0.037	1024908	12.335	12.503
13	13.446	VV	0.034	5290776	13.381	13.511
14	13.881	PV	0.043	407792	13.747	14.060
15	15.966	VV	0.030	2240602	15.849	16.046
16	16.303	VV	0.046	5833797	16.233	16.360
17	16.428	VV	0.030	5579577	16.360	16.496
18	19.474	VV	0.031	6379586	19.381	19.530
19	20.004	VV	0.031	6273777	19.916	20.074
20	22.981	VV	0.029	5620766	22.924	23.026
21	23.078	VV	0.028	5390418	23.026	23.161
22	25.405	VV	0.032	5032668	25.319	25.431
23	25.463	VV	0.040	4789514	25.431	25.527
24	26.044	PV	0.047	4012580	25.964	26.140
25	28.251	VV	0.038	3360868	28.170	28.285
26	28.324	VV	0.038	2985370	28.285	28.502
27	28.798	VV	0.044	3153388	28.685	28.910

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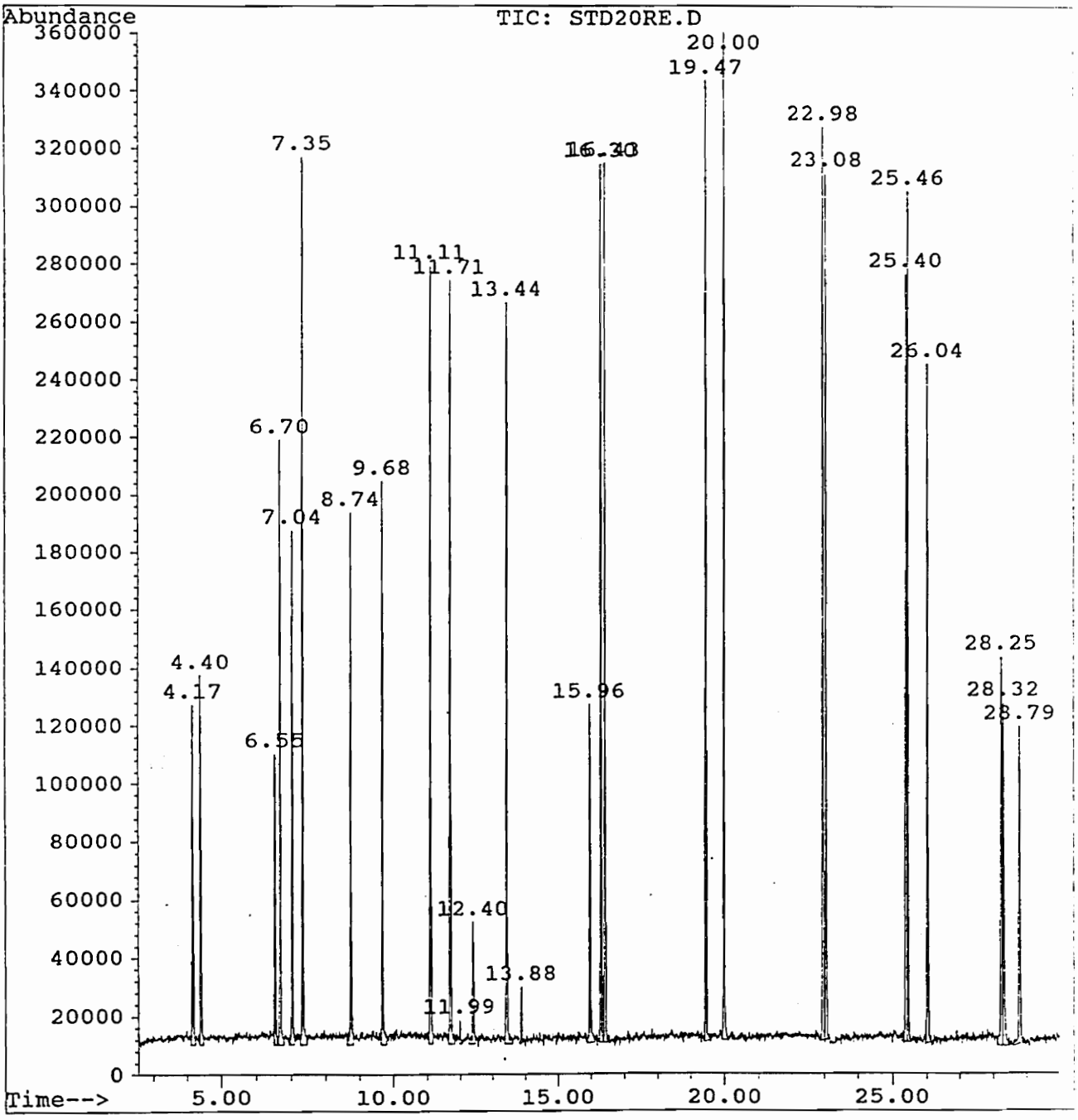
Vial Number: 1



TIC: STD20RE.D

Phenols and PAHs

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1	4.169	VV	0.032	2089427	4.102	4.240
2	4.398	VV	0.031	2186547	4.355	4.465
3	6.548	VV	0.027	1566920	6.509	6.598
4	6.700	VV	0.028	3304354	6.633	6.795
5	7.043	VV	0.028	2693112	6.978	7.090
6	7.345	VV	0.028	5211323	7.258	7.466
7	8.740	VV	0.052	2789707	8.626	8.807
8	9.676	VV	0.028	3037286	9.627	9.794
9	11.114	VV	0.033	5372678	11.042	11.170
10	11.707	VV	0.036	5610440	11.638	11.831
11	11.986	VV	0.034	176887	11.959	12.037
12	12.397	VV	0.040	1100471	12.260	12.454
13	13.442	VV	0.035	5664280	13.372	13.612
14	13.878	PV	0.031	347256	13.833	13.943
15	15.964	PV	0.031	2172873	15.864	16.100
16	16.301	VV	0.031	5784450	16.227	16.361
17	16.427	VV	0.043	5539616	16.361	16.507
18	19.471	PV	0.032	6352945	19.399	19.601
19	20.002	VV	0.041	6230495	19.921	20.092
20	22.980	PV	0.031	5691179	22.873	23.026
21	23.076	VV	0.030	5255966	23.026	23.134
22	25.403	VV	0.047	5190411	25.268	25.430
23	25.459	VV	0.029	4901210	25.430	25.546
24	26.043	VV	0.047	4099964	25.985	26.103
25	28.247	VV	0.040	3319156	28.122	28.282
26	28.320	VV	0.039	2866331	28.282	28.503
27	28.795	PV	0.045	2975534	28.584	28.883

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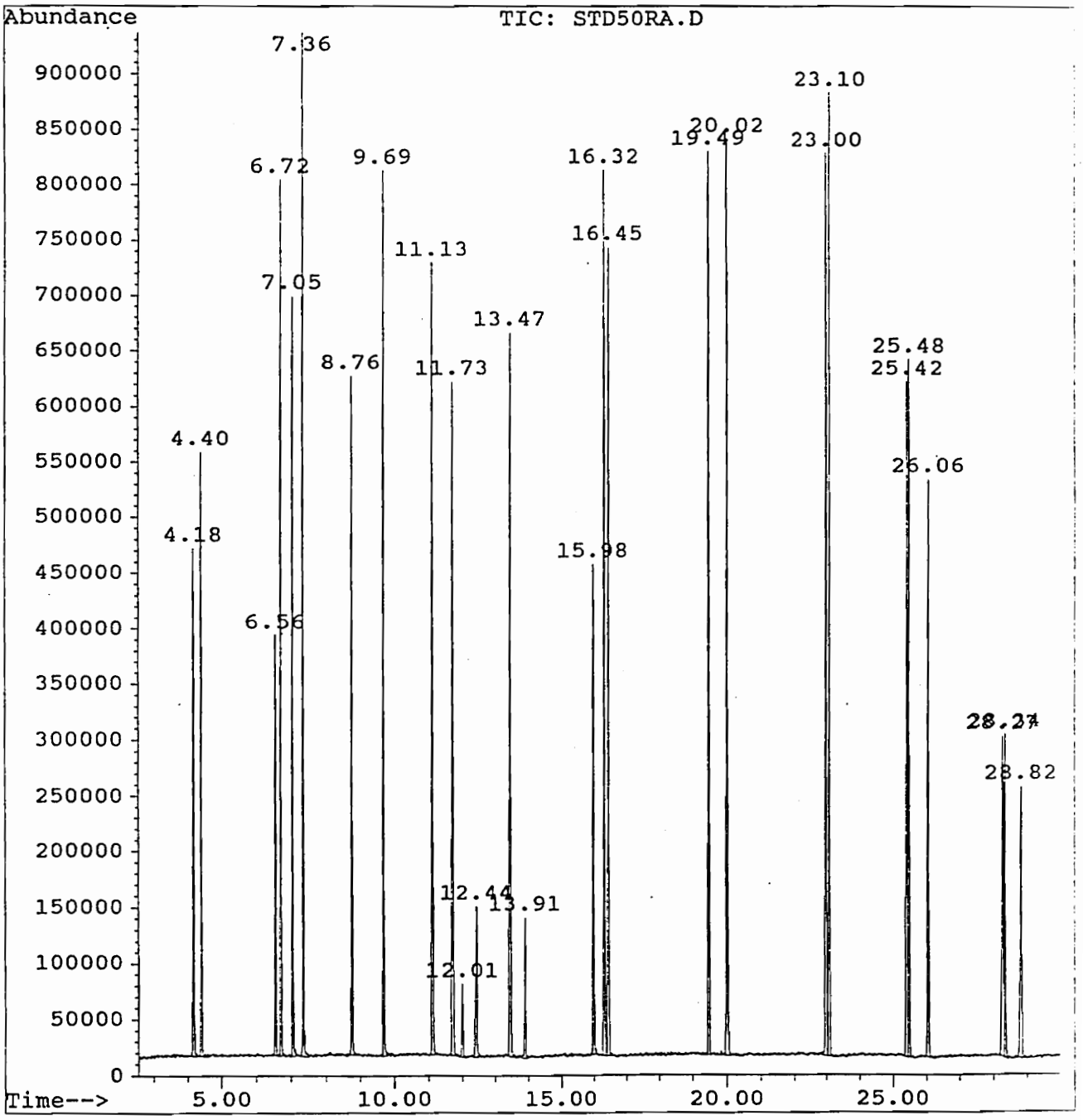
Vial Number: 2



TIC: STD50RA.D

Phenols and PAHs

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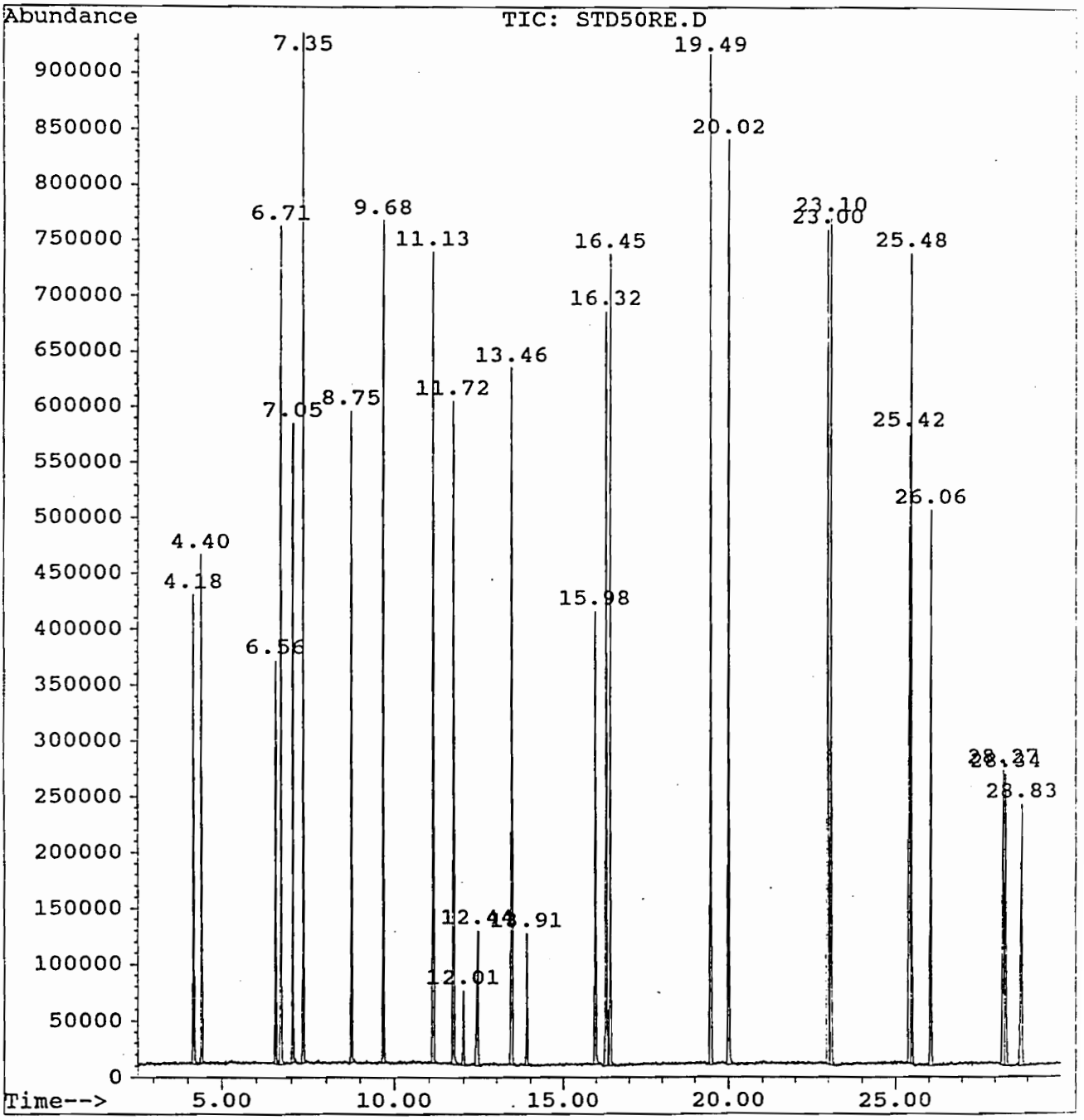
2	4.404	BB	0.028	8984715	4.338	4.537
3	6.559	BB	0.028	6280751	6.500	6.630
4	6.716	BB	0.040	12270178	6.662	6.787
5	7.054	BB	0.041	11036534	6.965	7.178
6	7.355	BB	0.029	14783377	7.297	7.515
7	8.756	BV	0.030	10227534	8.700	8.900
8	9.686	BV	0.039	11867110	9.611	9.793
9	11.128	BB	0.035	14729631	11.043	11.216
10	11.729	BV	0.038	15265710	11.650	11.837
11	12.012	VB	0.039	1611232	11.837	12.118
12	12.444	BV	0.046	4294234	12.323	12.492
13	13.465	BB	0.042	14948698	13.290	13.622
14	13.907	VB	0.034	2658379	13.782	13.954
15	15.983	BB	0.037	10003210	15.889	16.088
16	16.323	BV	0.034	16310673	16.211	16.368
17	16.453	VB	0.042	15772086	16.368	16.534
18	19.490	BB	0.034	17559849	19.255	19.534
19	20.022	BB	0.035	17443005	19.948	20.128
20	22.998	BV	0.035	16450345	22.793	23.034
21	23.102	VV	0.044	15627049	23.034	23.210
22	25.421	BV	0.037	13697779	25.306	25.448
23	25.484	VB	0.035	13256956	25.448	25.553
24	26.062	BB	0.055	10977258	25.989	26.188
25	28.270	BV	0.045	8295830	28.179	28.299
26	28.341	VV	0.038	7096811	28.299	28.420
27	28.822	BV	0.046	7478071	28.691	28.907

File : C:\HPCHEM\1\DATA\STD50RE.D
Operator : Smith
Acquired : 16 Dec 95 6:24 am using AcqMethod SCOTT
Instrument : 5971 - In
Sample Name: Phenols and PAHs
Misc Info : Chloroform
Vial Number: 2

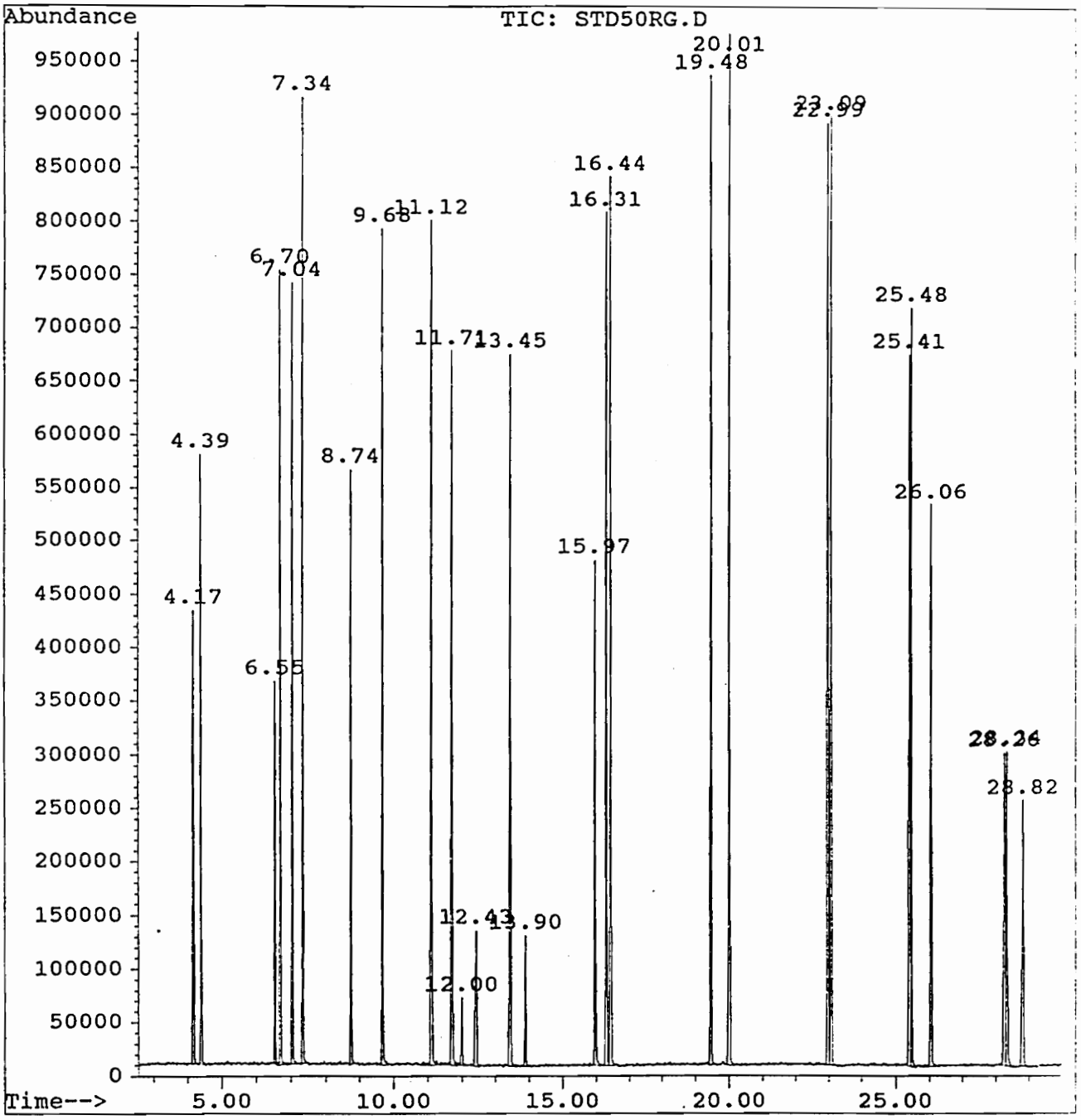


TIC: STD50RE.D

Phenols and PAHs

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	4.176	BB	0.047	7006424	4.091	4.291
2	4.399	BB	0.030	8036604	4.348	4.490
3	6.556	BV	0.028	5723602	6.424	6.599
4	6.713	PB	0.031	11607267	6.599	6.842
5	7.051	BV	0.028	9860647	6.965	7.108
6	7.353	BB	0.025	13380918	7.278	7.434
7	8.753	BV	0.049	9425074	8.681	8.817
8	9.685	BB	0.040	11110398	9.601	9.829
9	11.127	BB	0.032	14099063	11.033	11.235
10	11.725	BV	0.039	14682949	11.498	11.868
11	12.014	VB	0.036	1412707	11.868	12.076
12	12.443	VB	0.052	3879878	12.340	12.563
13	13.462	BB	0.037	14090607	13.376	13.594
14	13.907	BV	0.035	2499589	13.831	13.983
15	15.981	BB	0.036	9352647	15.794	16.088
16	16.322	BV	0.035	15710028	16.183	16.374
17	16.449	VB	0.034	15049851	16.374	16.562
18	19.489	PB	0.053	17172655	19.413	19.616
19	20.021	BB	0.032	17304486	19.929	20.156
20	22.996	BV	0.036	16241240	22.906	23.035
21	23.097	VB	0.036	15164644	23.035	23.210
22	25.421	BV	0.038	13863128	25.344	25.448
23	25.484	VV	0.043	13114185	25.448	25.554
24	26.062	BB	0.034	10933938	25.827	26.159
25	28.270	BV	0.045	7889085	28.160	28.302
26	28.343	VB	0.044	6542935	28.302	28.521
27	28.828	BV	0.048	6867291	28.720	28.927

File : C:\HPCHEM\1\DATA\STD50RG.D
Operator : Smith
Acquired : 16 Dec 95 11:10 am using AcqMethod SCOTT
Instrument : 5971 - In
Sample Name: Phenols and PAHs
Misc Info : Chloroform
Vial Number: 2

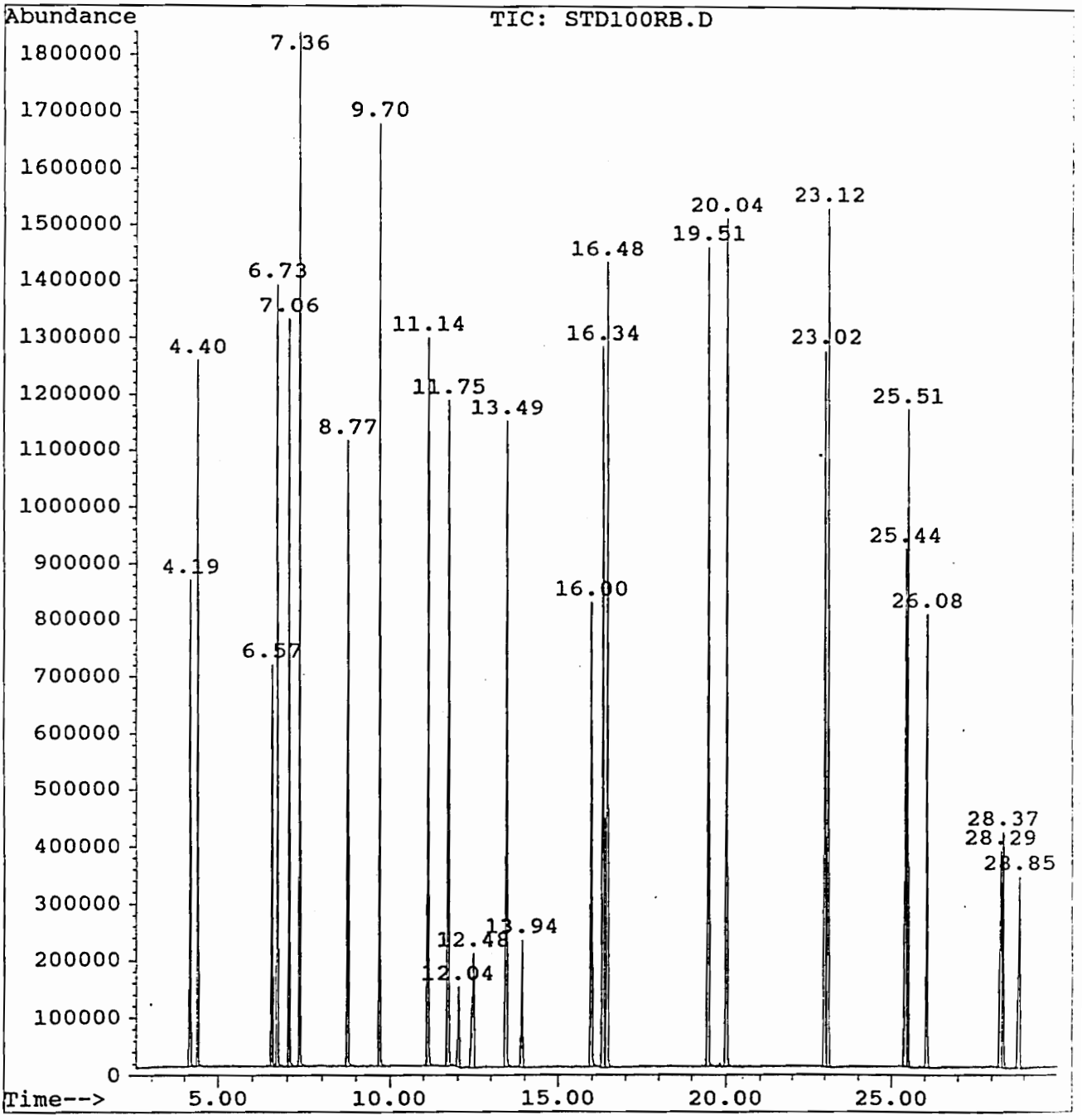


TIC: STD50RG.D

Phenols and PAHs

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	4.169	BV	0.030	7576860	4.110	4.246
2	4.393	PB	0.048	8838918	4.246	4.499
3	6.547	BV	0.029	5959030	6.500	6.593
4	6.702	PV	0.031	12063470	6.593	6.813
5	7.041	VB	0.027	11001216	6.813	7.164
6	7.344	BB	0.028	14842399	7.287	7.449
7	8.745	PB	0.029	10373565	8.674	8.833
8	9.675	BB	0.028	12309148	9.592	9.813
9	11.116	BV	0.045	15953741	10.796	11.228
10	11.712	BV	0.038	16026844	11.631	11.839
11	11.999	BB	0.035	1424397	11.944	12.076
12	12.430	BV	0.047	4137270	12.323	12.498
13	13.451	BV	0.038	15463342	13.347	13.556
14	13.896	PV	0.038	2952107	13.556	13.971
15	15.971	BV	0.051	10109094	15.870	16.105
16	16.312	PV	0.035	17069549	16.105	16.361
17	16.439	VB	0.035	16096362	16.361	16.542
18	19.480	BV	0.034	18830881	19.398	19.525
19	20.012	BB	0.053	19031965	19.919	20.137
20	22.989	BV	0.035	17810166	22.916	23.027
21	23.093	VB	0.044	16786707	23.027	23.172
22	25.414	BV	0.038	15092804	25.334	25.441
23	25.479	VB	0.034	14892774	25.441	25.600
24	26.055	BB	0.036	11946313	25.751	26.200
25	28.262	BV	0.046	8639425	28.170	28.294
26	28.337	VB	0.061	7314342	28.294	28.474
27	28.817	BB	0.046	7391214	28.720	28.919

File : C:\HPCHEM\1\DATA\STD100RB.D
Operator : Smith
Acquired : 15 Dec 95 11:51 pm using AcqMethod SCOTT
Instrument : 5971 - In
Sample Name: Phenols and PAHs
Misc Info : Chloroform
Vial Number: 3

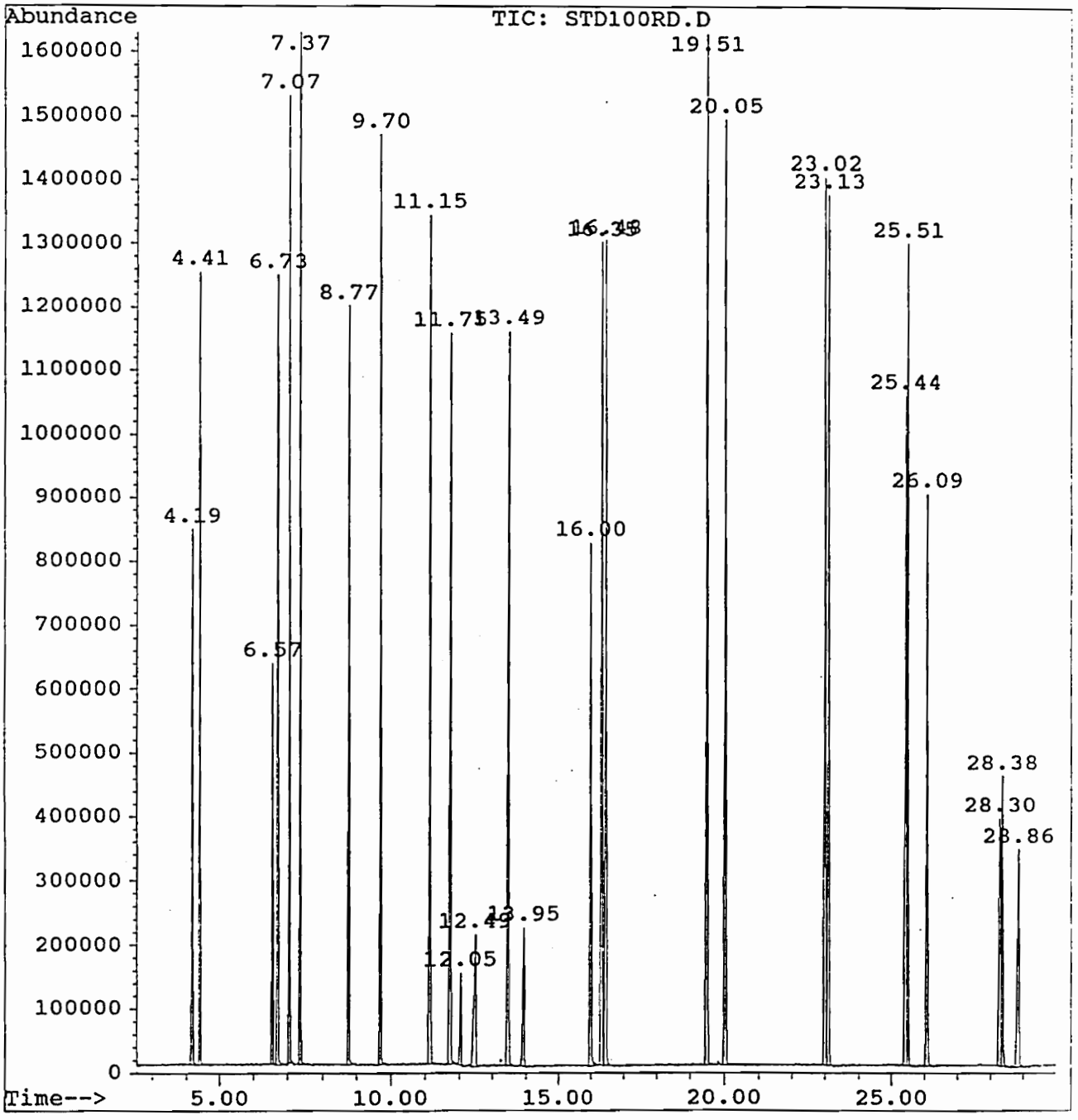


TIC: STD100RB.D

Phenols and PAHs

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	4.185	BB	0.031	15790379	3.902	4.272
2	4.404	BB	0.026	19051618	4.319	4.456
3	6.566	BV	0.031	13034440	6.500	6.648
4	6.726	PB	0.041	25333747	6.648	6.851
5	7.062	BB	0.029	24058438	6.993	7.183
6	7.364	BB	0.031	30799267	7.268	7.534
7	8.766	BB	0.034	22357308	8.700	8.856
8	9.696	BB	0.027	26963943	9.620	9.829
9	11.145	BB	0.039	31267837	11.043	11.263
10	11.746	BV	0.047	31224455	11.631	11.820
11	12.042	BV	0.045	3955048	11.944	12.114
12	12.483	BB	0.062	8617098	12.351	12.589
13	13.487	BB	0.041	30042126	13.385	13.633
14	13.942	BV	0.046	5988736	13.821	13.985
15	15.999	BB	0.041	22262185	15.709	16.145
16	16.344	BV	0.041	32849672	16.230	16.389
17	16.477	VB	0.038	31794008	16.389	16.591
18	19.508	BB	0.040	36291406	19.398	19.587
19	20.042	BV	0.041	35109889	19.938	20.104
20	23.018	BV	0.039	33418338	22.906	23.052
21	23.123	VB	0.037	31277298	23.052	23.219
22	25.438	BV	0.041	28003553	25.344	25.466
23	25.505	VV	0.036	28358580	25.466	25.568
24	26.081	BB	0.037	25003121	25.989	26.169
25	28.291	BV	0.059	16977137	28.179	28.323
26	28.366	VB	0.041	12299212	28.323	28.437
27	28.853	BV	0.059	14730717	28.710	28.957

File : C:\HPCHEM\1\DATA\STD100RD.D
Operator : Smith
Acquired : 16 Dec 95 4:37 am using AcqMethod SCOTT
Instrument : 5971 - In
Sample Name: Phenols and PAHs
Misc Info : Chloroform
Vial Number: 3

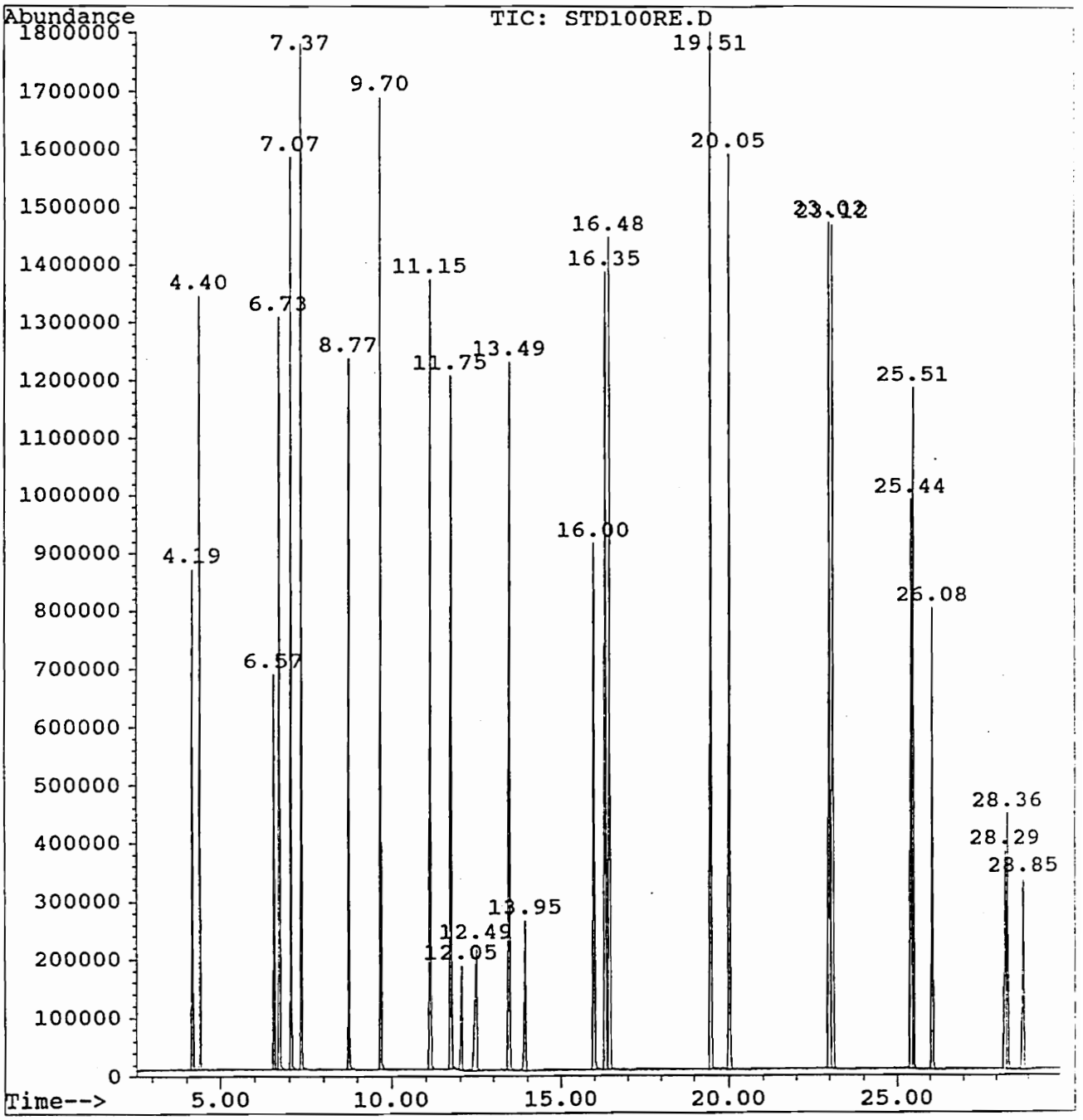


TIC: STD100RD.D

Phenols and PAHs

Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	4.186	BB	0.033	15191404	4.120	4.300
2	4.405	BV	0.027	18466990	4.348	4.482
3	6.571	BB	0.035	12755771	6.510	6.643
4	6.729	BV	0.033	25395960	6.652	6.862
5	7.067	PB	0.027	22875771	6.862	7.145
6	7.370	BB	0.030	31018981	7.306	7.506
7	8.773	BB	0.039	21910574	8.681	8.900
8	9.699	BB	0.032	26274784	9.601	9.791
9	11.150	BB	0.038	30265070	11.062	11.251
10	11.751	VB	0.041	30537464	11.645	11.852
11	12.048	BB	0.041	3726411	11.972	12.173
12	12.491	BB	0.054	8073686	12.342	12.555
13	13.494	BB	0.049	29633836	13.357	13.613
14	13.946	BB	0.042	6200868	13.840	14.030
15	16.001	BB	0.043	21675211	15.898	16.167
16	16.350	BV	0.041	31866645	16.221	16.392
17	16.479	VB	0.040	31487879	16.392	16.554
18	19.513	BV	0.042	36120302	19.388	19.606
19	20.048	BB	0.051	35898341	19.929	20.156
20	23.019	BV	0.036	33163643	22.916	23.054
21	23.128	VB	0.041	32760547	23.054	23.179
22	25.445	BV	0.046	29382236	25.344	25.471
23	25.513	VB	0.034	28180801	25.471	25.600
24	26.090	BV	0.040	24452430	25.979	26.157
25	28.301	BV	0.060	17024388	28.170	28.332
26	28.375	VB	0.041	12491539	28.332	28.521
27	28.864	BB	0.054	14436858	28.587	28.966

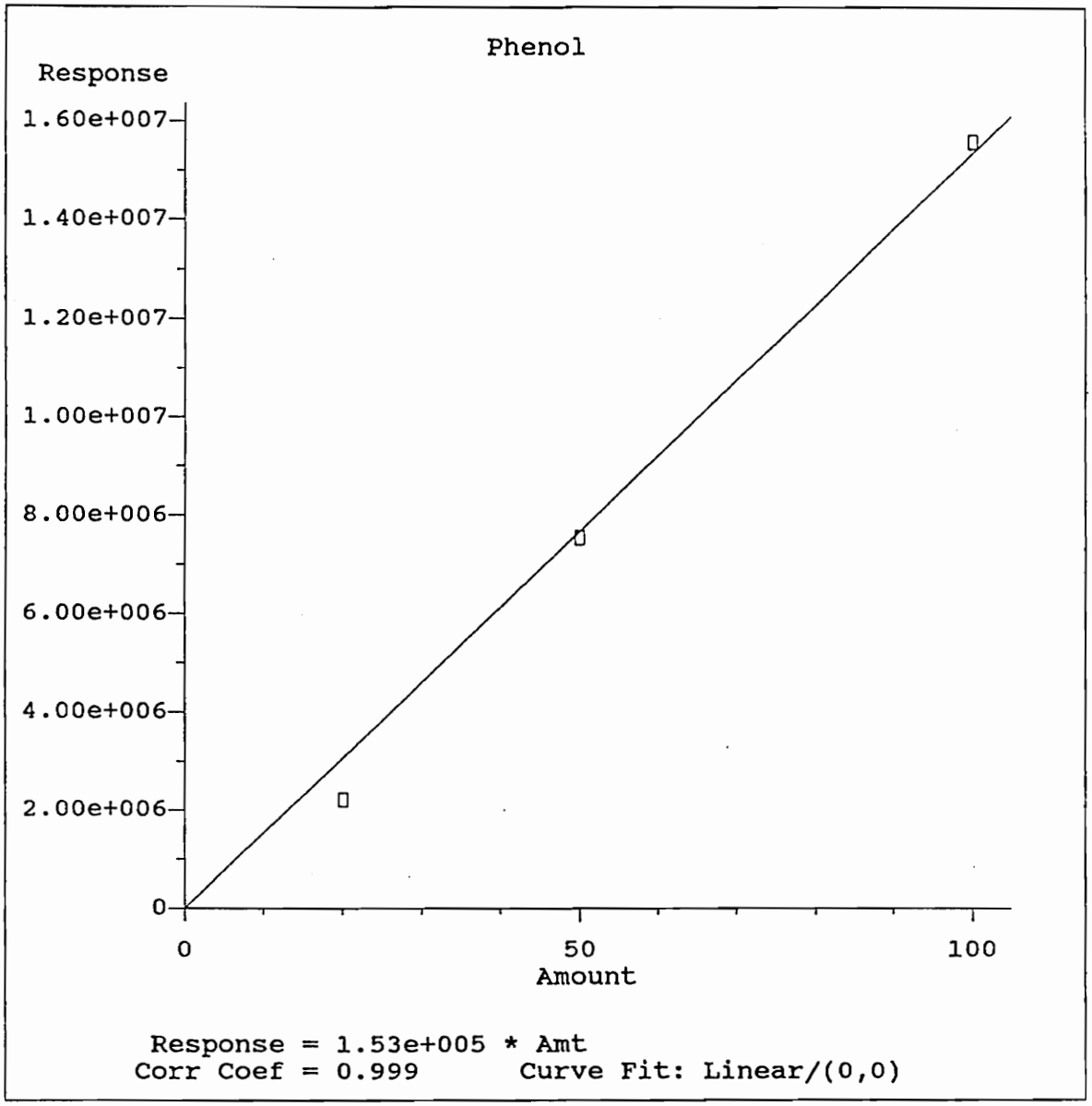
File : C:\HPCHEM\1\DATA\STD100RE.D
Operator : Smith
Acquired : 16 Dec 95 7:00 am using AcqMethod SCOTT
Instrument : 5971 - In
Sample Name: Phenols and PAHs
Misc Info : Chloroform
Vial Number: 3



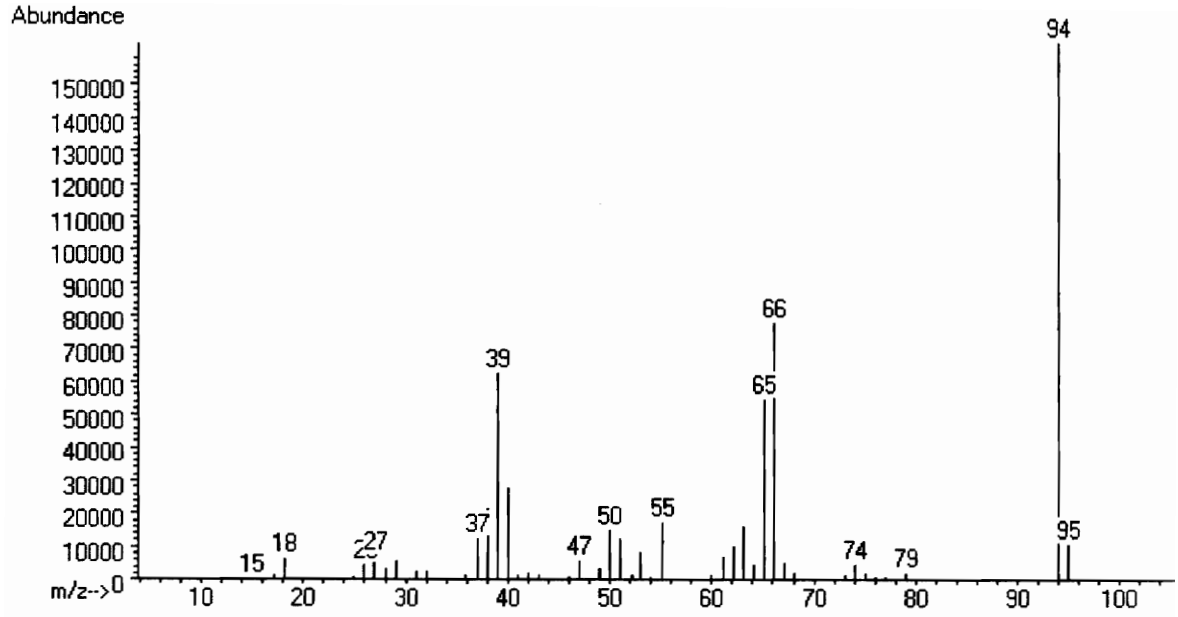
TIC: STD100RE.D

Phenols and PAHs

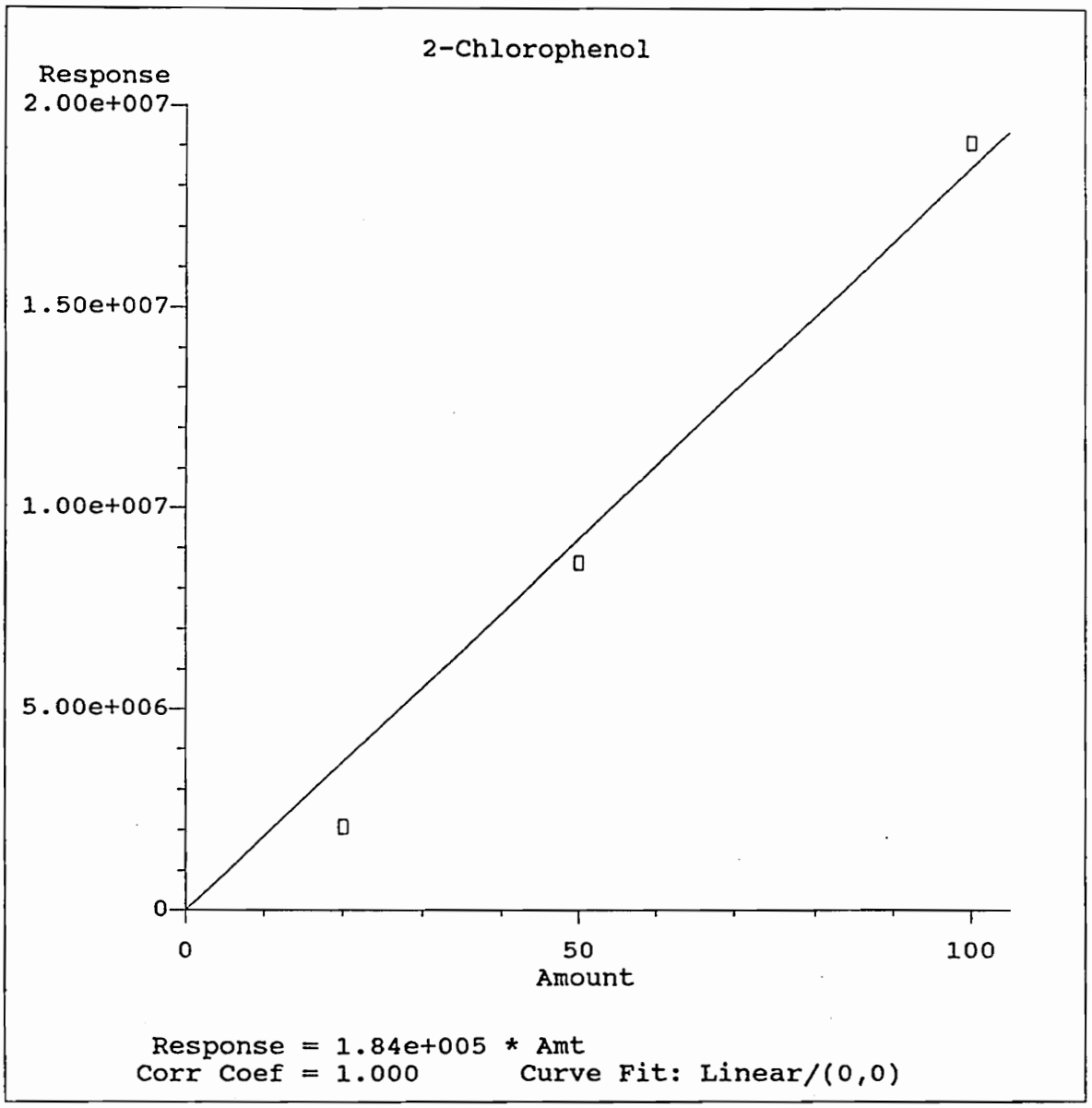
Peak#	Ret Time	Type	Width	Area	Start Time	End Time
1	4.186	BB	0.034	15743923	4.110	4.272
2	4.405	BB	0.027	19705716	4.348	4.490
3	6.568	BB	0.033	13692466	6.481	6.643
4	6.727	BB	0.034	27045165	6.643	6.842
5	7.066	BB	0.042	25699124	7.003	7.164
6	7.367	BV	0.033	33402317	7.297	7.458
7	8.771	BB	0.033	24308075	8.625	8.881
8	9.696	BB	0.031	28521721	9.630	9.819
9	11.146	BB	0.038	33968845	11.043	11.289
10	11.749	BV	0.043	33658146	11.640	11.795
11	12.049	BB	0.044	3846881	11.944	12.143
12	12.491	BB	0.057	9172025	12.171	12.608
13	13.491	BB	0.042	33242831	13.385	13.613
14	13.947	BV	0.044	6403472	13.812	14.000
15	16.000	BB	0.044	24302290	15.889	16.154
16	16.349	BV	0.043	35632160	16.230	16.388
17	16.478	VB	0.043	34880941	16.388	16.581
18	19.511	BB	0.041	39734906	19.398	19.597
19	20.046	PB	0.039	38971289	19.933	20.194
20	23.017	BV	0.039	33317829	22.925	23.052
21	23.123	VB	0.036	32749589	23.052	23.238
22	25.437	BV	0.042	28189607	25.325	25.462
23	25.507	VB	0.038	28799880	25.462	25.590
24	26.079	BV	0.048	25449791	25.989	26.169
25	28.291	BV	0.051	18339732	28.179	28.322
26	28.365	VB	0.054	12903020	28.322	28.483
27	28.850	BB	0.052	13127509	28.710	28.985



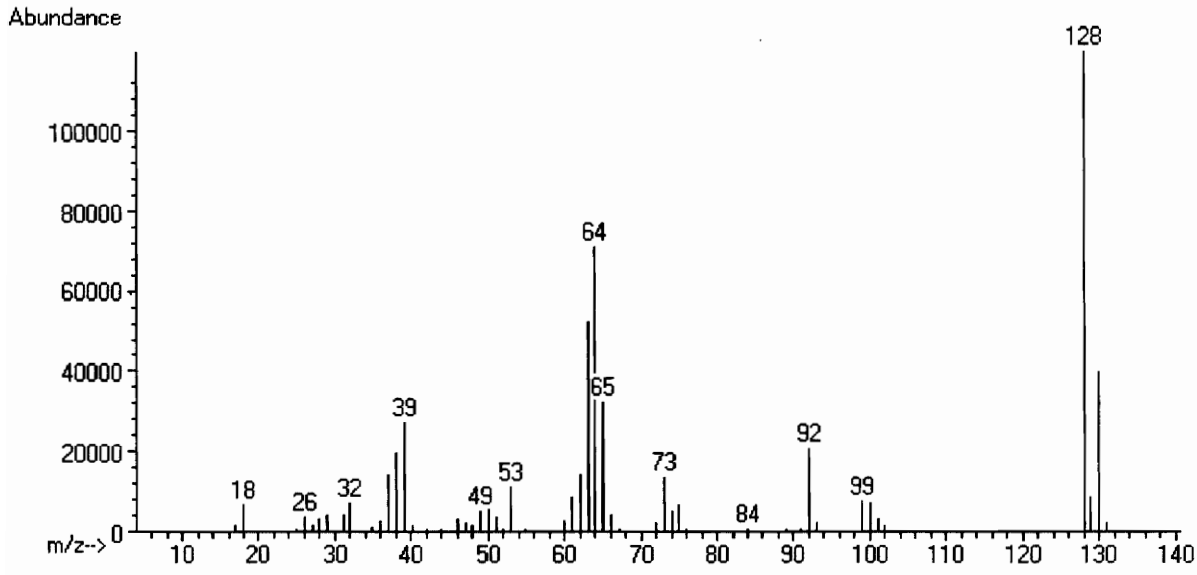
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



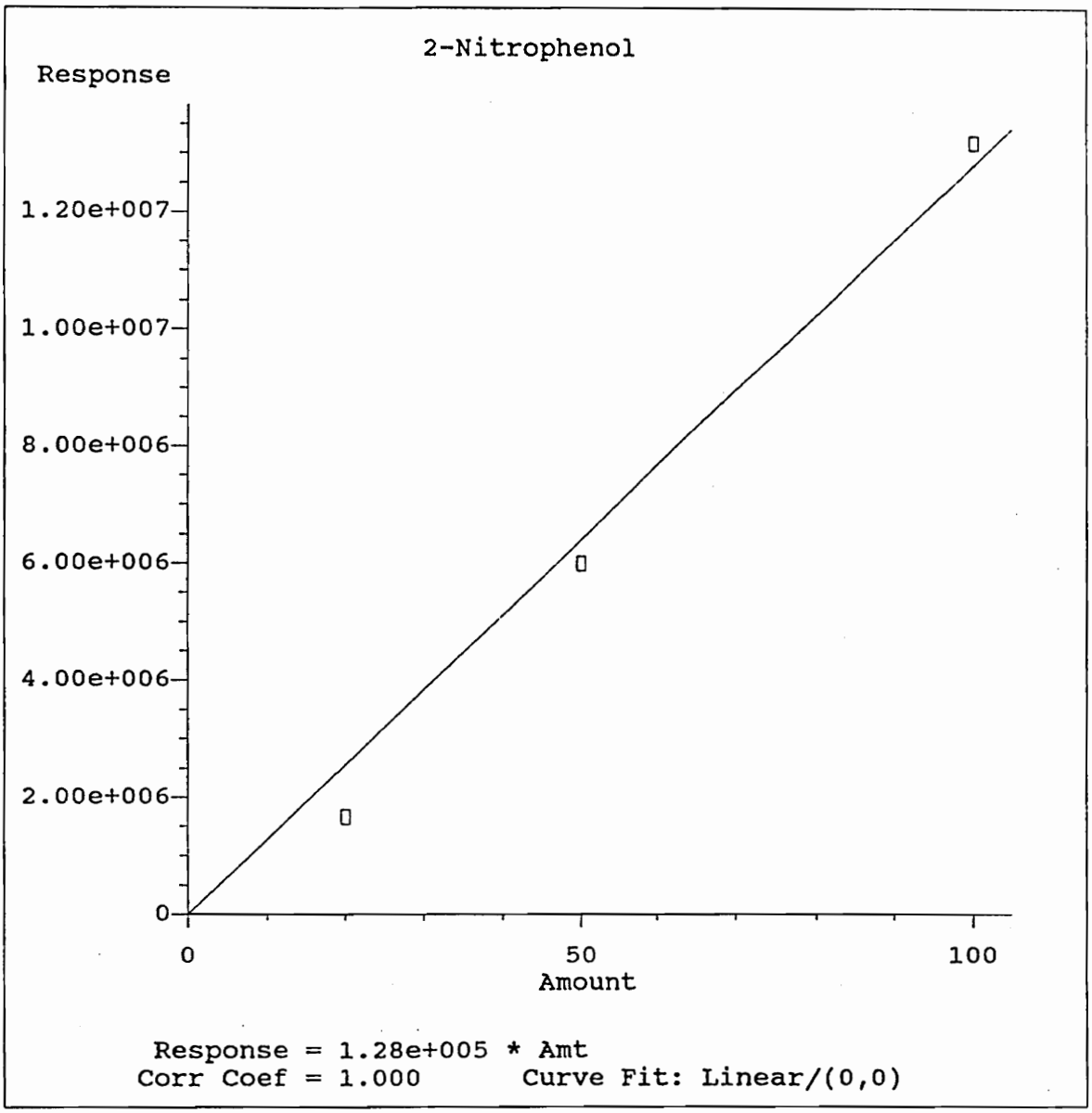
Mass Spectrum of Phenol (Peak #1)



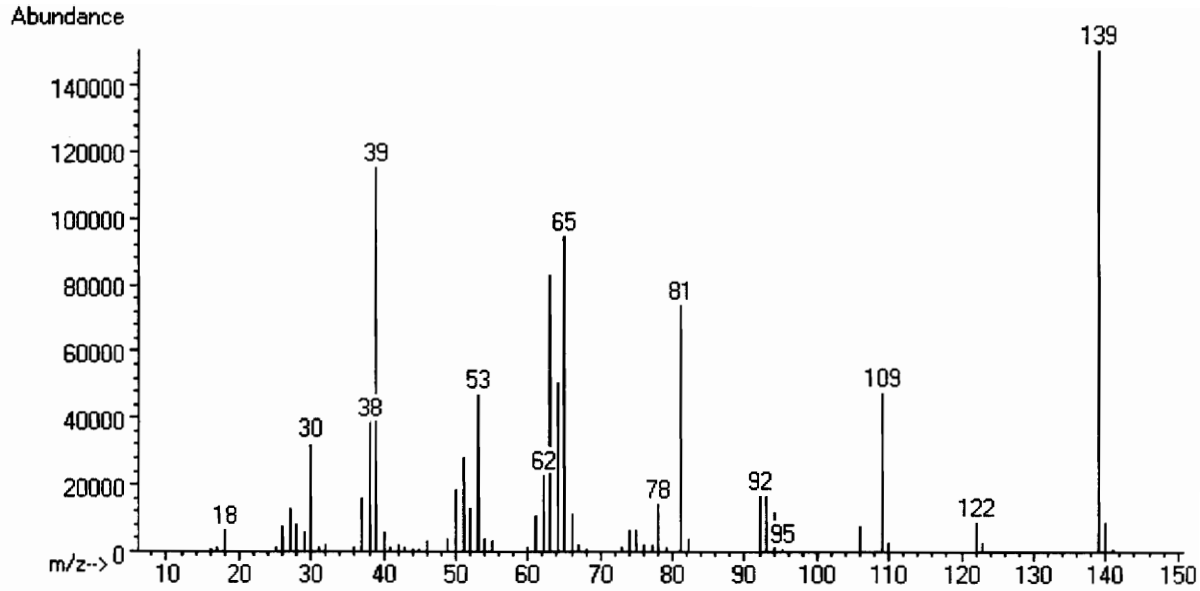
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



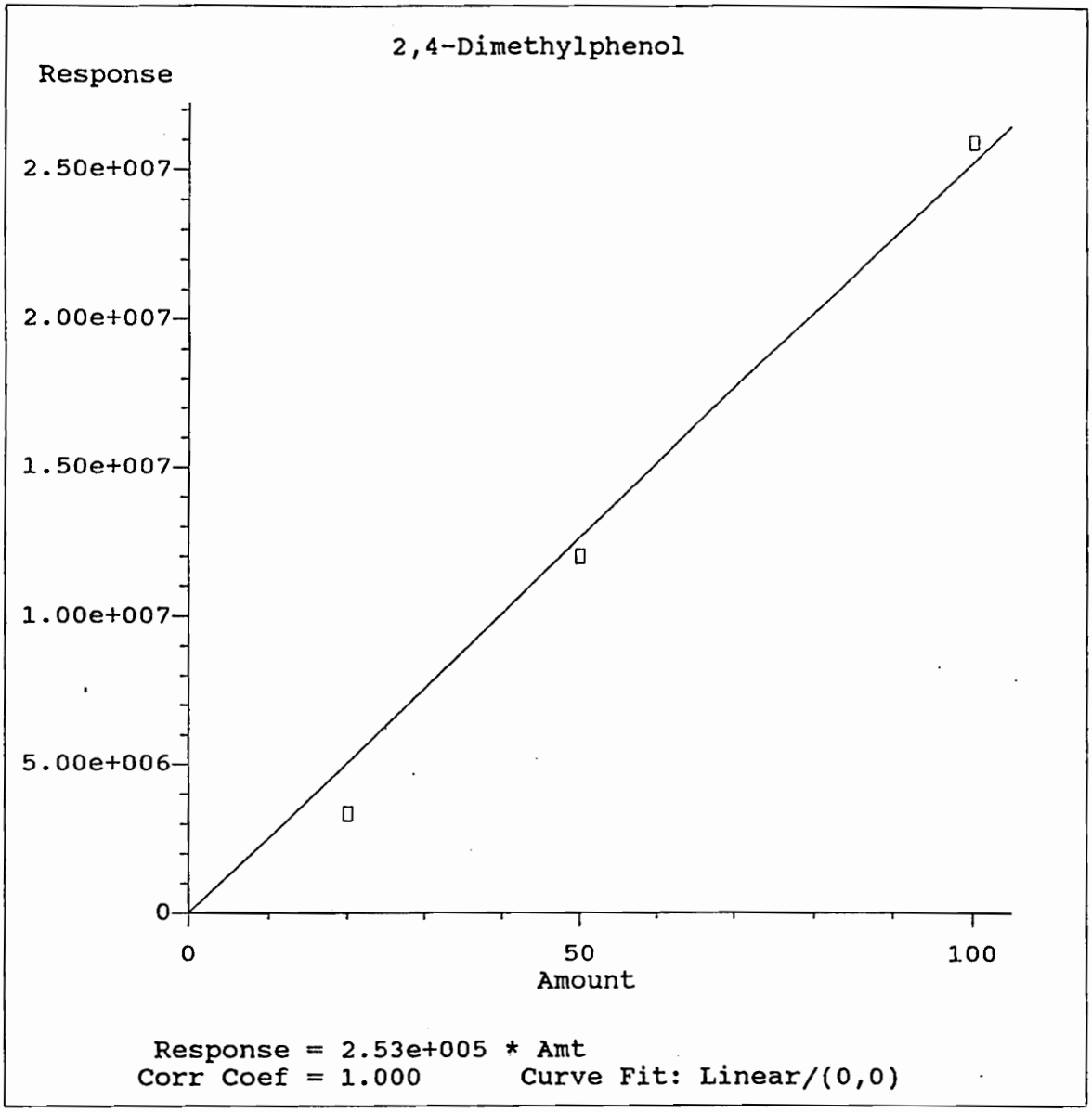
Mass Spectrum of 2-Chlorophenol (Peak #2)



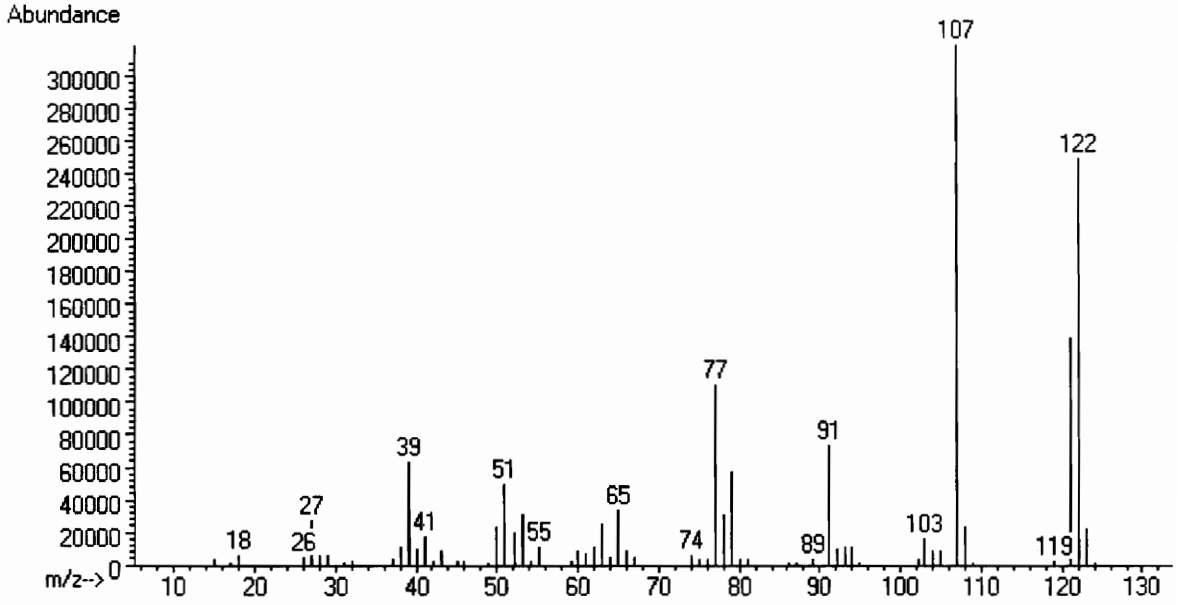
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



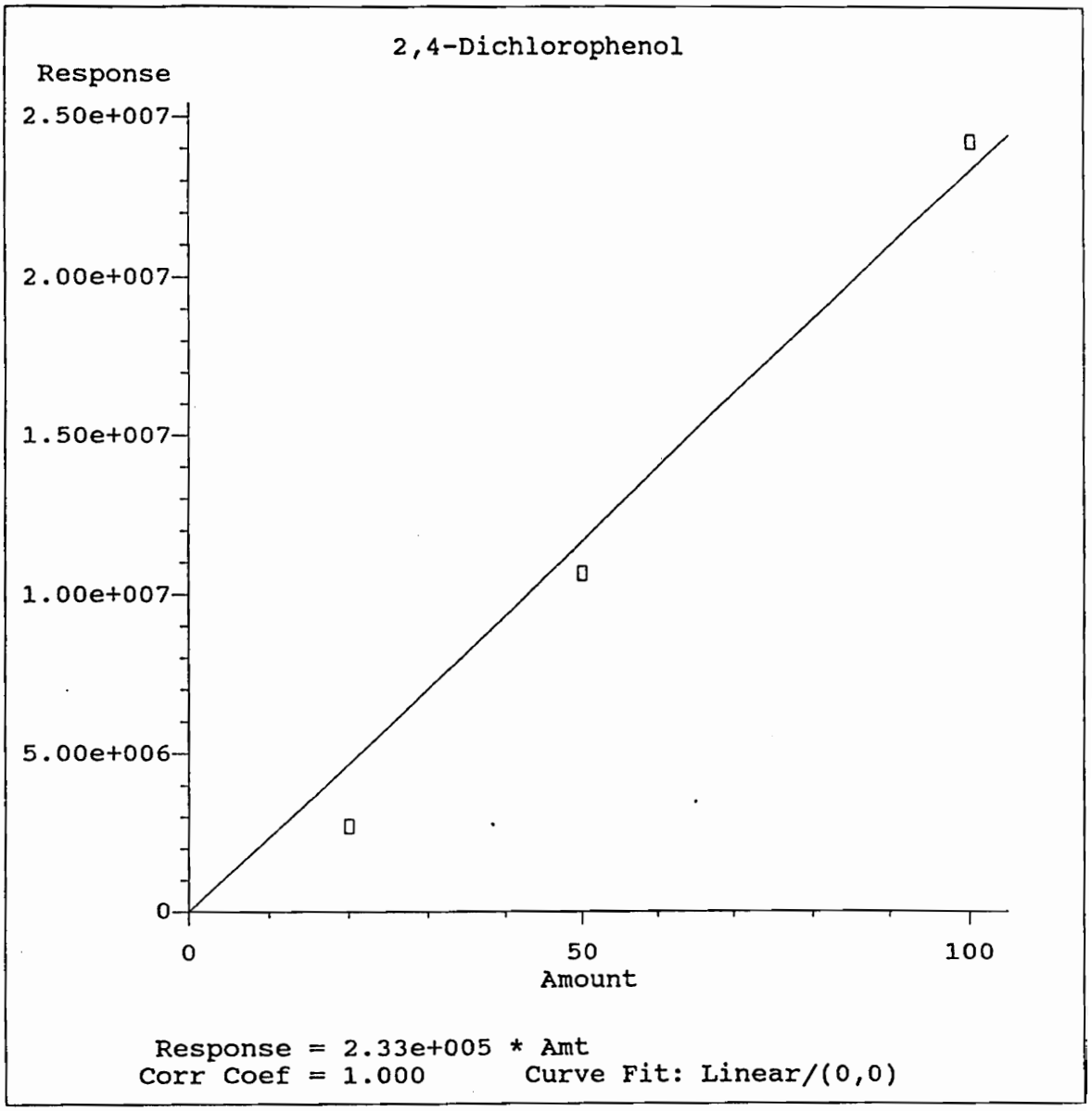
Mass Spectrum of 2-Nitrophenol (Peak #3)



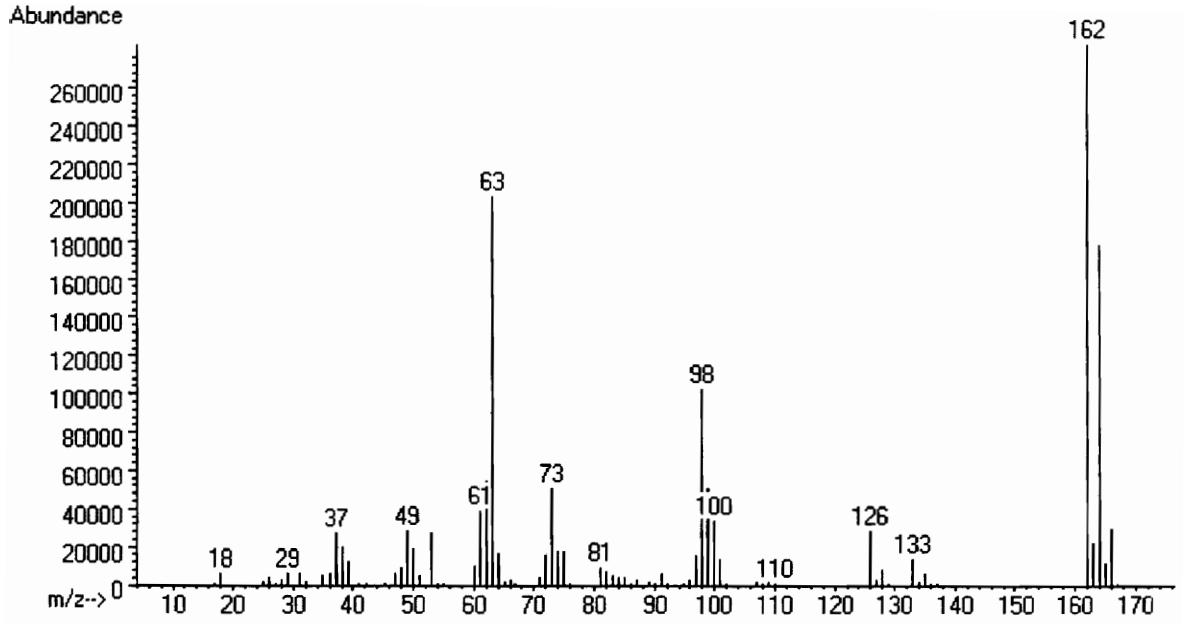
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



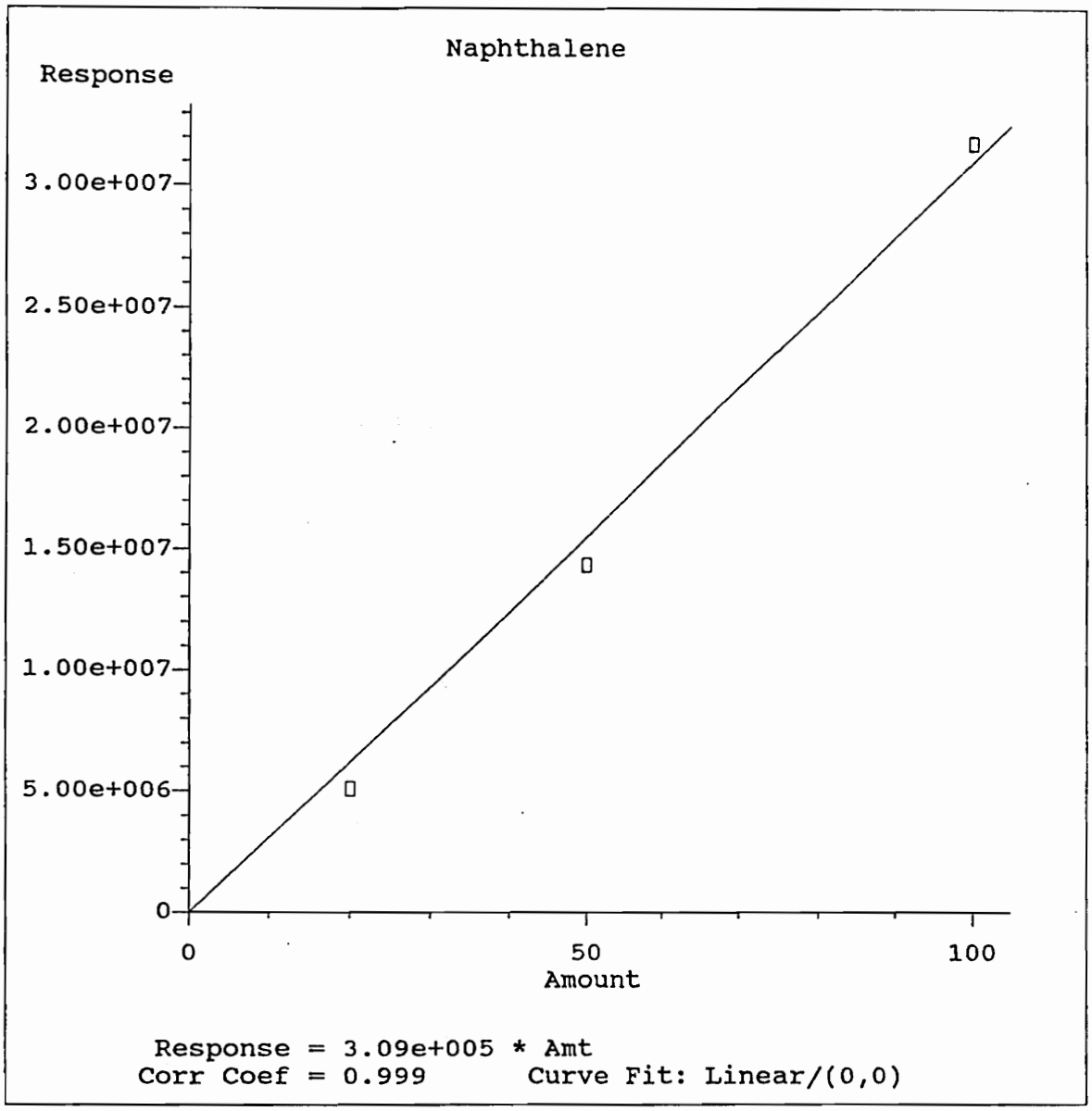
Mass Spectrum of 2,4-Dimethylphenol (Peak #4)



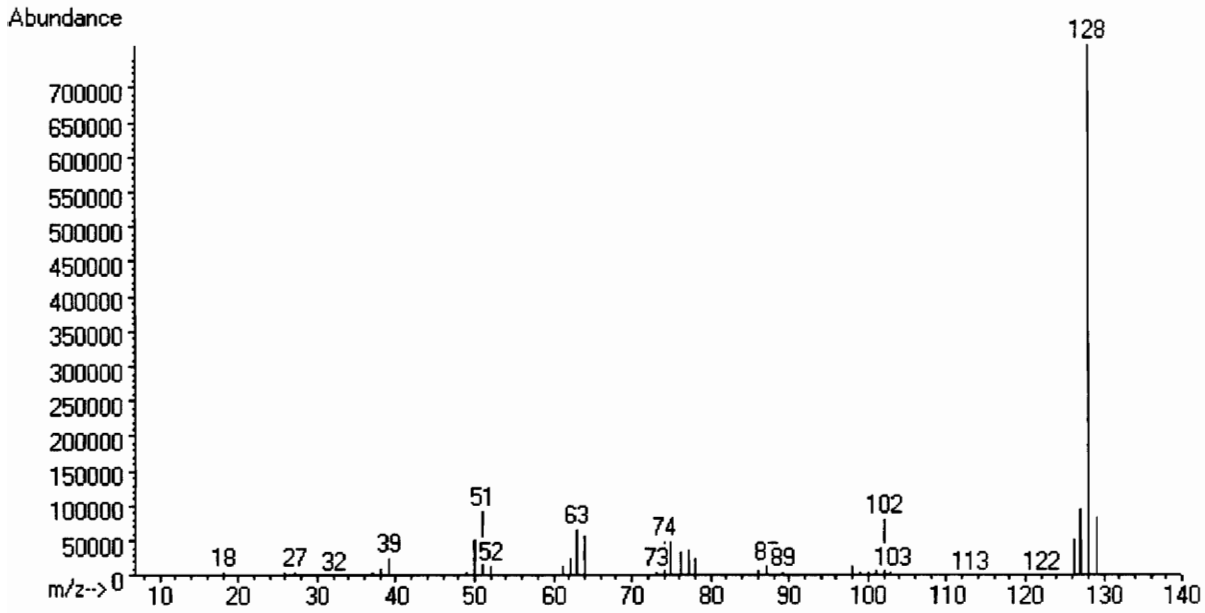
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



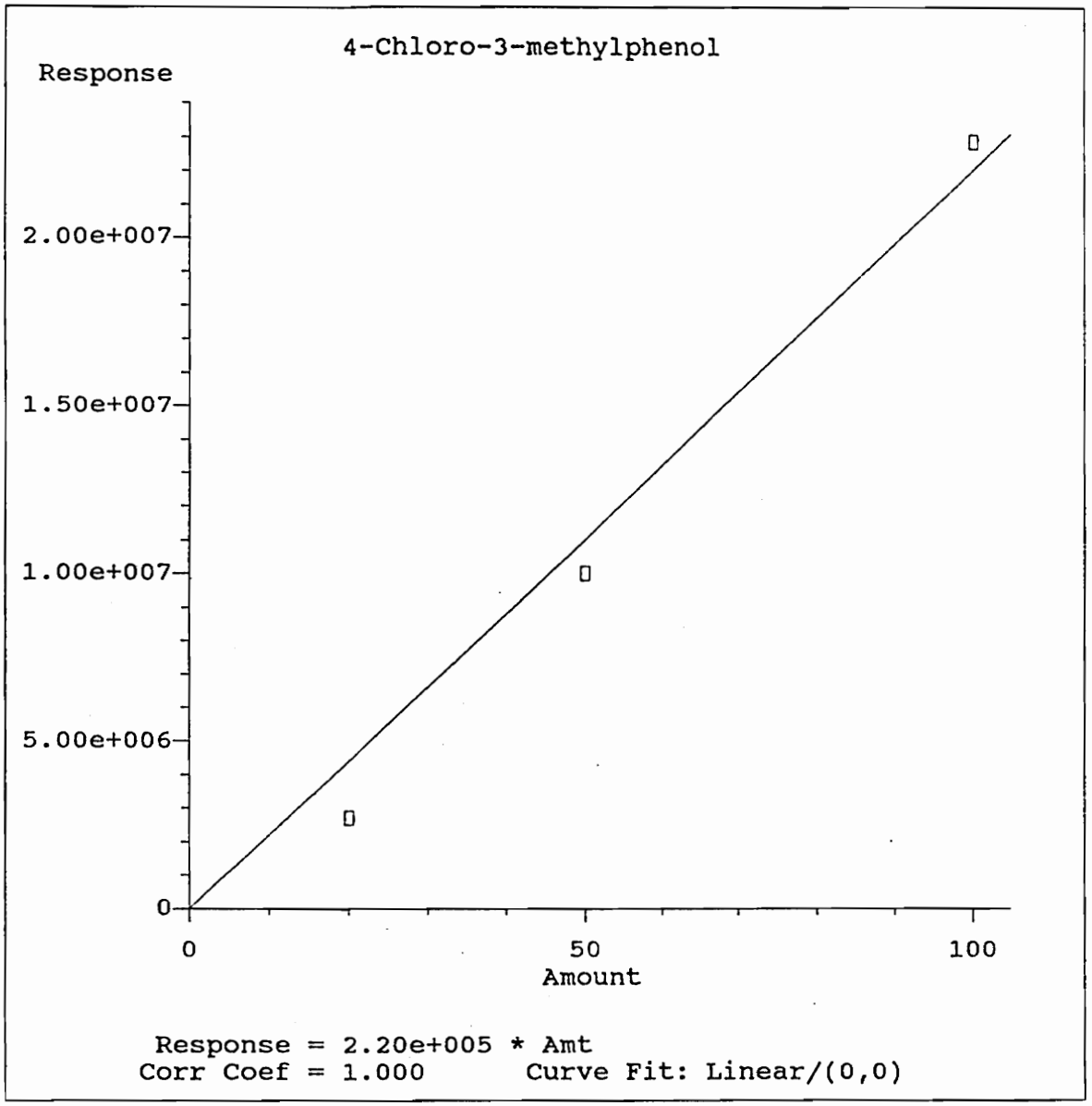
Mass Spectrum of 2,4-Dichlorophenol (Peak #5)



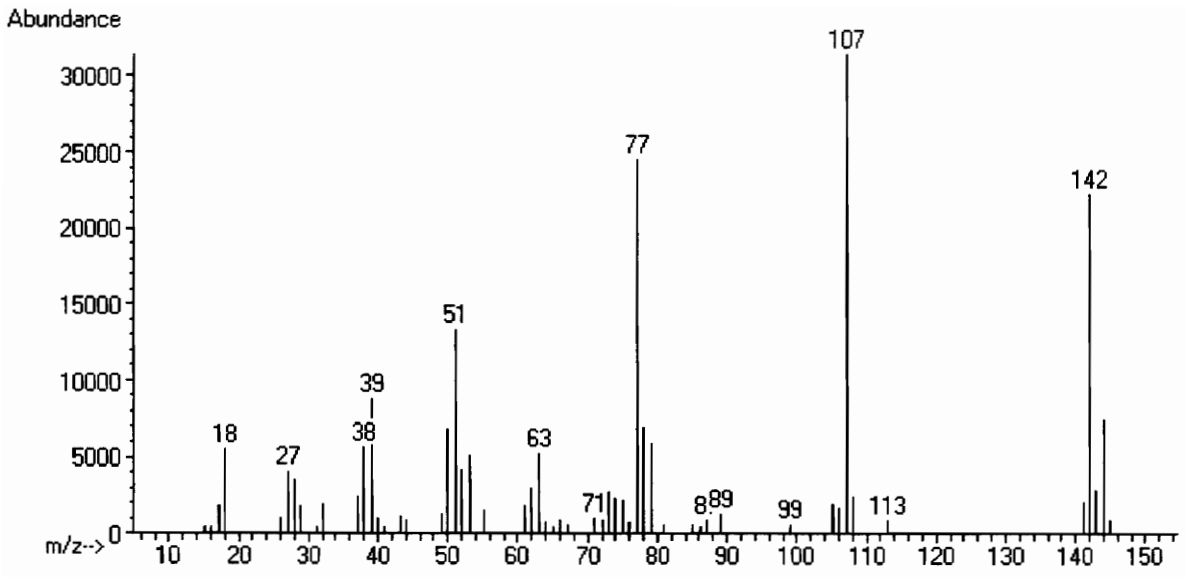
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



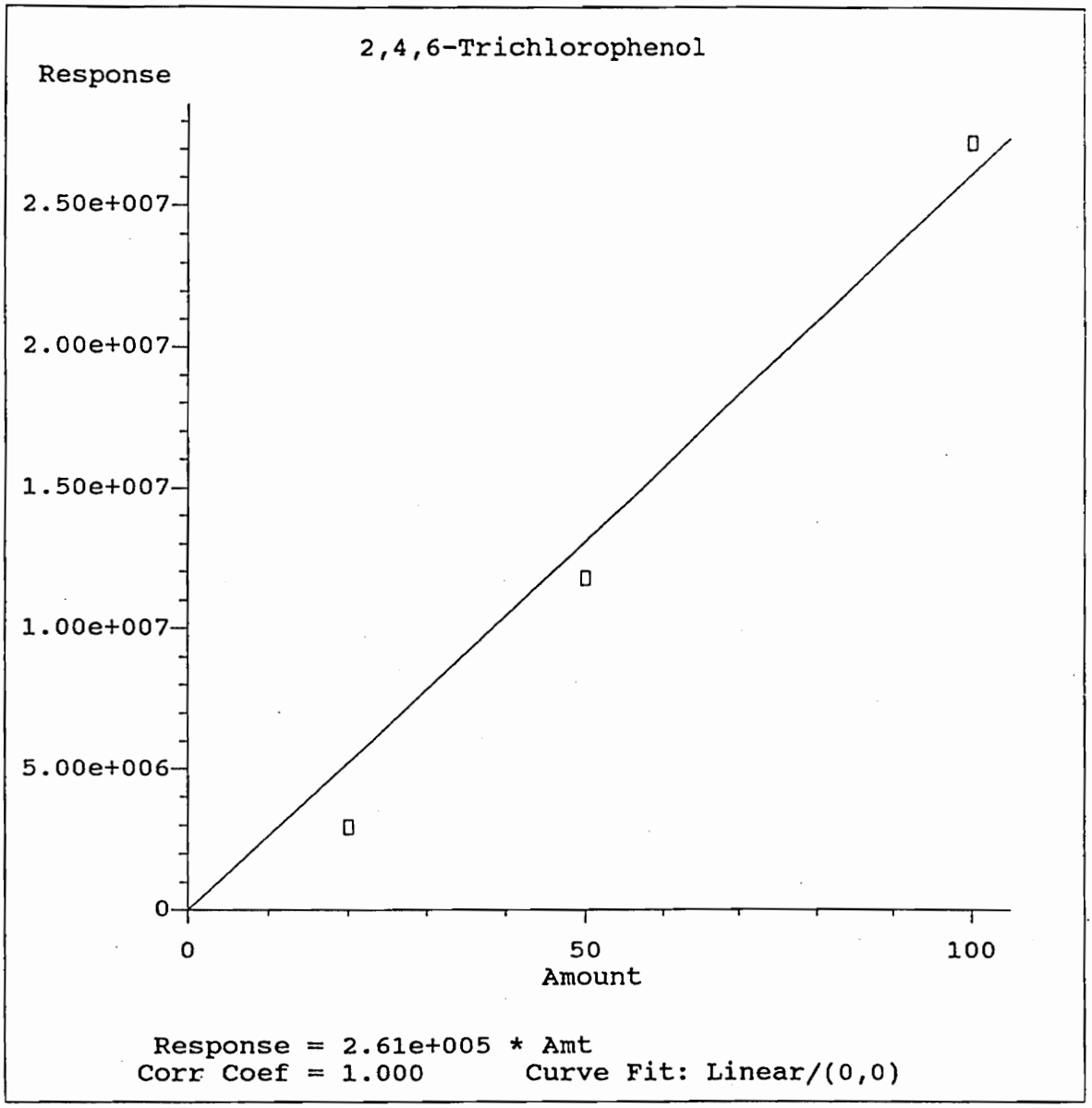
Mass Spectrum of Naphthalene (Peak #6)



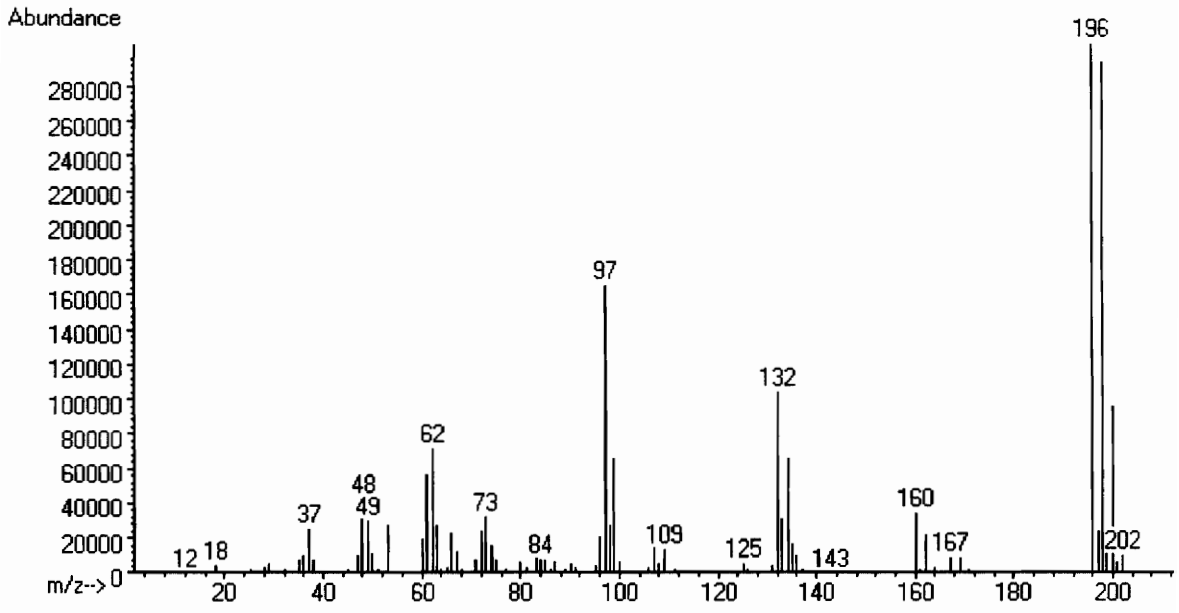
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



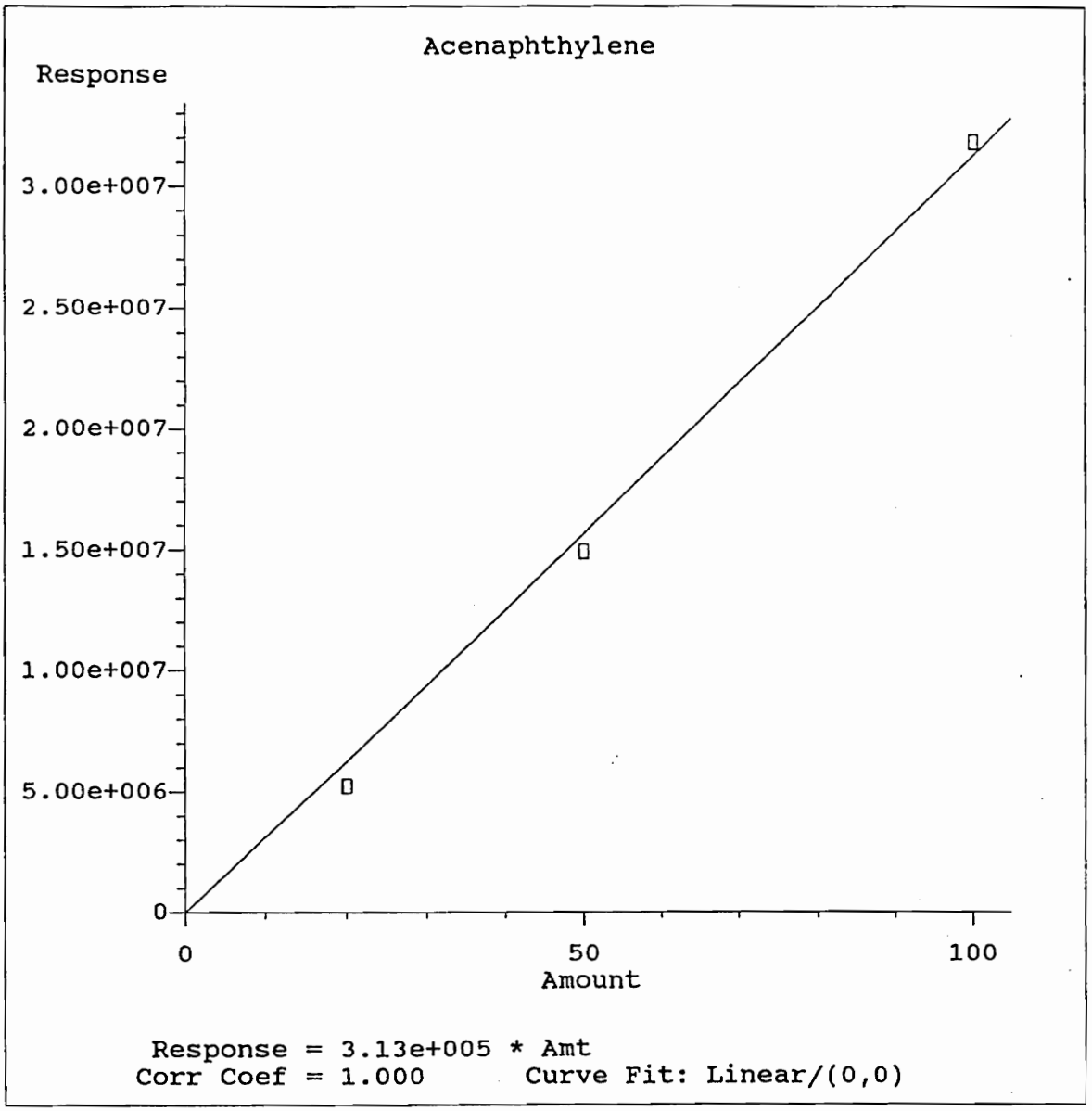
Mass Spectrum of 4-Chloro-3-methylphenol (Peak #7)



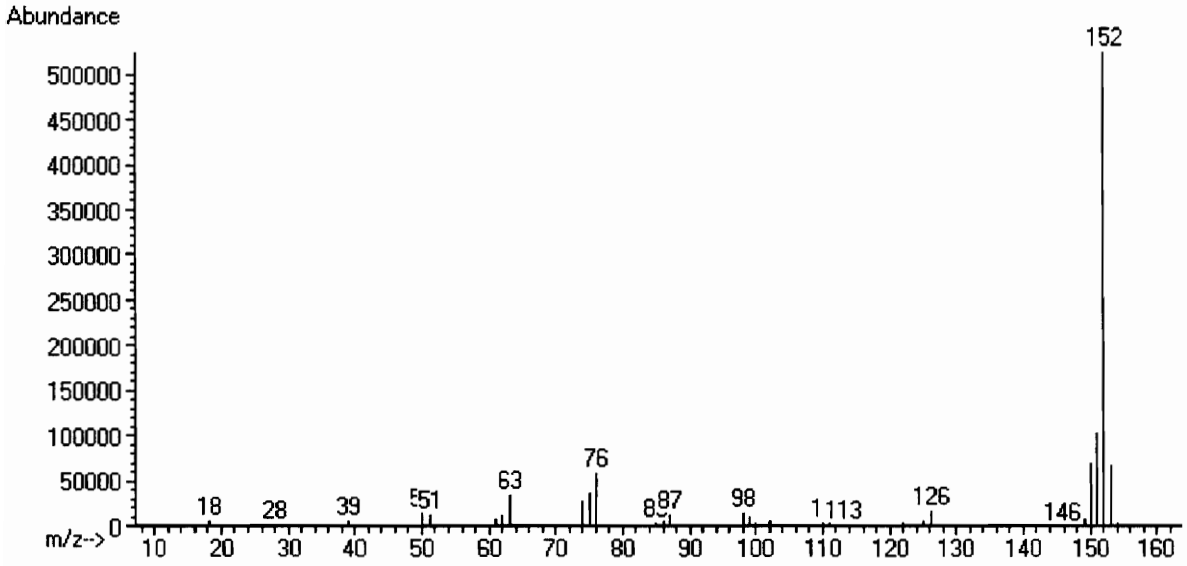
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



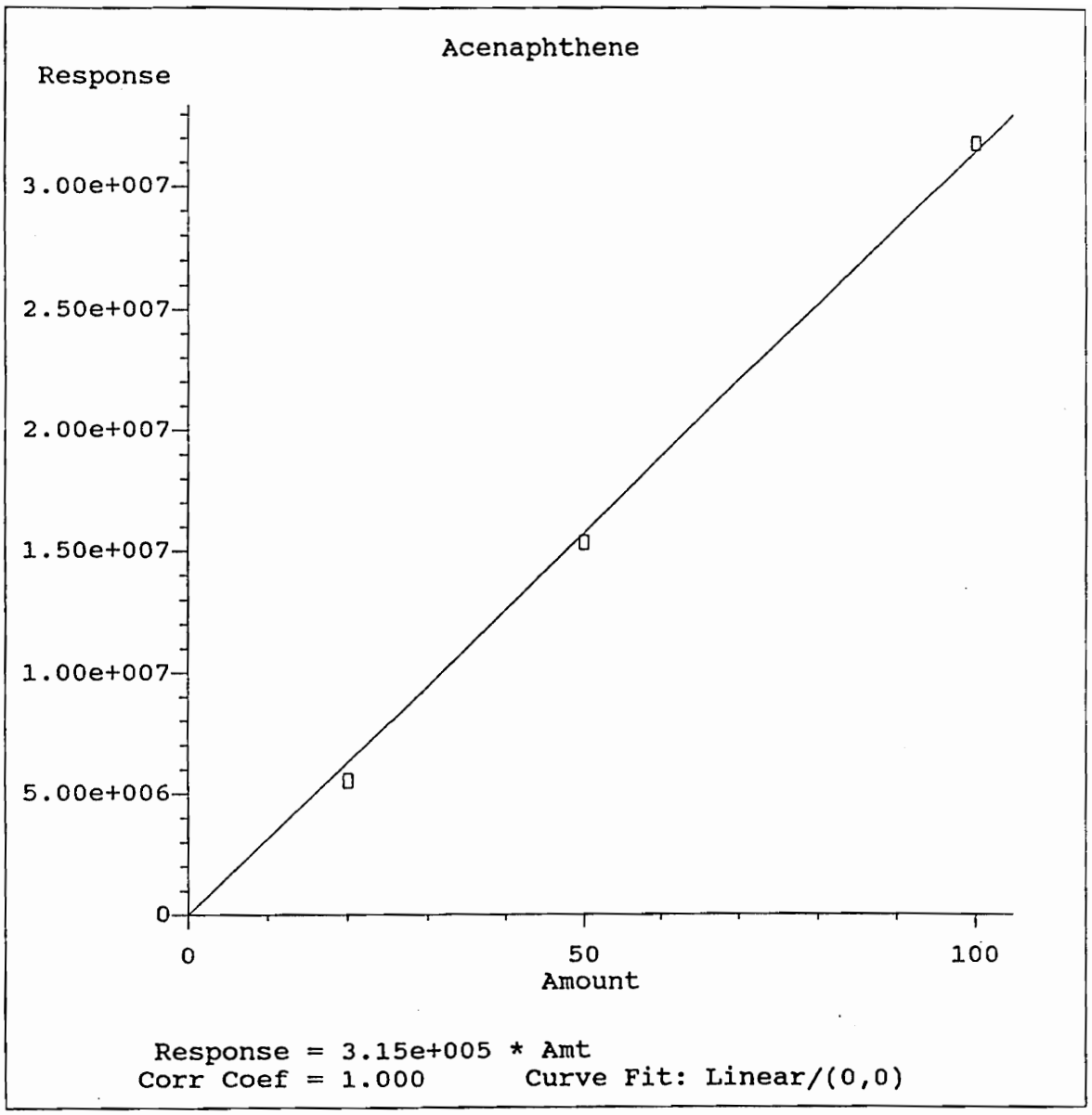
Mass Spectrum of 2,4,6-Trichlorophenol (Peak #8)



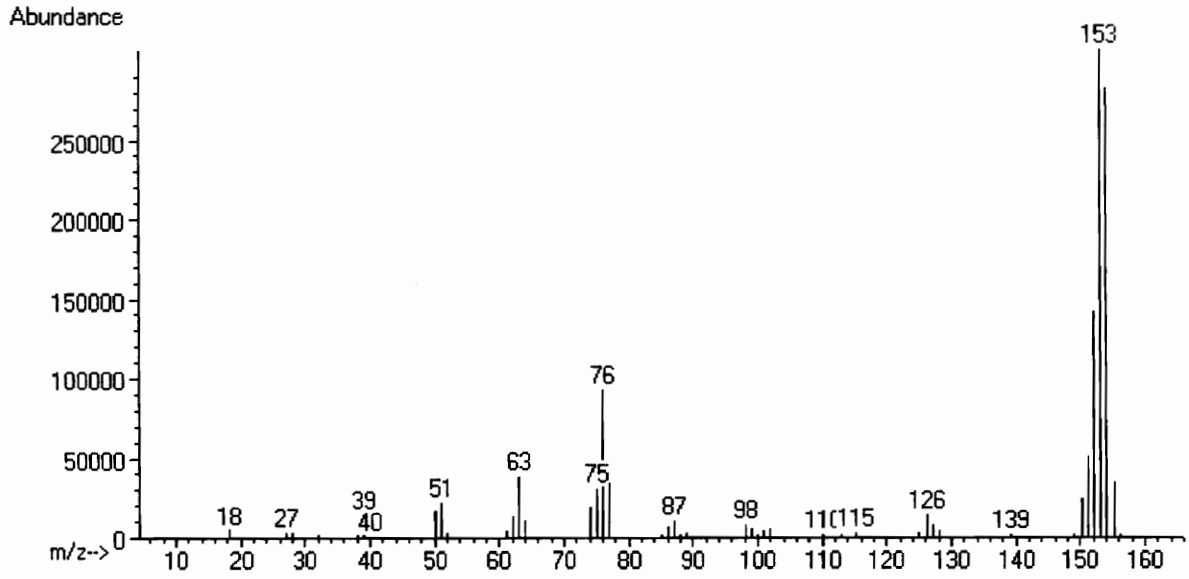
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



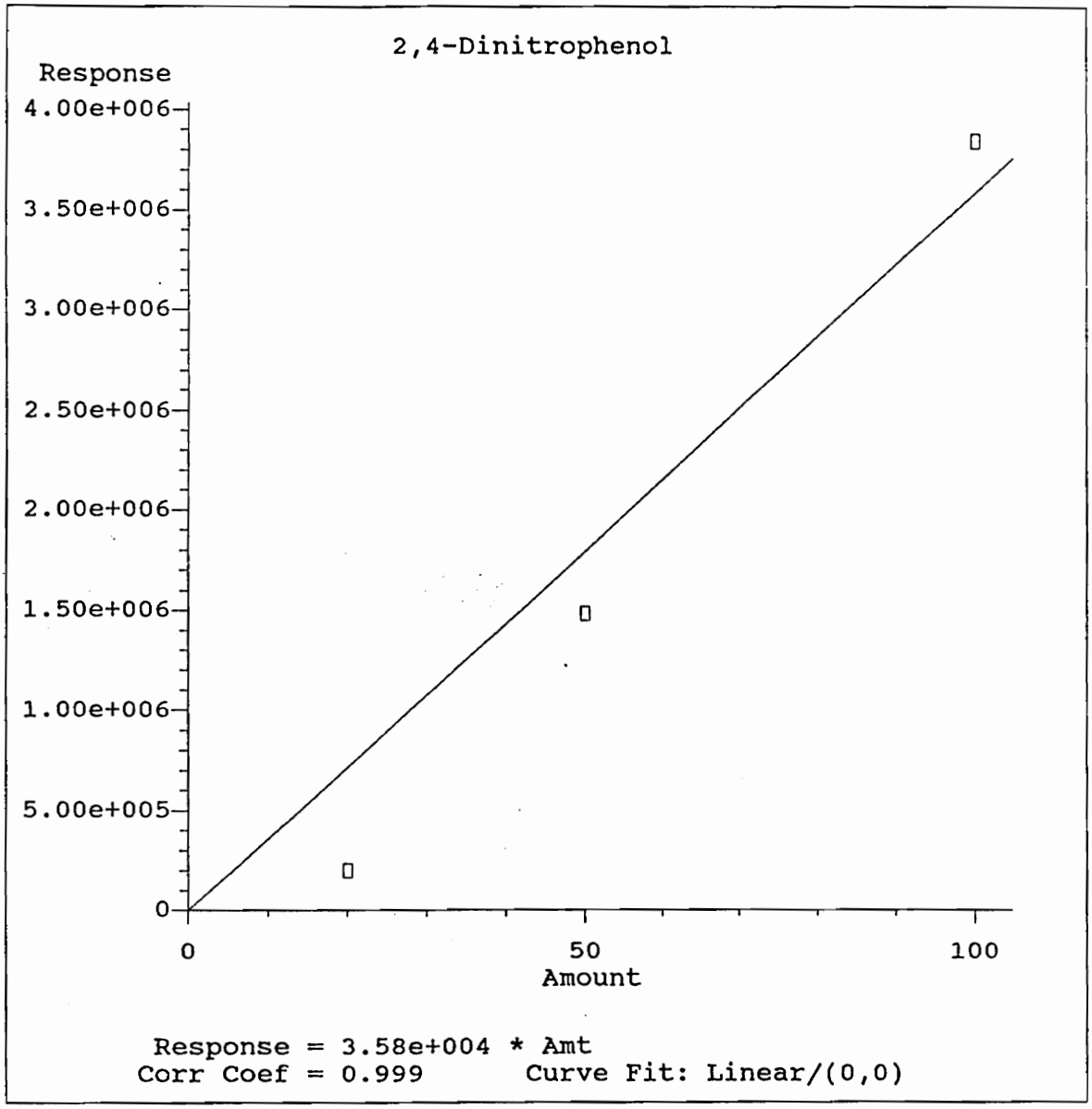
Mass Spectrum of Acenaphthylene (Peak #9)



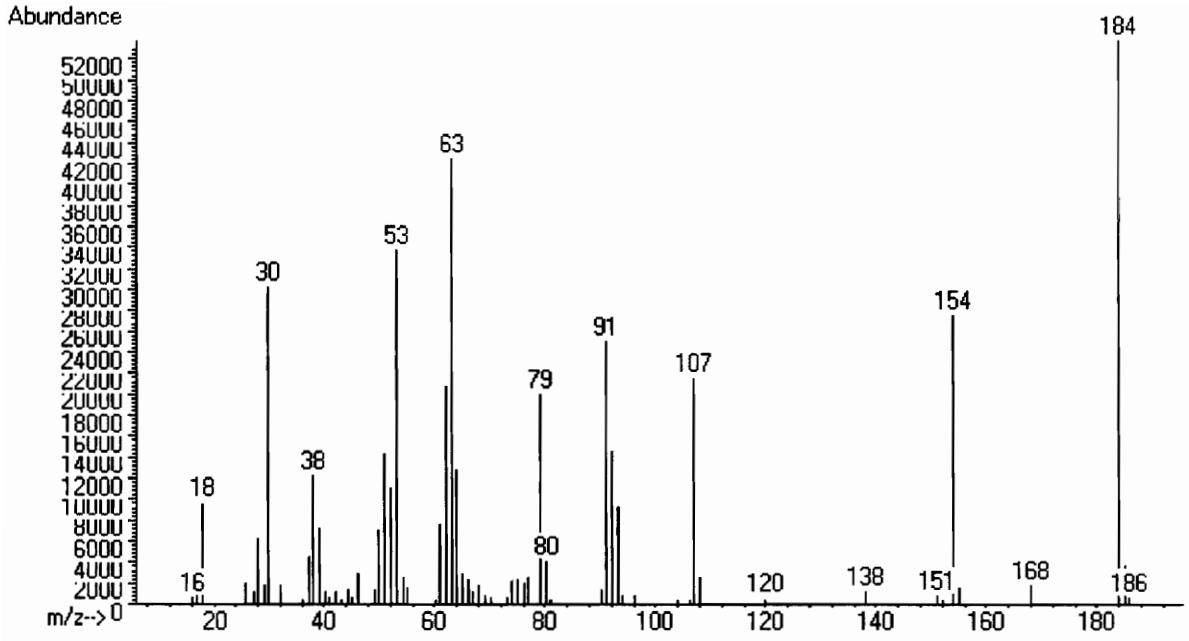
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



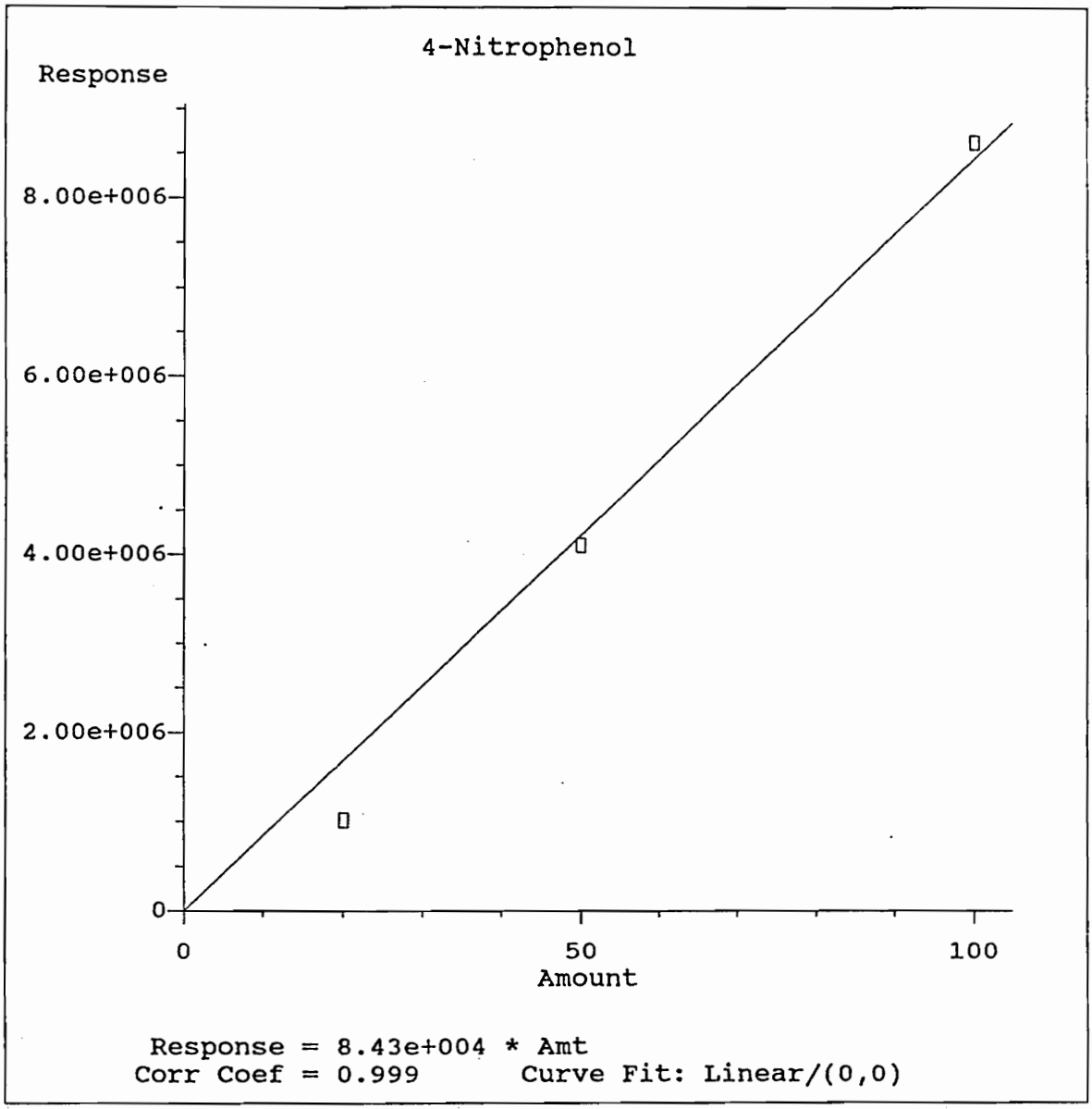
Mass Spectrum of Acenapthene (Peak #10)



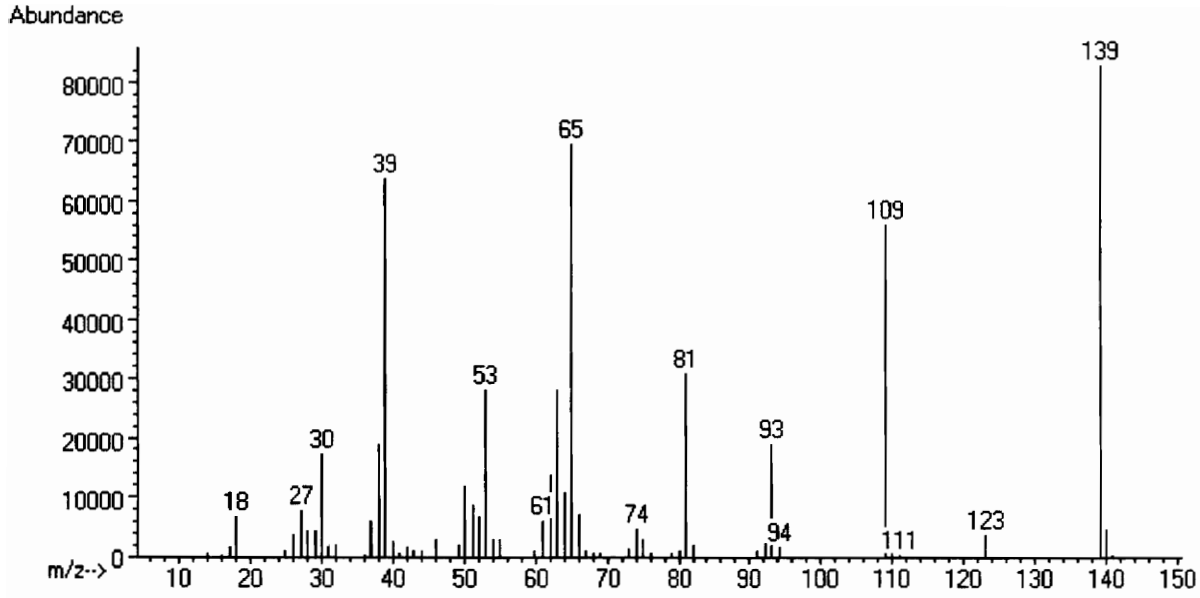
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



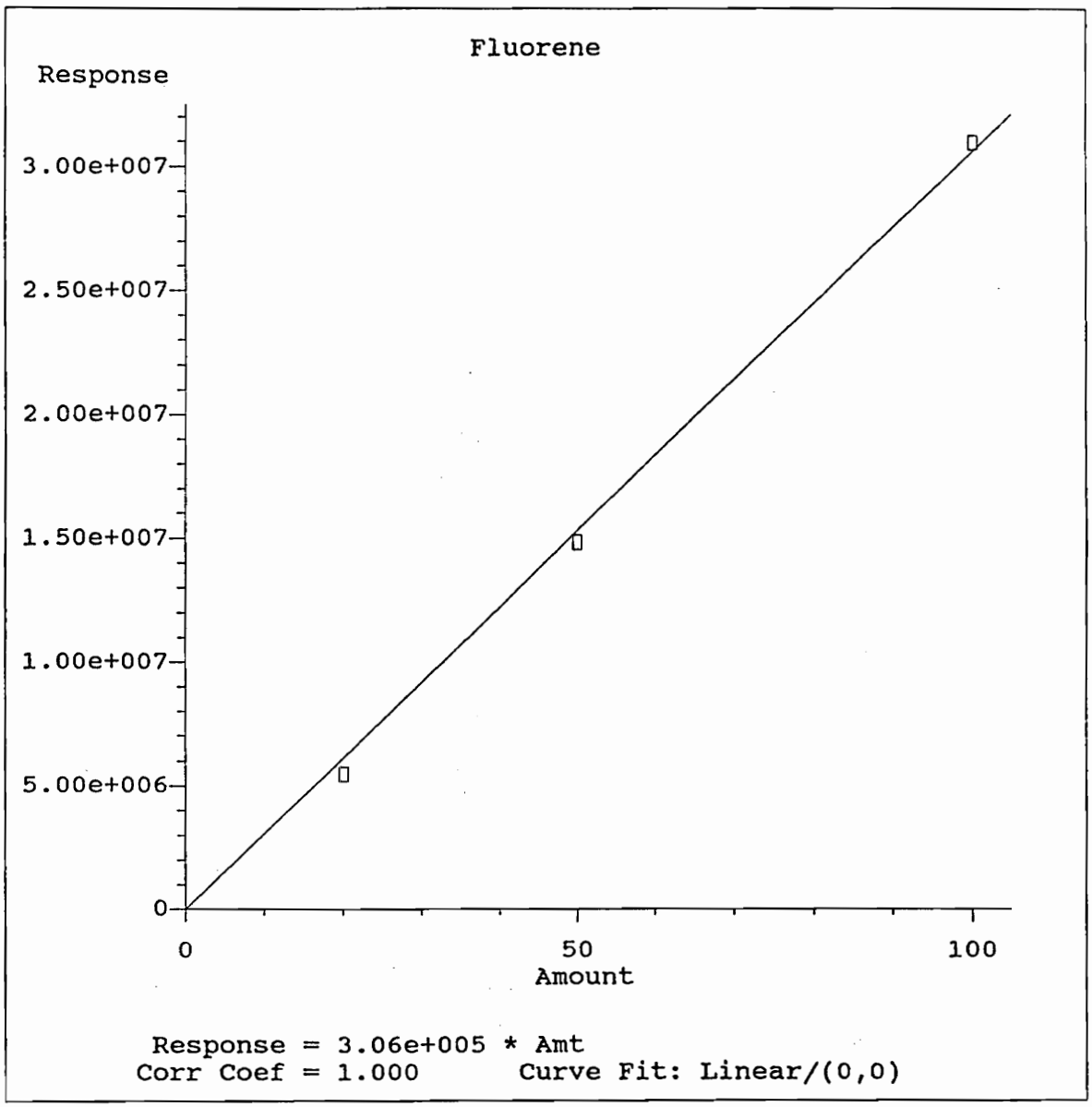
Mass Spectrum of Dinitrophenol (Peak #11)



Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



Mass Spectrum of 4-Nitrophenol (Peak #12)



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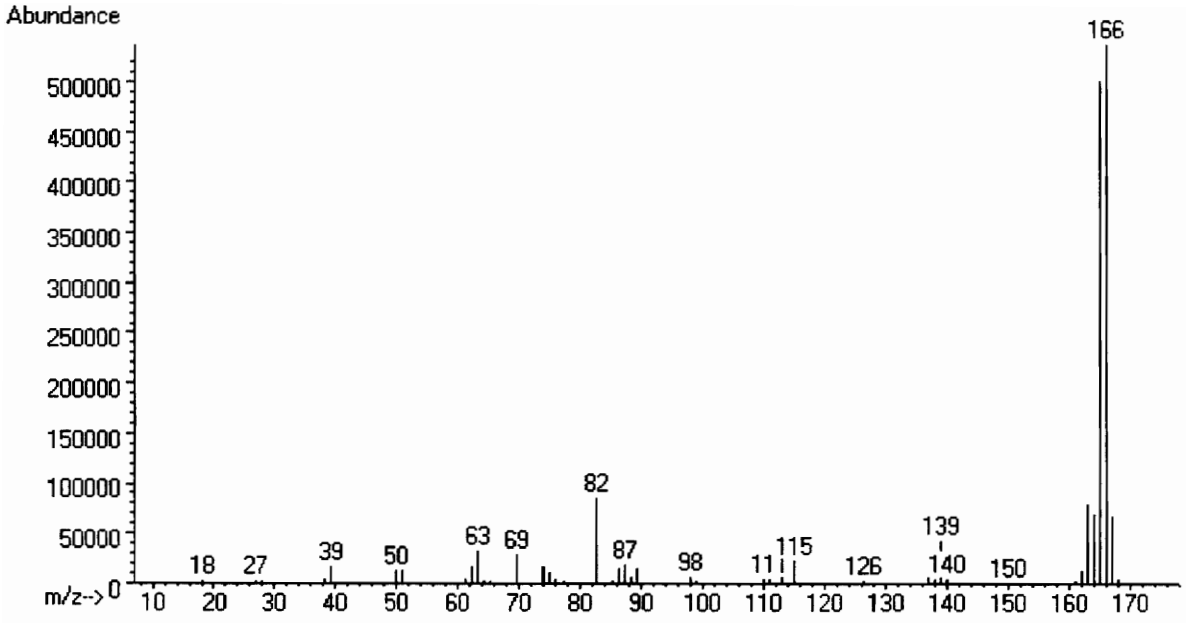
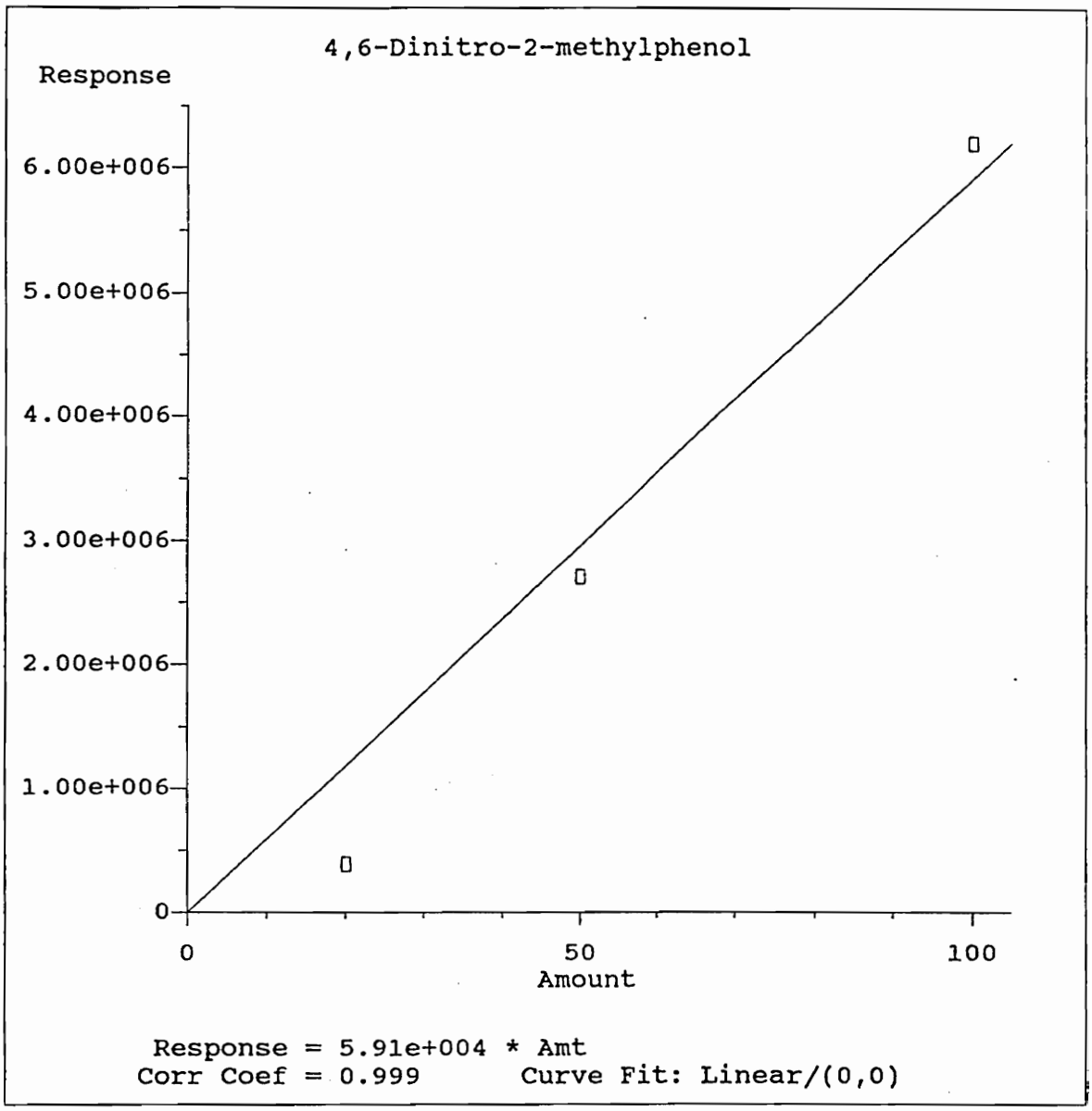
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



Mass Spectrum of Fluorene (Peak #13)



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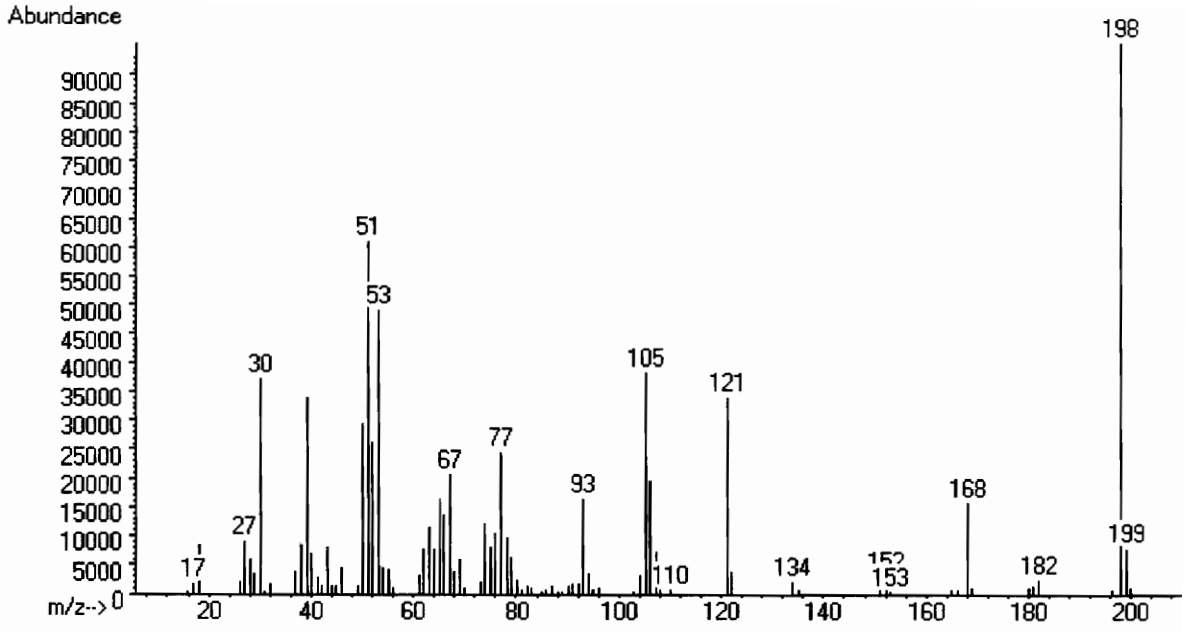
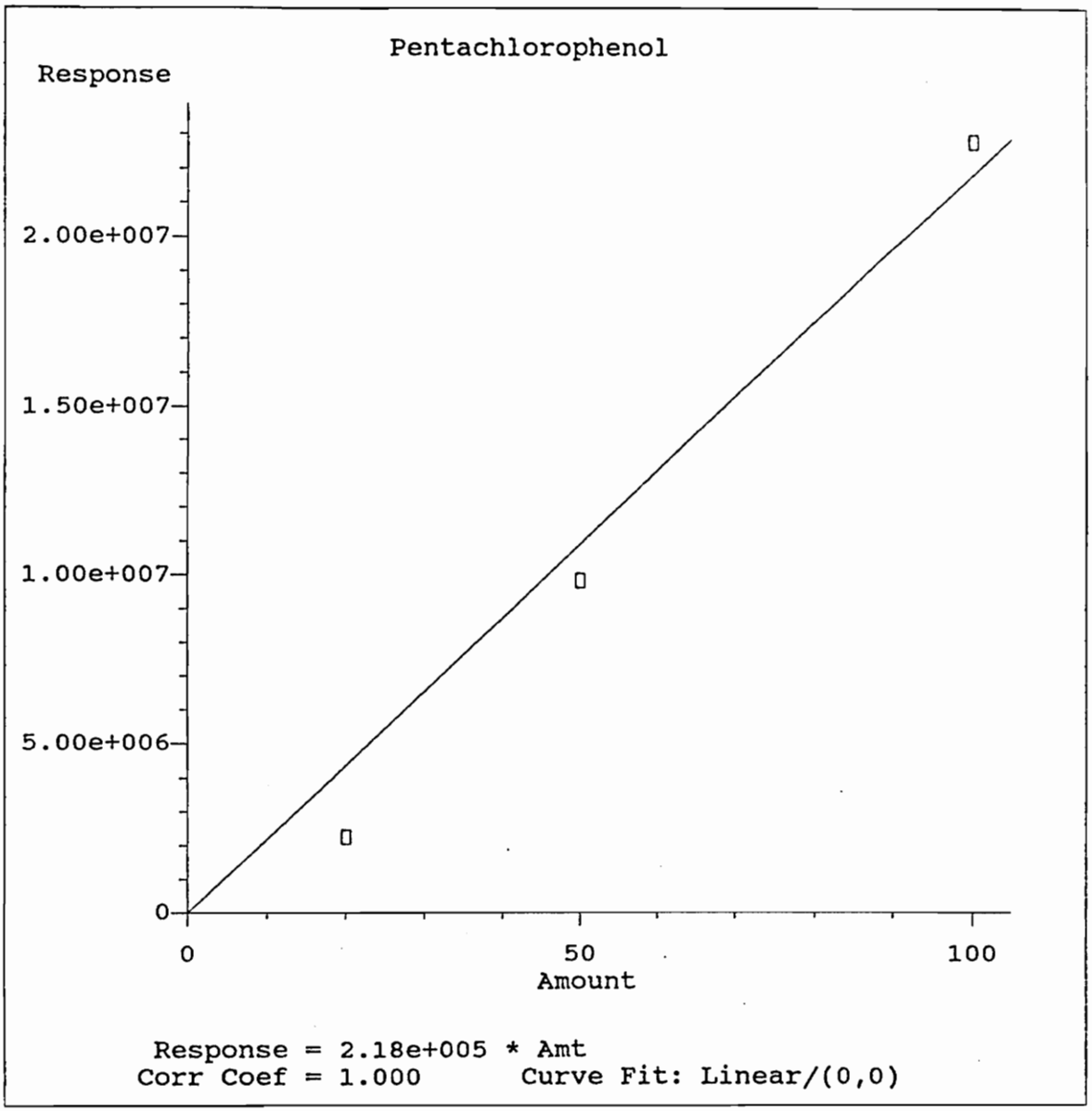
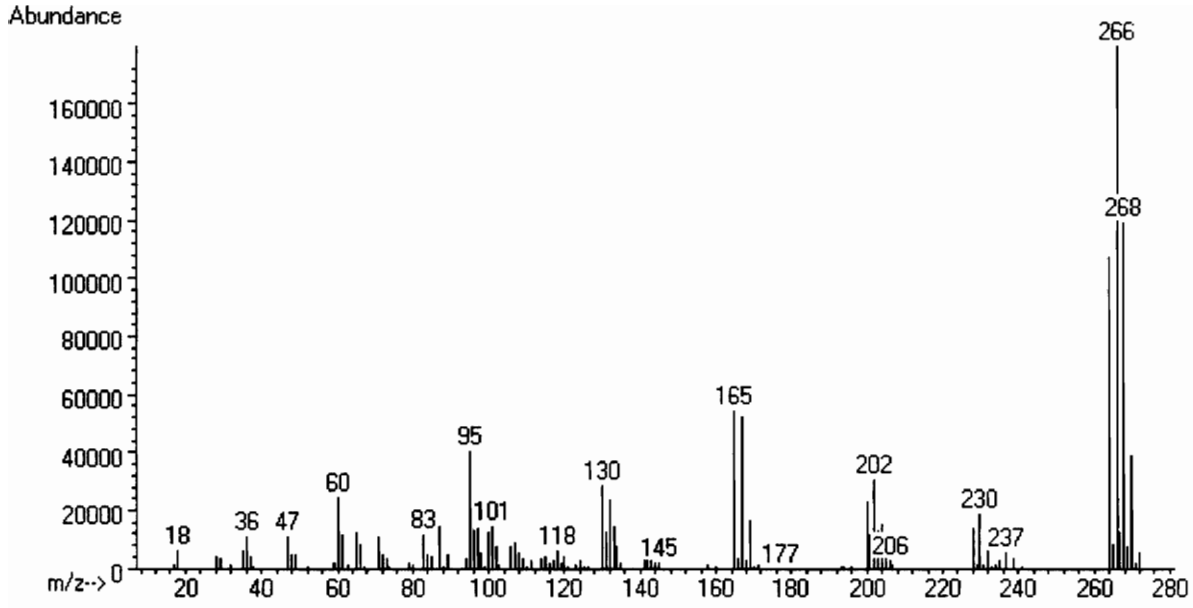


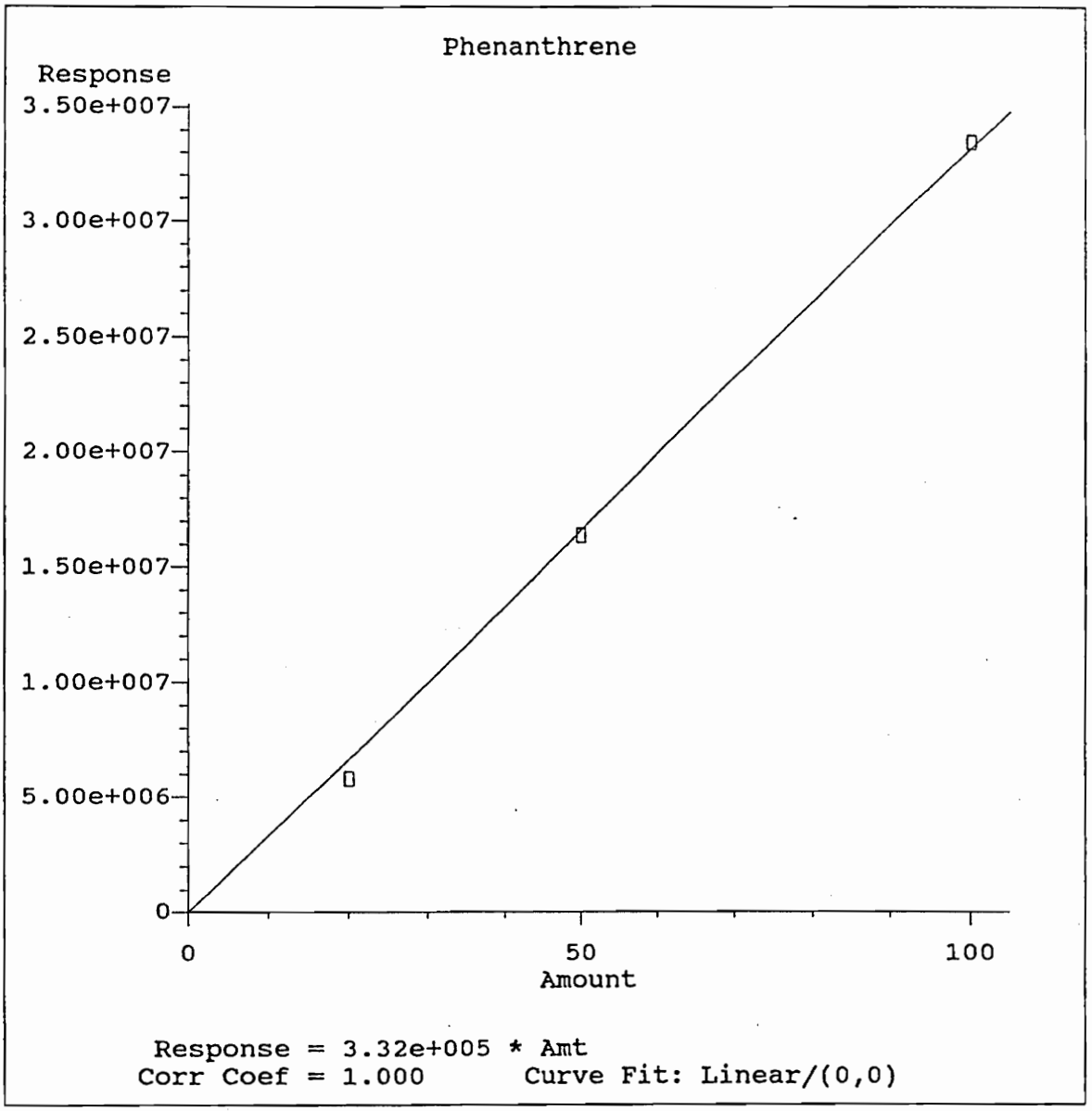
Mass Spectrum of 2-Methyl-4,6-dinitrophenol (Peak #14)



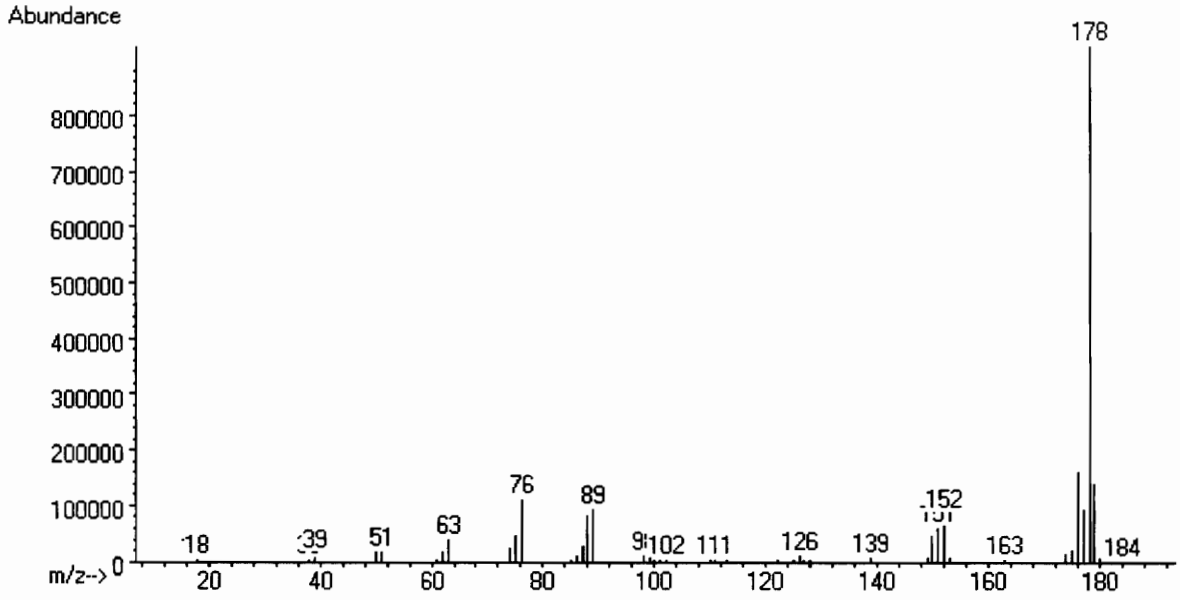
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



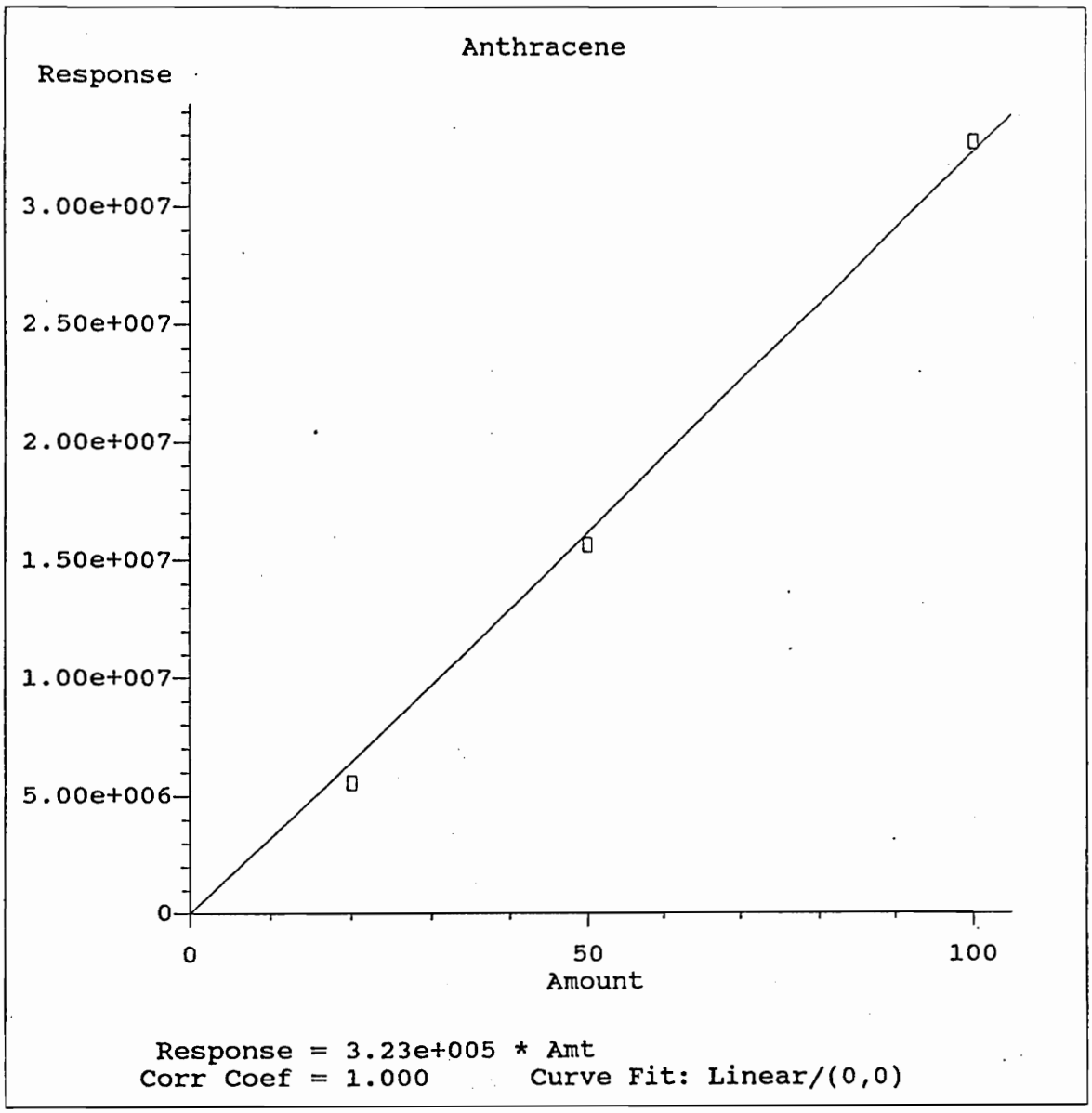
Mass Spectrum of Pentachlorophenol (Peak #15)



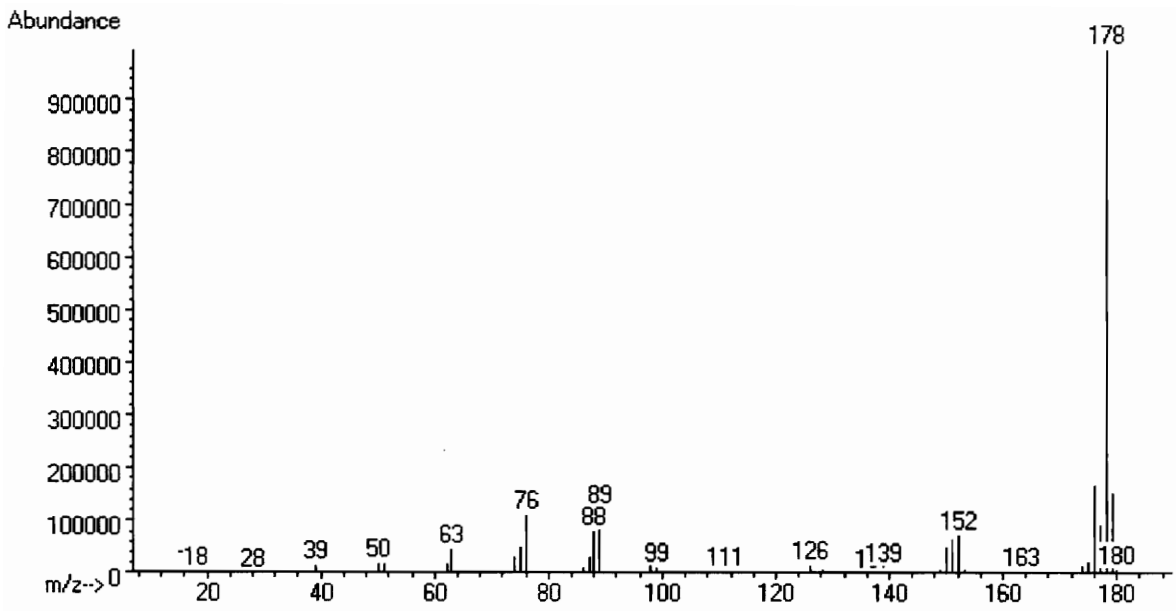
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



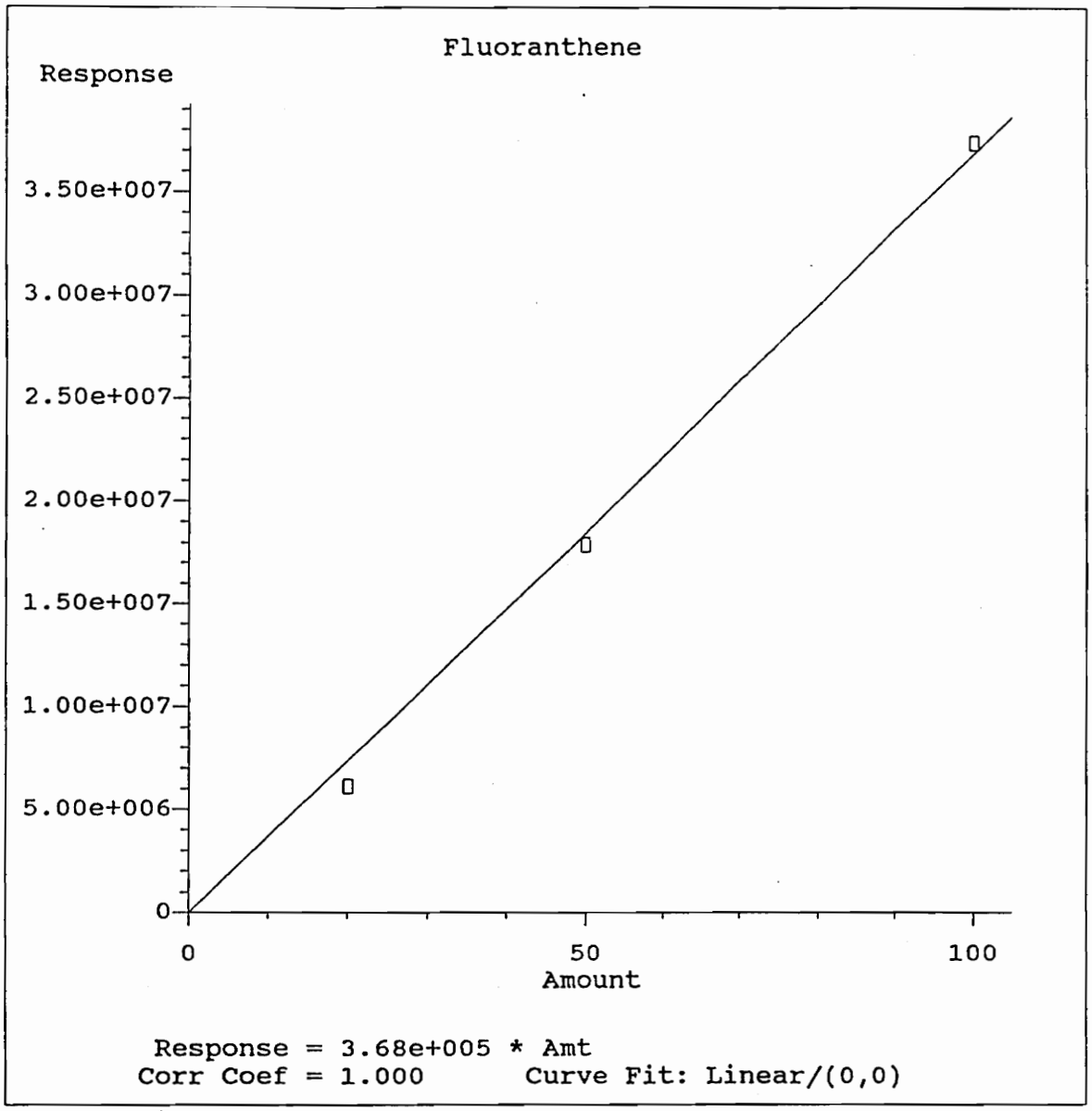
Mass Spectrum of Phenanthrene (Peak #16)



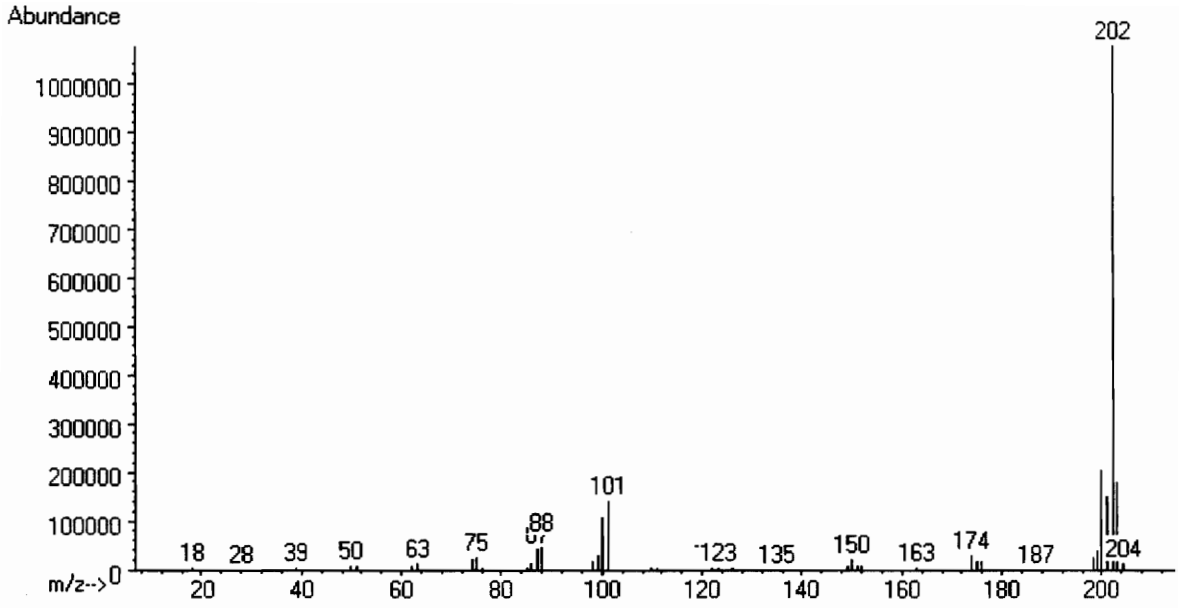
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



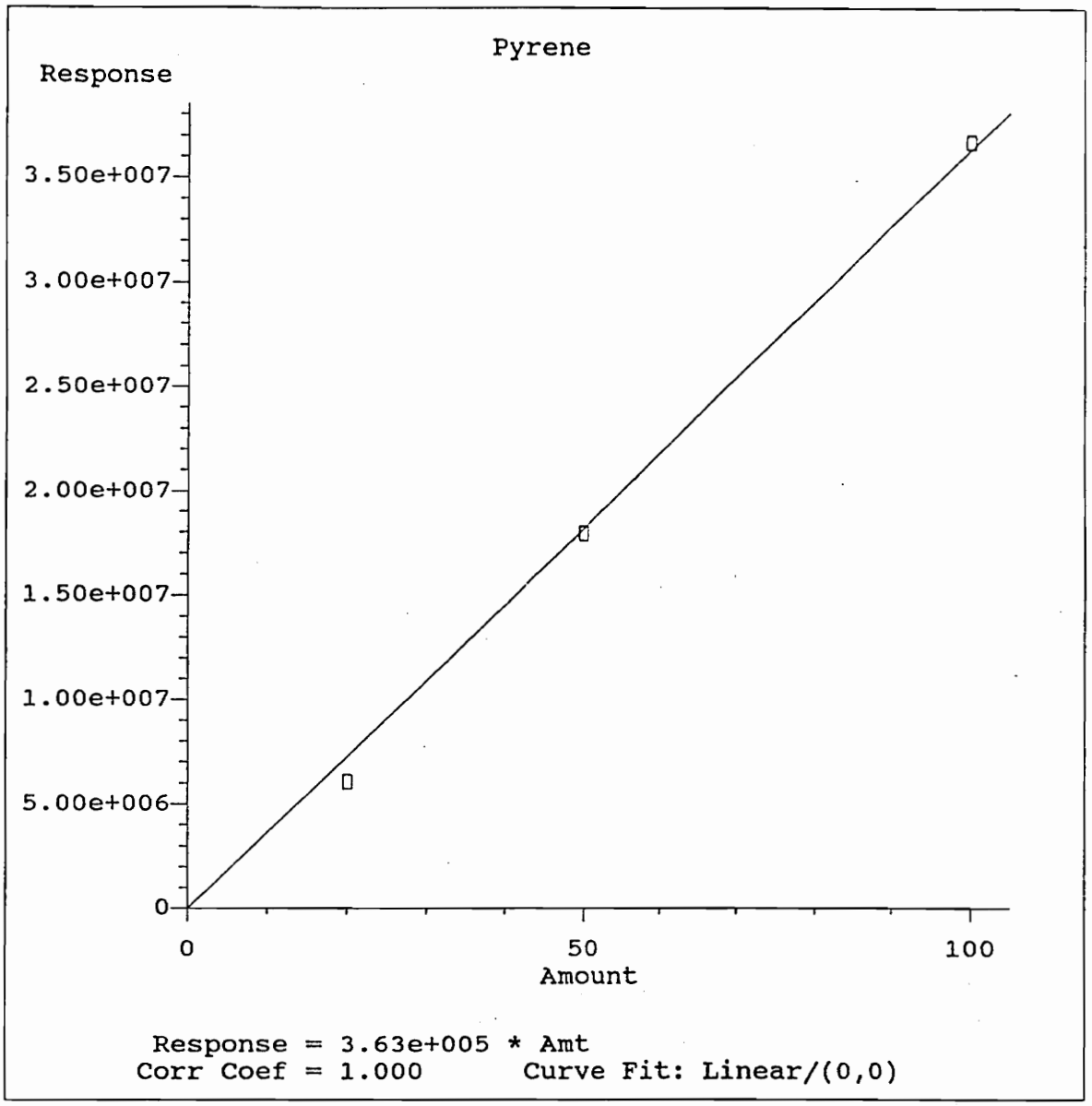
Mass Spectrum of Anthracene (Peak #17)



Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



Mass Spectrum of Fluoranthrene (Peak #18)



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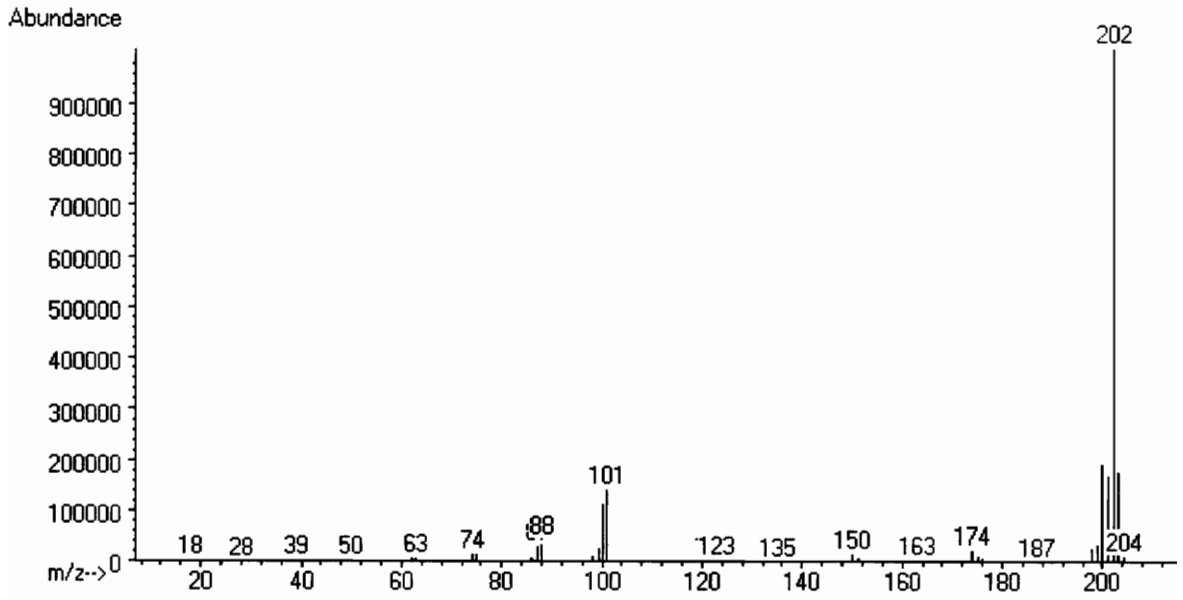
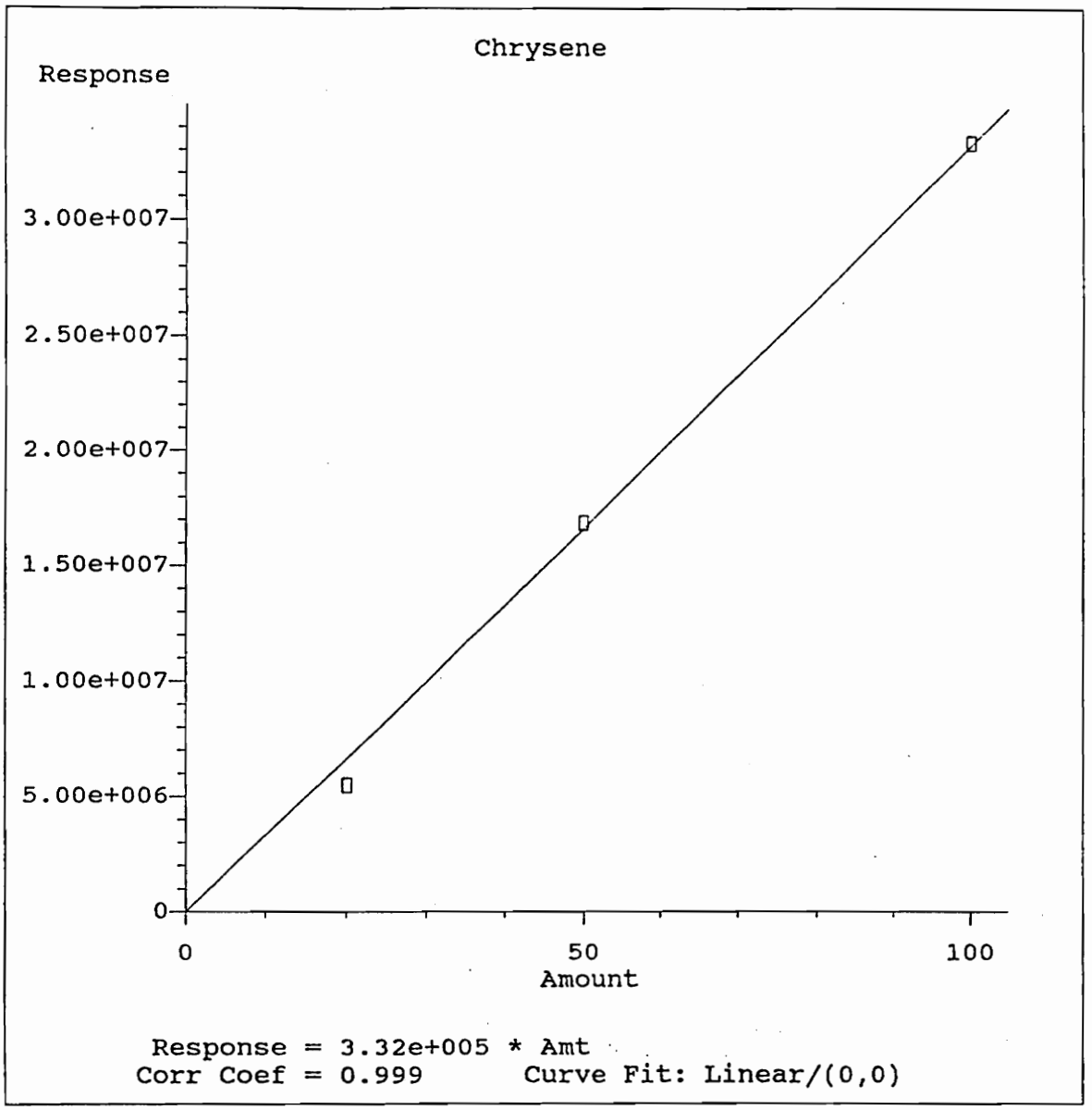
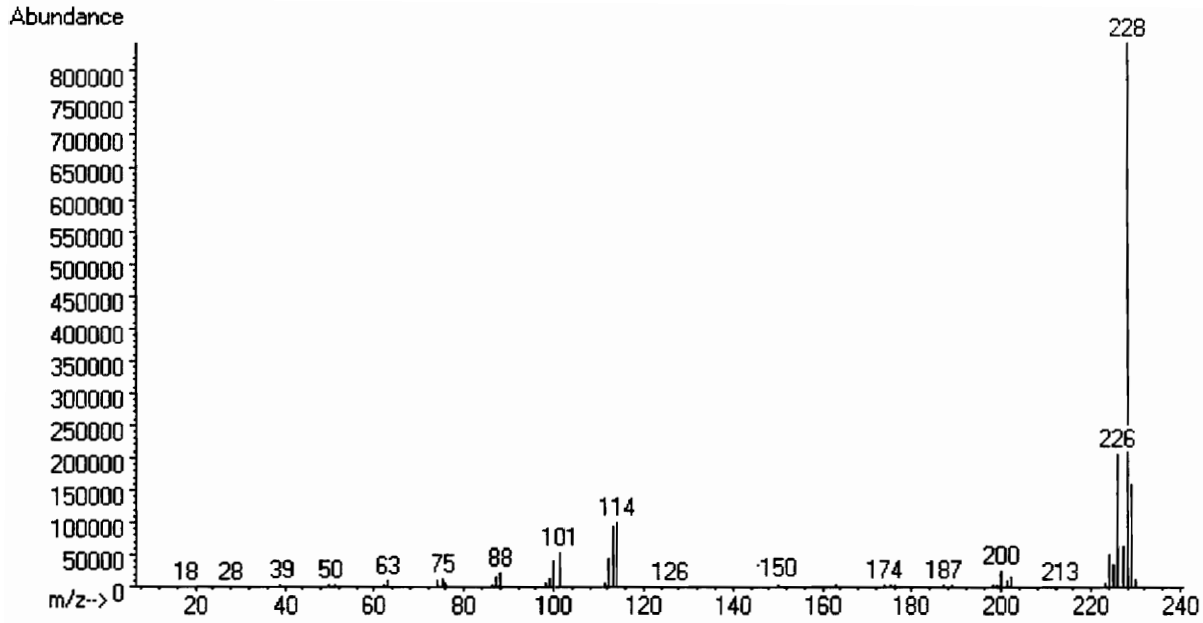
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



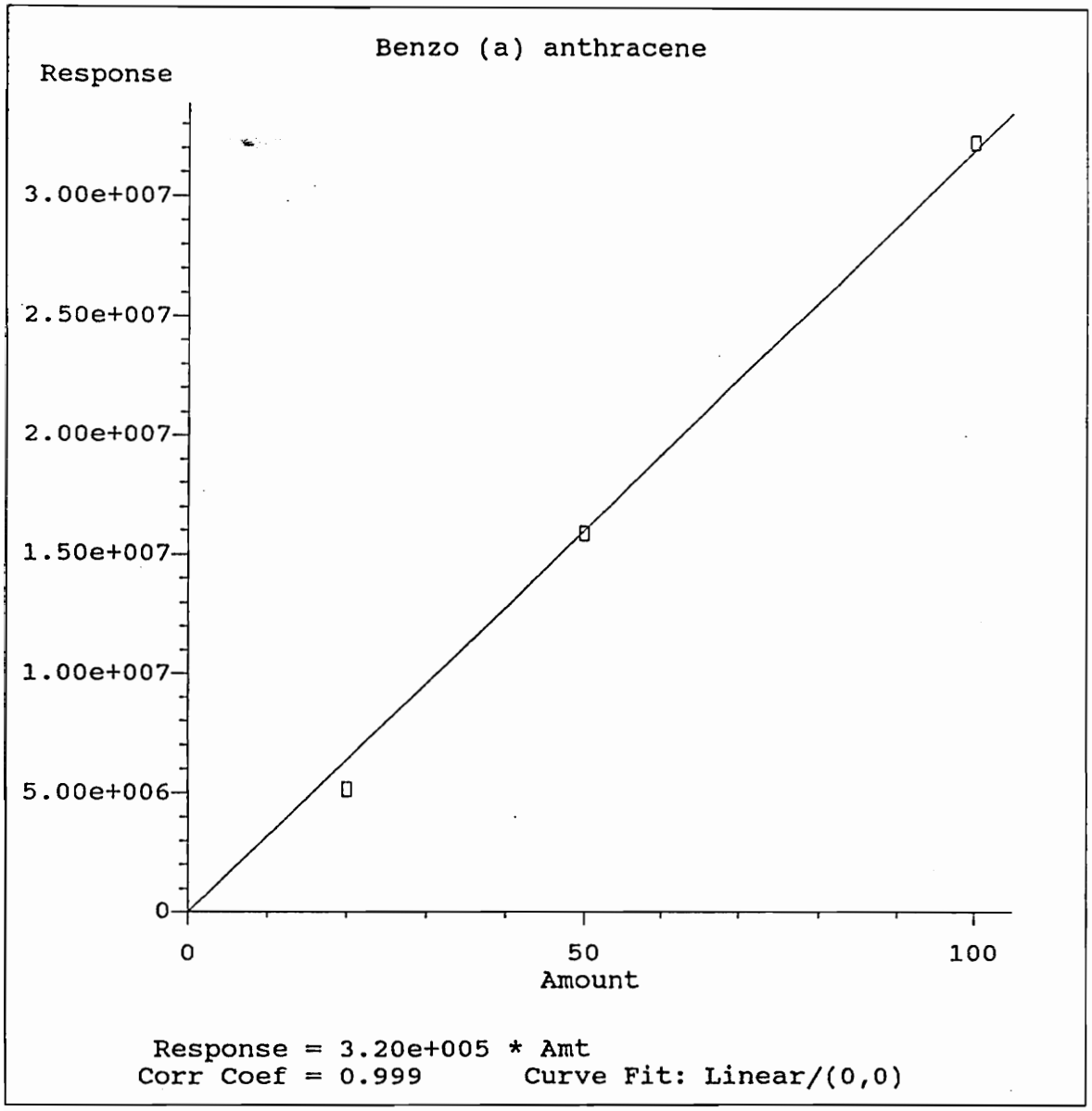
Mass Spectrum of Pyrene (Peak #19)



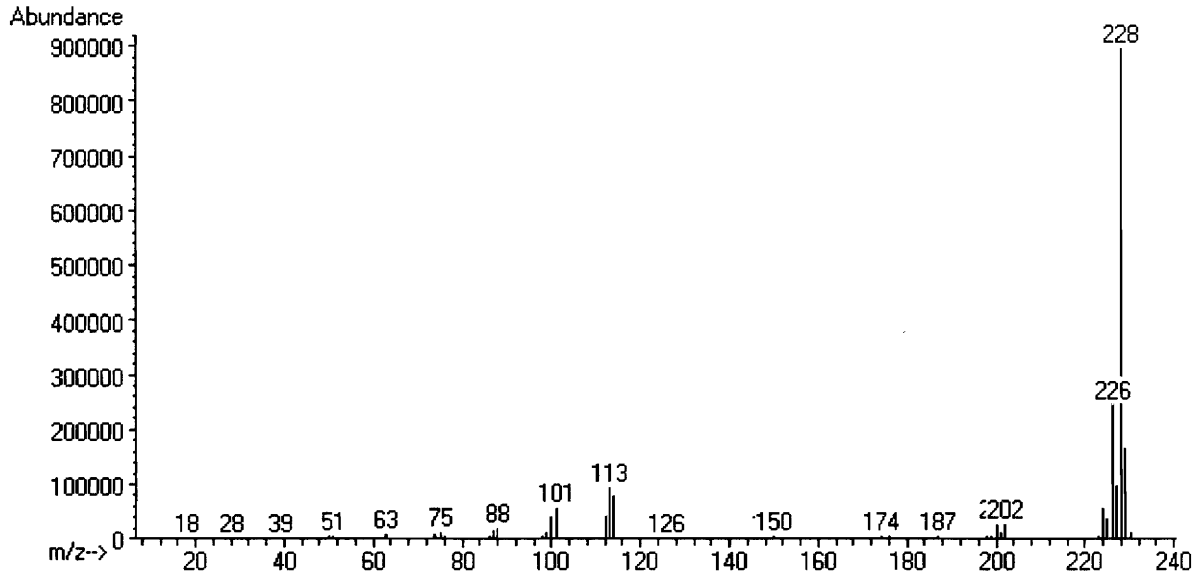
Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:30:35 1995



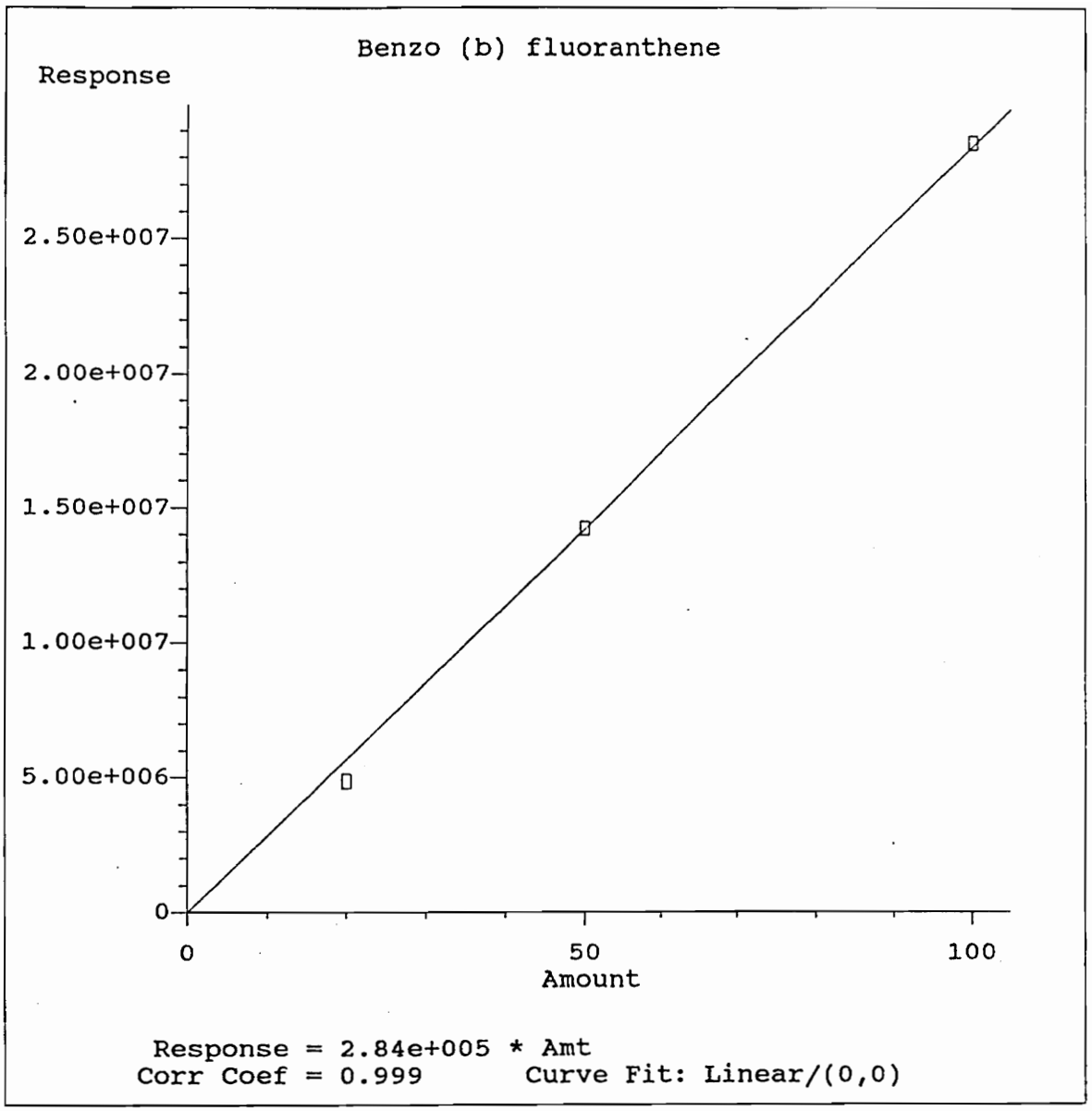
Mass Spectrum of Chrysene (Peak #20)



Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:37:07 1995



Mass Spectrum of Benzo (a) anthracene
(Peak #21)



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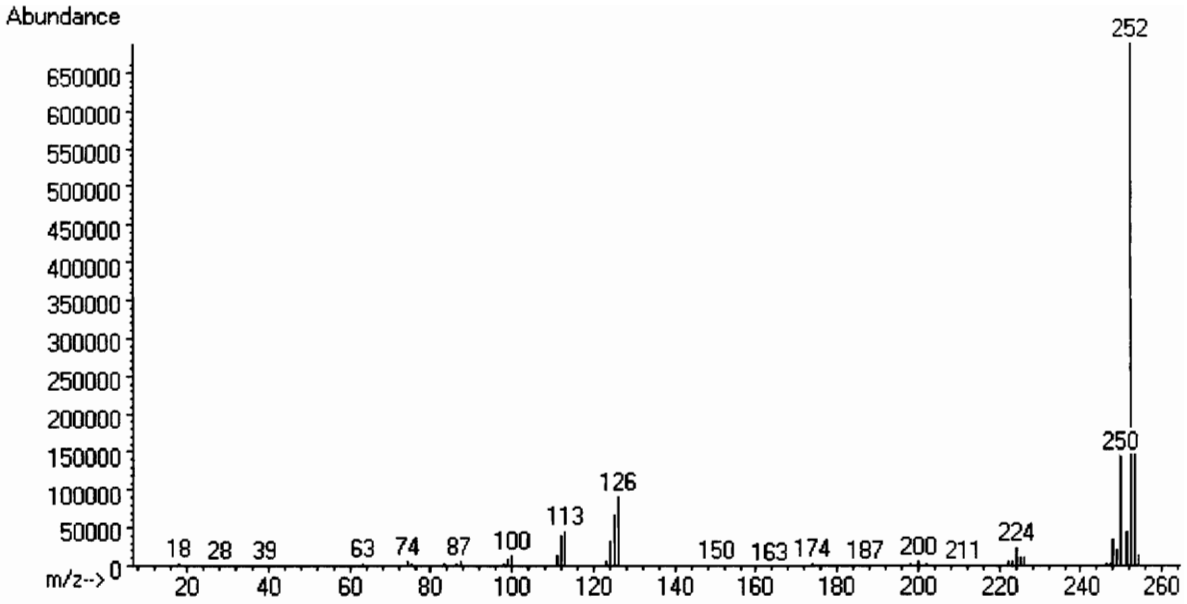
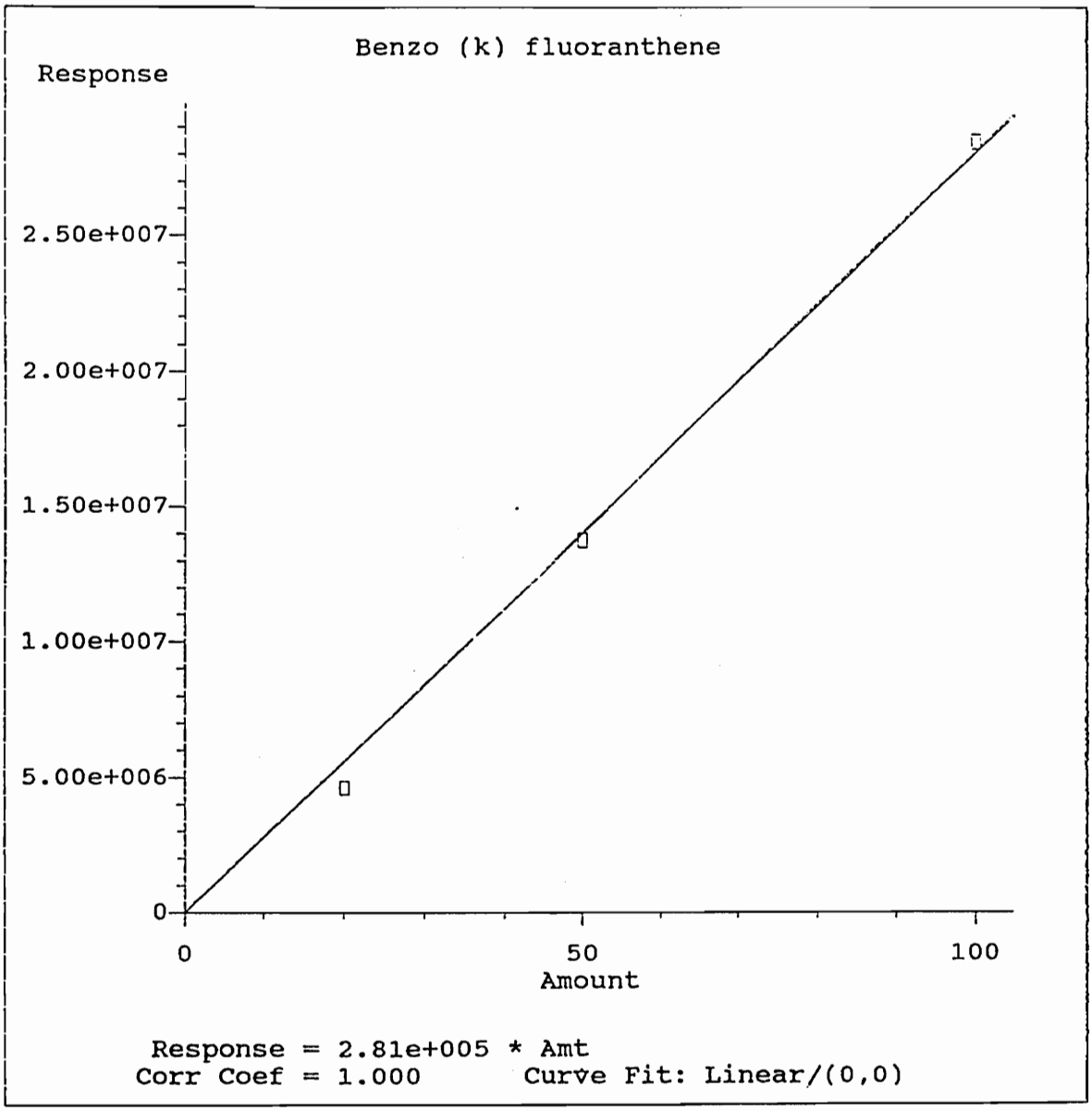
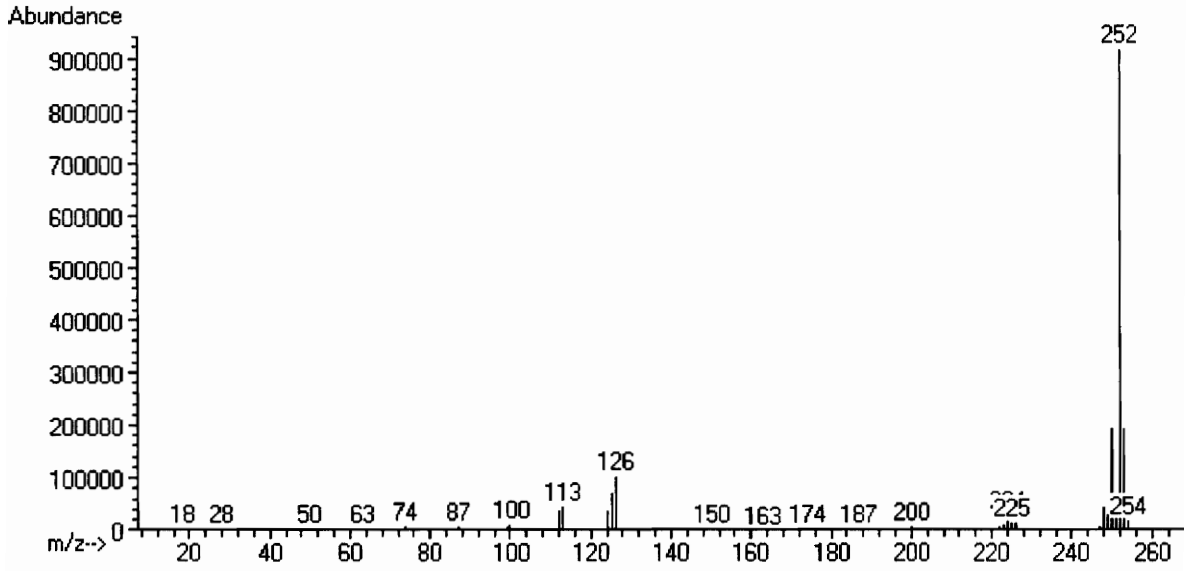


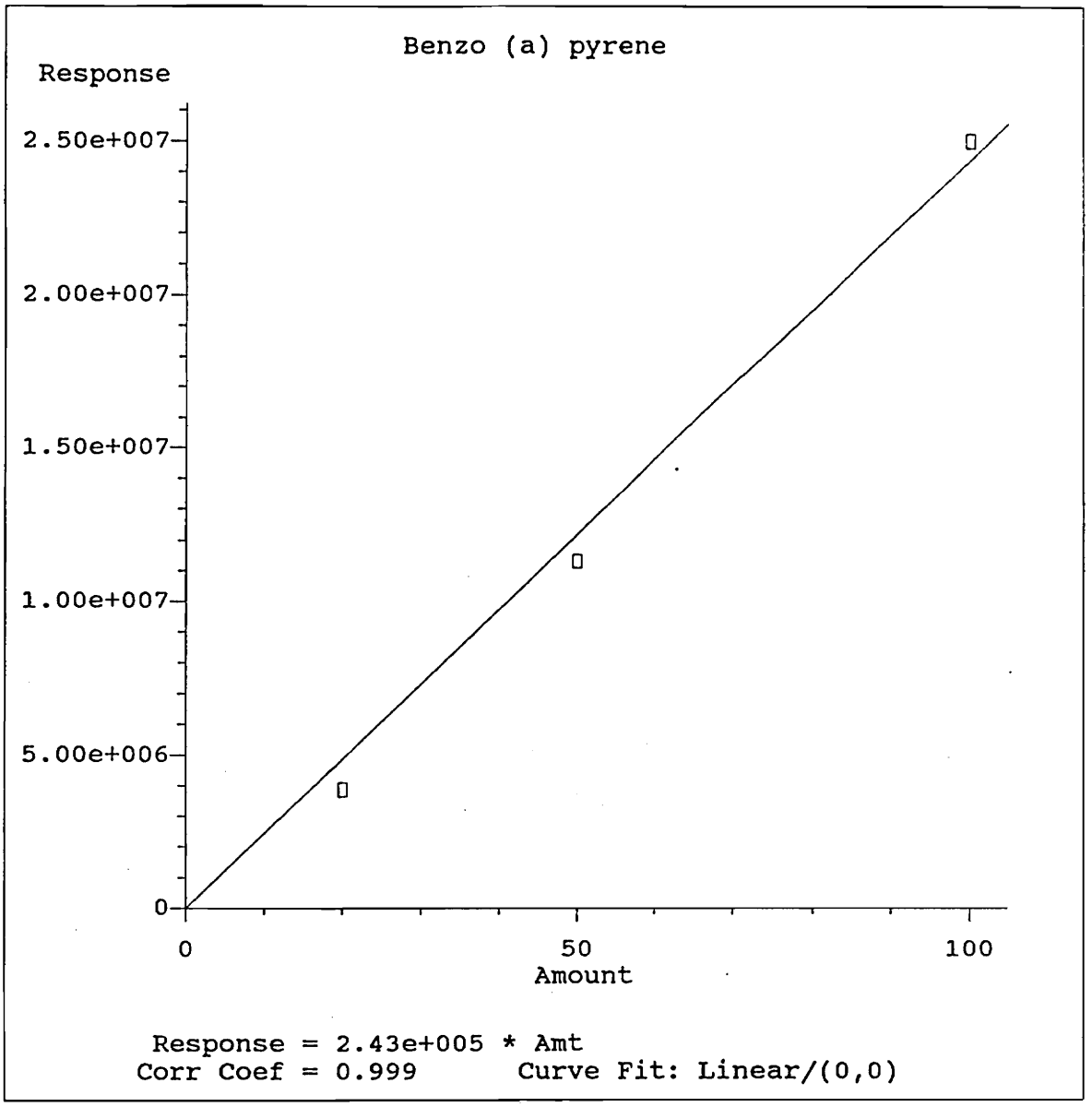
Mass Spectrum of Benzo (b) fluoranthene (Peak #22)



Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:37:07 1995



Mass Spectrum of Benzo (k) fluoranthene (Peak #23)



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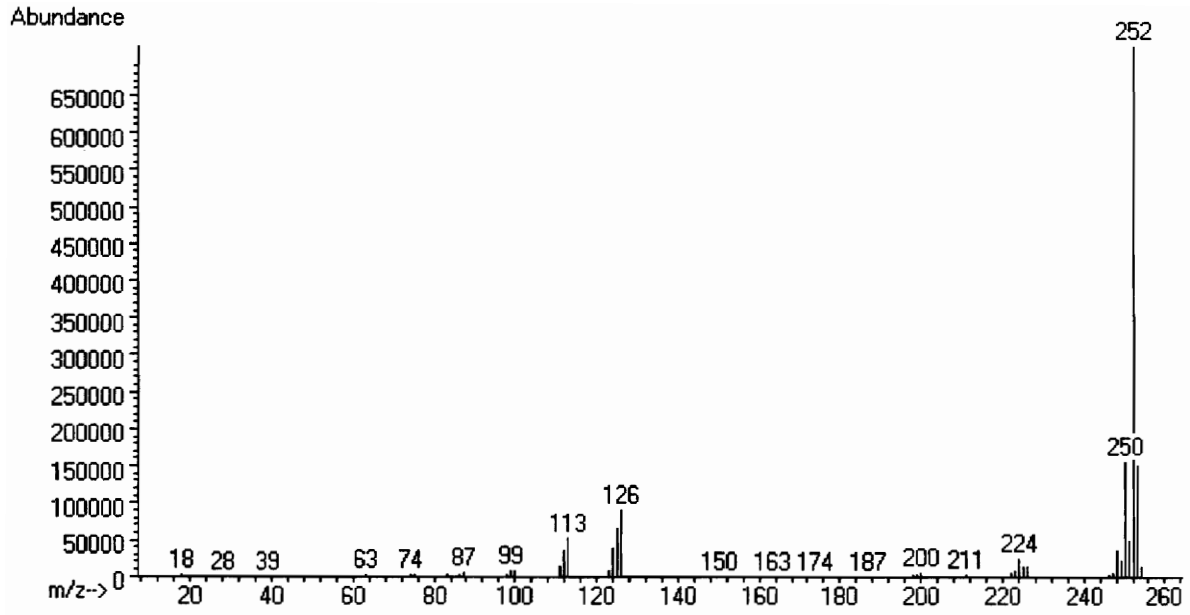
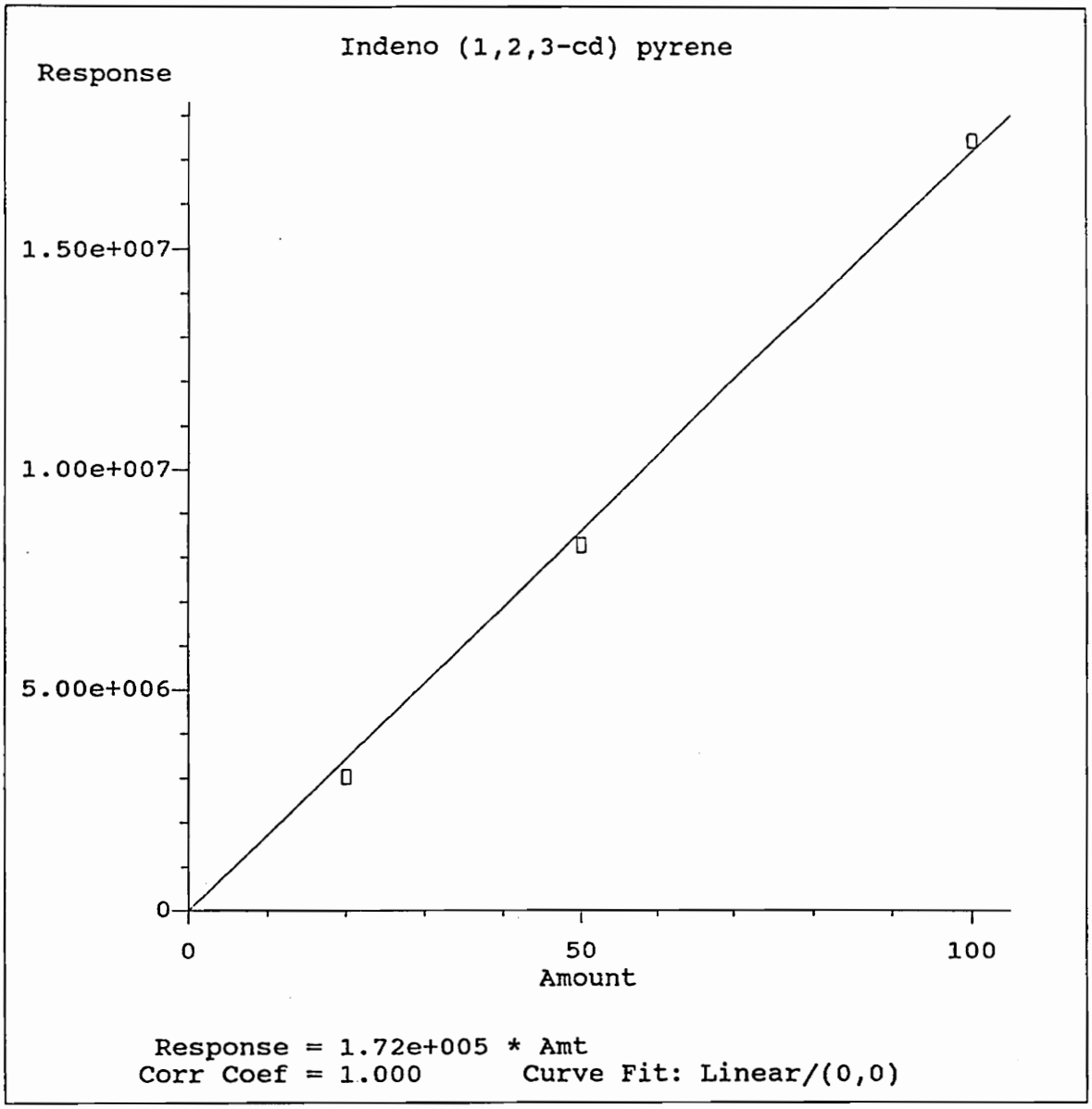
Calibration Table Last Updated: Tue Dec 19 16:37:07 1995



Mass Spectrum of Benzo (a) pyrene (Peak #24)



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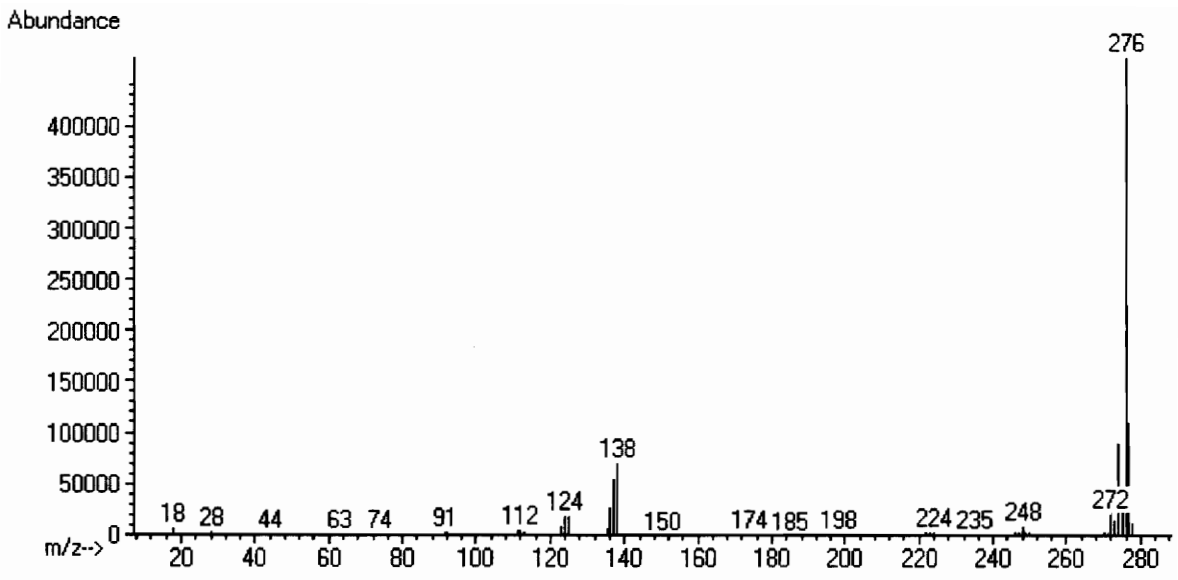
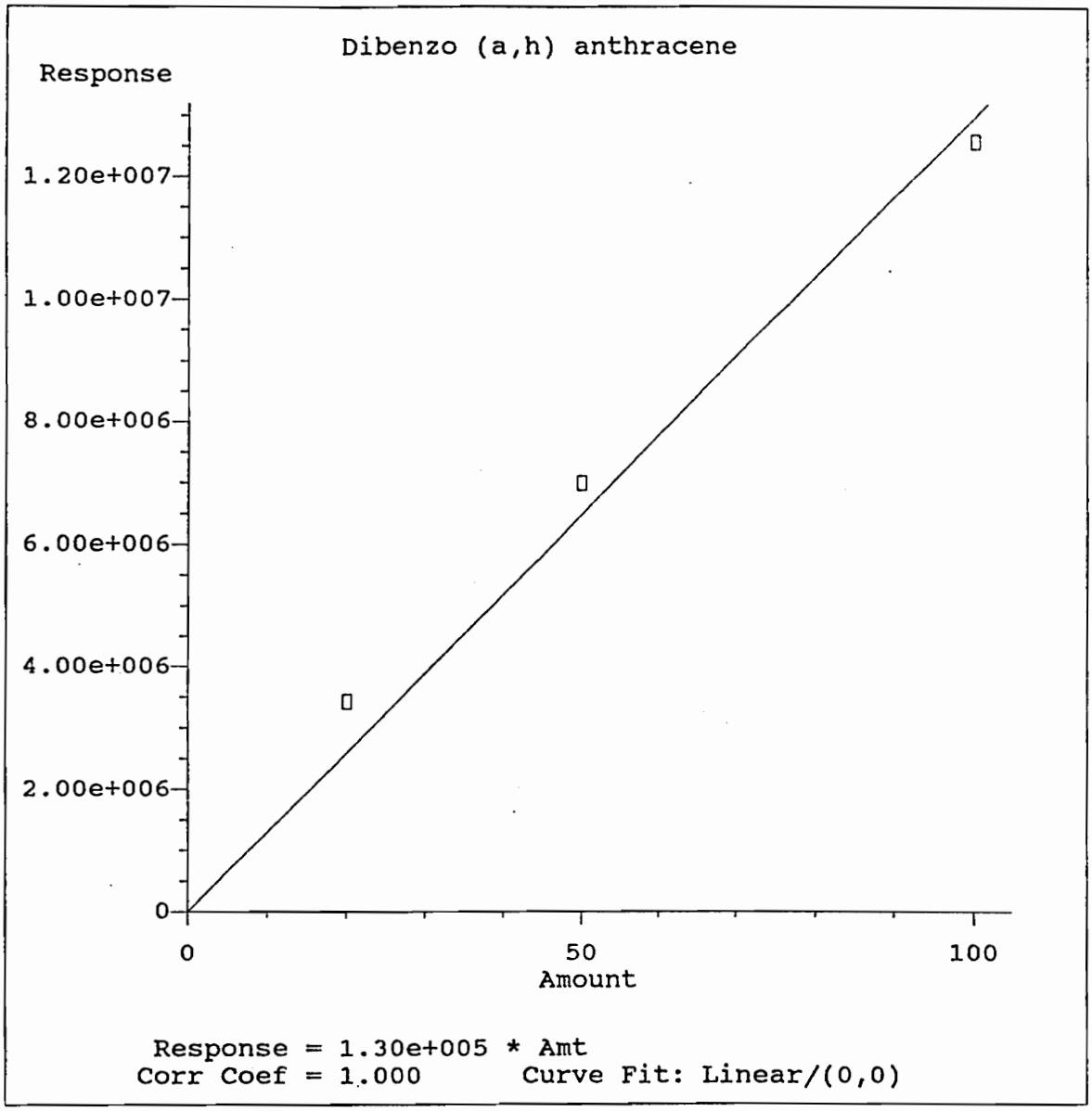
Calibration Table Last Updated: Tue Dec 19 16:37:07 1995



Mass Spectrum of Indeno (1,2,3-cd) pyrene (Peak #25)



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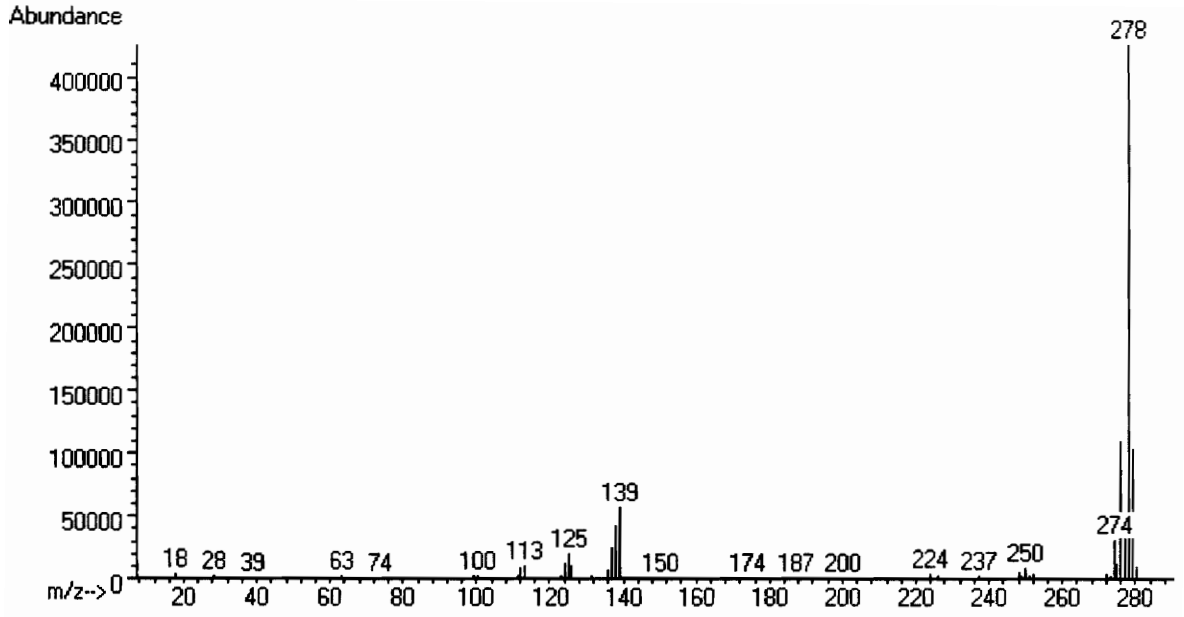
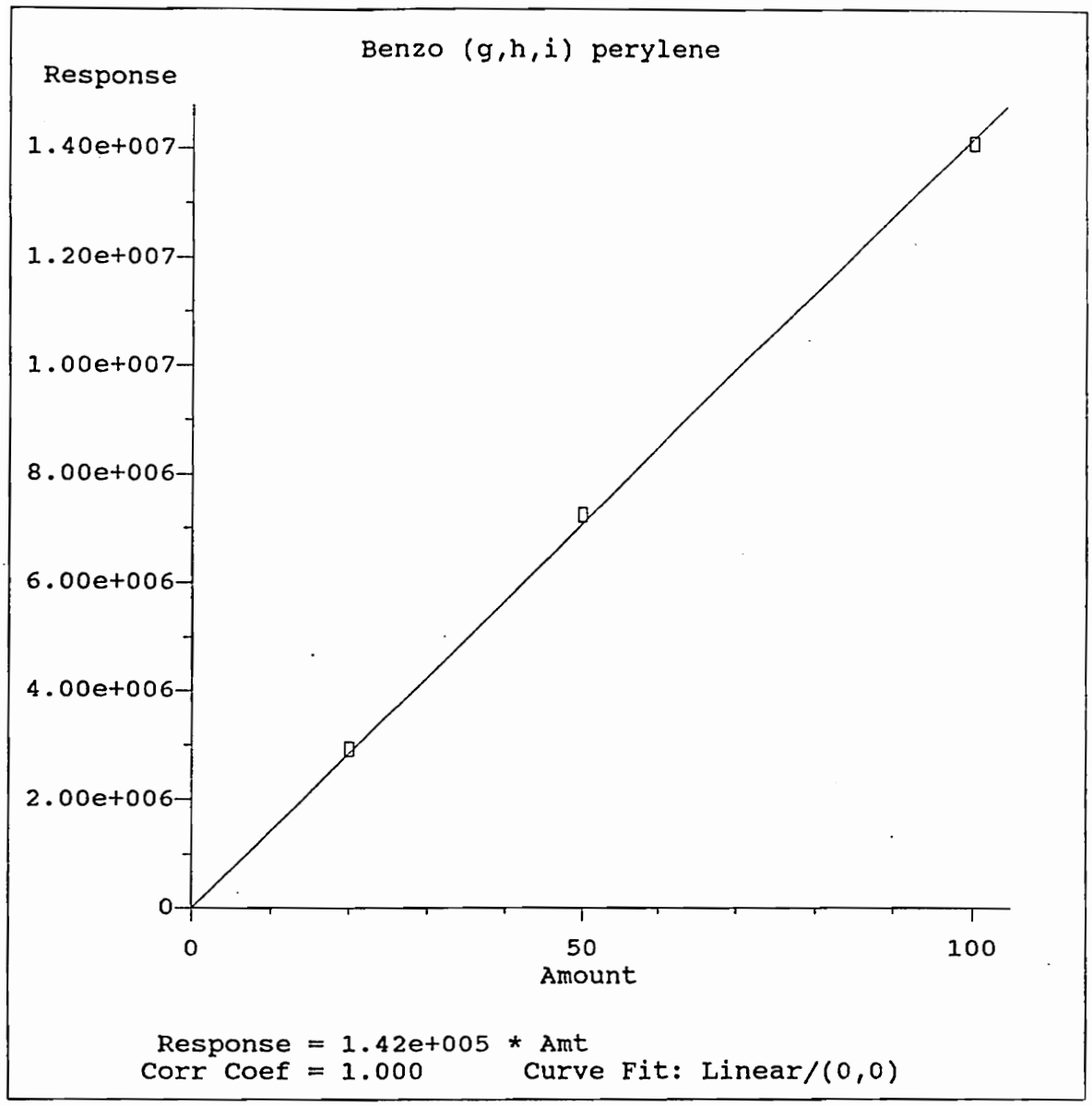
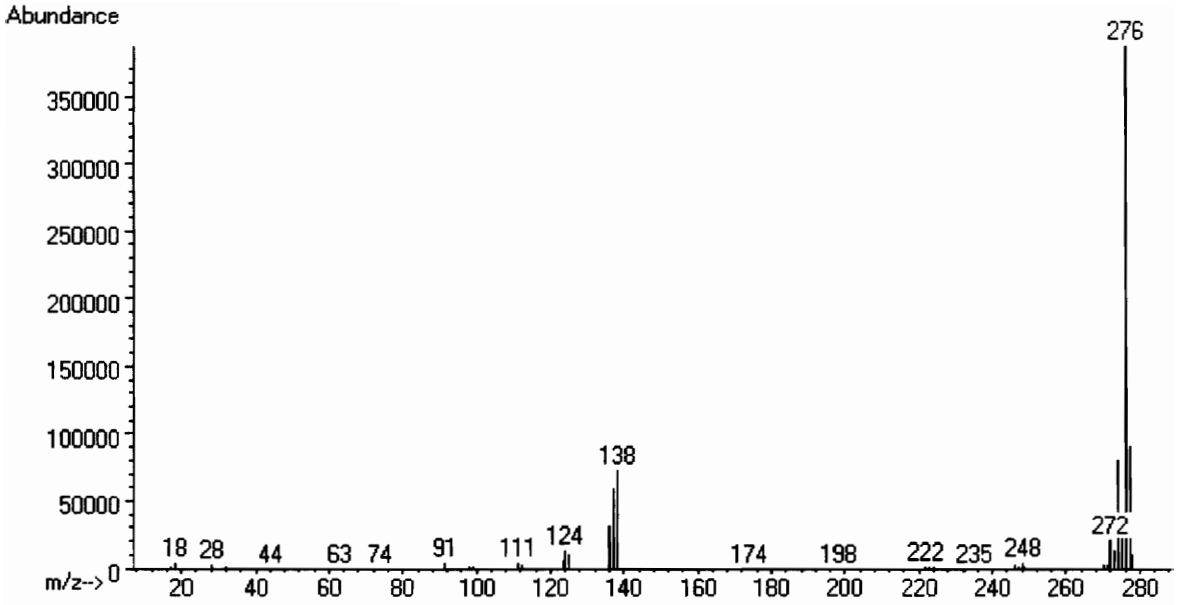
Calibration Table Last Updated: Tue Dec 19 16:37:07 1995



Mass Spectrum of Dibenzo (a,h) anthracene (Peak #26)



Method Name: C:\HPCHEM\1\METHODS\SCOTT.M
Calibration Table Last Updated: Tue Dec 19 16:37:07 1995



Mass Spectrum of Benzo (g,h,i) perylene (Peak #27)

VITA

Scott Smith, son of Frederick C. and Marianne L. Smith, was born on July 21, 1970 in Rockville Center, NY. He attended Berner High School in Massapequa, NY, from September 1984 to June 1987, then graduated from Massapequa High School in June 1988. His undergraduate work was performed at State University of New York at Oneonta from August 1988 to May 1992. While attending Oneonta, he taught General, Organic, Instrumental and Quantitative chemistry laboratories. He was exposed to industrial work as a summer intern with Anitec, Inc. in Binghamton, NY. He pledged a national fraternity and met his future wife in a local establishment. He received his BS in chemistry degree, with ACS certification, in May 1992.

Scott began graduate work in chemistry at Virginia Tech in August 1992. From August 1992 until August 1993 he was a chemistry teaching assistant under Dr. Glanville. During the summer of 1993, he got his first glimpse at a Research and Development Laboratory, while employed, on site, with the Naval Explosive Ordinance Detection Center in Indianhead, MD. He resigned from school, due to monetary hardships, and worked for two years in the

environmental industry. In October 1995, he returned to Virginia Tech to complete his masters program.

Scott completed the course requirements for the degree of Master of Science in Chemistry, in February, 1996. He has accepted employment with NYCOMED, Inc. in Albany, NY.