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ABSTRACT

Title of Thesis: Thermal Desorption of Hazardous and Toxic Organic Compounds (o-Xylene, p-Xylene, Ethyl Benzene, p-Chlorotoluene, and Anthracene) From Soil Matrices

Manuel Nolau, Master of Science

Thesis directed by: Dr. Joseph W. Bozzelli,
Principal Investigator

The purpose of this thesis is to study the thermal desorption behavior of toxic organic compounds, specifically o-xylene, p-xylene, ethyl benzene, p-chlorotoluene, and anthracene, from soil with respect to temperature and time, and to develop a mathematical model that will describe this desorption behavior. The model will allow prediction of the concentration of an organic contaminant in commercial soil desorption system over time given a known set of parameters of the specific chemical as well as flow rate and temperature. For this purpose, two sets of experiments were done: These are identified as 1. Thermal desorption and 2. plug flow experiments.

In the plug flow experiments, 1 microliter (ul) of the selected organic chemical was injected into a heated packed soil column with purge flow, residing in a constant temperature oven. The desorption curves resulting from the adsorption and desorption of the chemical to the soil-like packing materials were analysed for change in retention time versus the inverse temperature. Runs were made with four

packing materials: organic soil, sand, gaschrom-R and silica gel. An equation was developed for each material. These plug flow runs were made with ethyl benzene, p-chloro-toluene, p-xylene, and o-xylene. Results demonstrate that the retention time decreased with increases in temperature. Correlation factors of the fitted data for all runs were greater than .93, according to the following equation:

$$Rt = Aie^{Bi/T}$$

(See text for parameter definitions)

Thermal desorption runs consisted of heating a soil matrix, uniformly precontaminated with anthracene, in a quartz tube reactor and purging the contaminated soil with nitrogen. The nitrogen flow through the soil matrices was constant at 30 cm³/min, at a nearly constant temperature. The analysis of the desorbed organic was done with a gas chromatograph (GC) equipped with a flame ionization detector (FID). A relationship was developed based on time and temperature requirements for complete desorption of the selected pollutant species with inert gas purge. This relationship was used to develop an engineering equation where for any given hydrocarbon compound the time necessary for removal from soil, can be predicted for any given temperature close to the boiling point (± 40 °C). The results showed that the rate of desorption from soil of anthracene increased with increases in temperature as shown in the following mathematical relationship:

$$C(t)/C_0 = e^{-kt}$$

It was also observed that the natural logarithm of constant k was proportional to the inverse temperature as shown in the following equation:

$$k = 7.86 \times 10^3 \text{ (min}^{-1}\text{) exp}(-2.0 \text{ DHvap/RT}).$$

THERMAL DESORPTION OF
HAZARDOUS AND TOXIC ORGANIC COMPOUNDS
(o-XYLENE, p-XYLENE, ETHYL BENZENE, p-CHLOROTOLUENE)
(ANTHRACENE)
FROM SOIL MATRICES

by
Manuel Nolau

Thesis submitted to the Faculty of the Graduate School of
the New Jersey Institute of Technology in partial
fulfillment of
the requirements for the degree of
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APPROVAL SHEET

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FOREWORD

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I. INTRODUCTION

A. Objective

The objective of this experiment is to collect data on thermal desorption of hazardous organic chemicals from soil matrices so that some working thermal desorption and adsorption relationships or rules of operation can be developed. This will enable us to establish conditions of temperature, time and purge flow to achieve a final concentration of contaminant in the soil after thermal desorption allowing for better engineering size of the various process units. Thermal desorption has been shown to be a feasible, effective, efficient and economic method for purging hazardous chemicals from soil, as shown in the literature section of this paper. Previous selected organic chemicals used in similar types of studies are shown on Appendix 1(1)(2). Other objectives are:

1. To characterize specific types of soil for studies of desorption on known toxic and hazardous chemical substances.
2. To develop a database of time and temperature requirements for nearly complete thermal desorption with inert gas purge.
3. Develop relationships or engineering models that will take into account several basic parameters of the organic compounds such as dipole moment, heat of vaporization and boiling point, and allow prediction of the required flow period, velocity and temperature for removal of toxic

organic compounds from soil matrices to acceptable levels so that more expensive removal procedures such as incineration or granulated carbon adsorption are not required. To determine if the soil after treatment could be delisted as a hazardous waste or toxic waste the EP⁽³⁾(4) toxicity test would be used as a criterion.

I. PHYSICAL CONSTANTS OF SELECTED COMPOUNDS

COMPOUND	BOIL. POINT (°K)	FORMULA WEIGHT gr/mole	HEAT OF VAP. cal/mole	Dens. gr/cm ³	EMPIRICAL FORMULA
o-Xylene	417.	106.17	9998.5	.8802	1,2(CH ₃) ₂ C ₆ H ₄
p-Xylene	411.	106.17	9809.9	.8611	1,4(CH ₃) ₂ C ₆ H ₄
Ethyl Benzene	409.	106.17	9301.3	.8670	C ₈ H ₁₀
p-Chloro-toluene	435.	126.58	10151.7	1.0697	C ₇ H ₇ Cl
Anthracene	613.	178.23	16823.6	1.25	C ₁₄ H ₁₀
Biphenyl	529.	154.2	12910.0	.8660	C ₆ H ₅ -C ₆ H ₅

B. Environmental Overview

Soil contamination from volatile organic compounds (VOCs) or any other type of toxic organic chemical can occur from spills, from leaking vessels like underground storage tanks⁽⁵⁾, from improperly secured landfills, etc. The major concern when soil contamination occurs is that people will be exposed to these chemicals which may cause adverse health

effects. Another concern is that the environment will be adversely impacted.

When a case of soil contamination occurs the following questions should be asked:

- i. What type of contaminants are in the soil?
- ii. Are these chemicals dangerous to man or the environment?
- iii. Are these chemicals likely to reach the groundwater which is used for drinking?
- iv. If cleaning of the soil is required, which method is better in terms of cost, efficiency of removal, and in providing an optimum material for release to the reclamation site?
- v. Finally but not least, one should ask if federal, state, and local regulations relevant to the subject are being followed.

One should know the environmental and biological fate before action, such as clean-up, is taken so that one knows beforehand if a cleanup is required or not. Depending on the transport and transformation properties of contaminant organic compounds, they will tend to be more or less dangerous in certain types of media such as the atmosphere, the soil, the water and the biota. As a result, the degree of hazard a chemical represents to man and his environment from the contaminated soil can be estimated by answering the following questions: How much is taken away by solution in rainwater?; how much of it stays in the soil due to the thermodynamics of adsorption?; and how much of this contaminated soil is taken away by runoff or wind erosion⁽⁶⁾? Some mathematical models that account for contaminant transport or degradation through soils have been developed to describe the vaporization and diffusion through

the soil of low volatility organic such as tetrachloro-dibenzodioxin (2,3,7,8-TCDD) (7). These models predict the degree and extent of contamination within reasonable values. Volatilization, leaching to groundwater, and uptake of the organic contaminants by living matter are also important factors. Lastly one should consider if the contaminants are persistent in the environment. This can be accomplished by studying the rate of biodegradation and photodegradation of the organic compounds of concern in the soil.

Then there is the problem of finding out how clean is clean? As of now there are very few guidelines or regulations pertaining to cleanup of contaminated soils. Environmental risk assessments must be done in Superfund sites⁽³⁾ before safe levels of contaminants in the soil are established, but these studies are expensive and time consuming and they tend to be avoided by all parties in other types of contaminated sites. Maximum levels of some chemicals in the soil have been established and they range from 1 ppb for dioxins, 5 ppm for pcbs, to 100 ppm for hydrocarbons such as gasoline. However most chemicals are not covered by cleanup regulations but are covered by state and federal guidelines which state presently that the concentration of volatile organic compounds should not exceed 1 ppm and the total concentration of toxic organic compounds should not exceed 100 ppm. Unfortunately these guidelines usually are subject to change with the most

probable direction being towards increased safety and decreased levels.

If at the end of a feasibility study, a cleanup of contaminated soil is required there are different methods that can be used to accomplish this purpose. The soil can be incinerated; it can be removed as is and sent to a secured landfill; it can be treated biologically; or it can be air stripped at atmospheric or higher temperatures.

C. Thermal Alternative Processes

There are several types of thermal treatment technologies that have been tested and can be used for site remediation(8). These systems have been used to decontaminate soils containing 2,3,7,8-tetrachlorodibenzodioxin (TCDD), polychlorinated biphenyls (PCB's), creosotes, army explosives, and volatile solvents. All of these systems are either mobile and/or transportable.

They are:

- i. Rotary Kiln.
- ii. Infrared Conveyor Furnace.
- iii. Fluidized Bed.
- iv. Thermal Desorber.
- v. Hybrid Thermal Treatment System.
- vi. Other similar technologies.

Of particular importance is the rotary kiln system which will be studied in greater depth by future reseachers on this project. The effluent air stream containing the volatilized organic contaminant from the rotary kiln will pass through granulated activated carbon, or will be sent into a catalytic reactor or afterburner to oxidize the

organic contaminants.

Contaminants can also be sent to the the atmosphere if conditions permit. This last option, although much more desirable in a economic sense, is however less desirable in an environmental sense and one should be carefull to obey federal and state regulations regarding organic emissions into the atmosphere.

D. Industrial Relevance

The model describing needed conditions to achieve required removal, obtained from experimental results using a laboratory scale desorption system, will enable industry to determine if soil stripping is feasible. Thermal desorption systems may utilize steam or filtered hot exhaust vapors as purge gases injected directly into the soil at a site. Purge gases can also be injected into a rotary kiln where excavated soil is continuously introduced. Here the desorbed effluent contaminant can be sent into a catalytic reactor, a recuperative or regenerative thermal incinerator (afterburners), or a carbon adsorption system to control hydrocarbon emissions⁽⁹⁾. These different types of technologies have already been used by a wide number of industries. Parameters that must be considered which will enable one to choose one technology over the other are waste gas temperature requirements, susceptibility to contaminants, sensitivity to organic species, sensitivity to a variation hydrocarbon mixtures and concentrations, size

and space requirements, and emissions requirements. In a catalytic incinerator system, a temperature of 600 °F will generally insure that required degree of oxidation of hydrocarbons will occur. Testing done in several industries demonstrated the feasibility of catalytic incineration. The organics destroyed to at least an eighty percent (80 %) efficiency were: toluene, xylenes, ethyl benzenes, MIBK, methyl ethyl benzenes, cellosolve, C12 to C18 hydrocarbons, phenol, cresols, and methyl ethyl ketone⁽⁹⁾.

E. Previous Research

There have been relative few studies on desorption of organic compounds from soil. One such study was done by Vossoughi, Willhite, Shoubary and Bartlett⁽¹⁰⁾. In this paper the researchers studied the effect of sand, silica and kaolinite on crude oil combustion. Three different regions were observed during the course of each experimental run. They were distillation, combustion and cracking with the distillation region being more important in the thermal desorption of organic compounds from soil matrices. A curious phenomenon was observed when the purge gas was nitrogen. Here oil was harder to desorb from clay than with air. With air, the fraction of oil remaining in the sand at the same time and temperature was less than with nitrogen. This could be explained by clay acting as a catalyst helping the combustion to occur. When only nitrogen gas was present the large area present in clay acted as an increased

adsorber. This is important since it is anticipated that desorption in the field will be done with hot air. As such performance by air desorbers is expected to be better than reactors that use inert nitrogen, whenever the temperature is high enough for the breakdown of organics.

In another study Bennedsen from Woodward-Clyde Consultants, Walnut Creek, California, reported that several soil gas Vapor Extraction Systems (VES) were installed and proved to be cost effective, with the major system operating costs being accounted for by sampling and analysis of the extracted soil and gas, i.e. to monitor the system performance⁽¹¹⁾. Desorption of contaminants occurred in this system when vacuum was applied producing a flow of air across the contaminated soil. The desorbed contaminants were then discharged to the atmosphere, or sent to a combustion chamber depending on the concentration of pollutant species being discharged. This system has the disadvantage in that it only works with loosely bound soil, and/or highly volatile organic compounds at ambient temperatures.

Additional studies done by the U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) have also proved the feasibility of thermal desorption⁽¹²⁾. They have developed two separate thermal technologies for the treatment of soils in-situ or for the thermal desorption of excavated soil. The in-situ process is very similar to the VES system, and

consists of injecting clean air into the soil carrying the VOC's to either a vented emission control system or the atmosphere, as required by existing concentrations of contaminants. Thermal stripping consisted of excavating the contaminated soil and introducing it into a thermal processor such as a rotary kiln, where the VOC's were volatilized (desorbed). These volatile compounds are then incinerated in an afterburner, and the clean desorbed soil is disposed in a municipal landfill.

Thermal desorption technology was also tested at the Times Beach site which had been contaminated with dioxins. With this process, the soil was excavated and introduced by a steel belt conveyor into a thermal desorption chamber provided with several infrared lamps. After the dioxins were driven off the soil, the off gases were burned in a secondary chamber, fired with propane at 2400 °F. This system was developed and tested by Shirco Infrared Systems of Dallas⁽¹³⁾. In another site this system treated around 2,000 pounds of soil containing 50 to 5,000 ppm polychlorinated biphenyls⁽¹⁴⁾. Preliminary results showed that final concentrations were below detection limits of 1 ppm.

Several studies done by Hornsby⁽¹⁾, and Chemburkar⁽²⁾ respectively, demonstrated the feasibility of removing toxic organic compounds from soil with a hot purge gas. Each reseacher did two types of experiments described

later in this report (see Experimental Section), using different organic compounds. In the first plug deposition experiment, toxic organic compounds were injected into a slightly modified GC, where the regular column had been replaced by a one fourth inch O.D. steel column filled with one of four selected packings. From this apparatus a relationship was developed that correlated retention times with temperature of the GC oven. In the second experiment, the initial concentration of the contaminant was maintained constant throughout the soil. This soil was introduced into a quartz column and placed inside an heated tube oven at a known temperature. Desorption was carried out using a nitrogen purge flow. A global mathematical model was then developed from the available data that could predict the desorption rate of toxic organic compound using only known chemical properties. From these studies it was concluded that thermal desorption with a purge flow gas can reduce concentrations of contaminants to acceptable levels, and that within the chosen parameters the rate of desorption was first order.

Other experiments were done by researchers from the University of Utah on thermal desorption of organic contaminants from soil(15)(16)(17). First a Particle-Characterization Reactor (PCR) was developed to study intraparticle effects with small thermal and concentration gradients external to the particle. In this apparatus a

desorbent flow of hot gas was passed through a soil bed inserted in a high-temperature furnace. The exiting gaseous mixture was then pumped to a gas-chromatographer (GC) system with a flame ionization system. From the PCR experiments the following was concluded: Desorption rates are a strong function of soil types; heavier hydrocarbons are more difficult to remove than lighter compounds; in complex hydrocarbon mixtures, the lighter compounds are desorbed first; desorption rates increase with increases in the local temperature; and the adsorption data can be represented by the Freundlich isotherm. This research group also developed a Bed-Characterization Reactor (BCR) to study organic transport within a bed of particles, and a rotary kiln fired with natural gas which was used to study the transient evolution of contaminants from soil considering realistic temperature and mixing effects. The BCR experiment consisted of passing a purge flow of gas over the top of two trays containing contaminated soil. The resulting contaminated exiting flow of gas was analyzed using GC/FID and GC/MS (mass spectrometry) methods. The temperature gradient of the soil was determined by placing twelve type-K thermocouples in one of the trays, and the evolution rate was determined by placing the second tray on a electronic balance. In the BCR experiment it was observed that higher temperatures increase the evolution rate of the contaminant species, and the evolution rate of the contaminant species

decreased with increasing depth of soil. The some conclusions were reached with the rotary kiln apparatus as applicable.

F. Brief Description of Experiments

Two types of experiments were done in this research to study the desorption rate of selected organic compounds from different soil and soil like matrices. The first experiment or simulation of desorption is termed Plug Flow Deposition. The second experiment or model is named Thermal Desorption and utilizes uniformly contaminated soil.

EXPERIMENTAL

A. Plug Flow Deposition Experiments

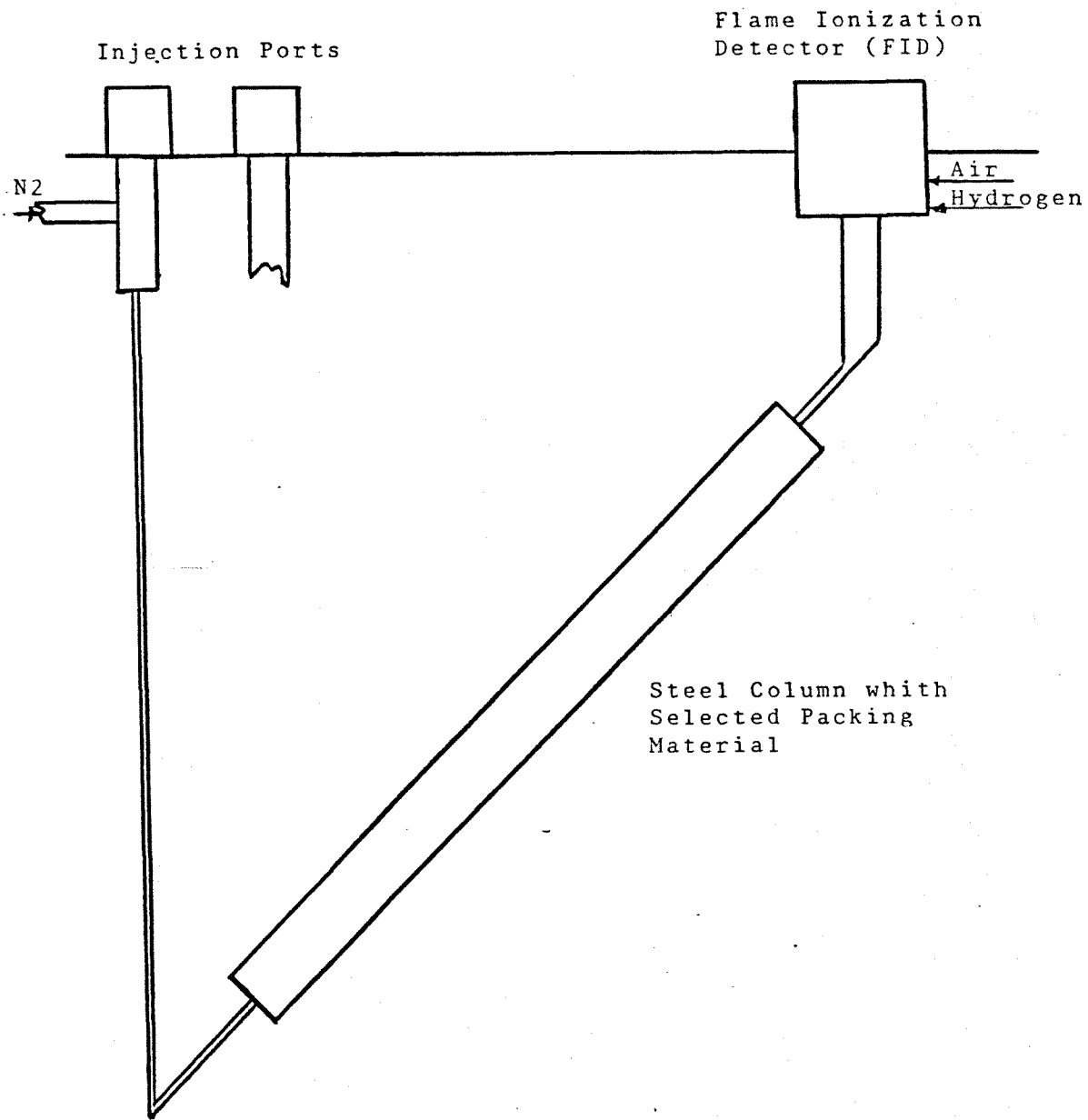
1. Apparatus

The plug flow experiments were performed in a Shimadzu Gas Chromatograph (GC) equipped with a Flame Ionization Detector (FID). This GC was equipped with two columns and connected to a Varian Model 4290 Reporting Integrator⁽¹⁸⁾⁽¹⁹⁾ (Spectra Physics). One of the standard columns was removed and was replaced with a stainless steel tube of 12.3 cm length by .4 cm ID, packed with one of the four selected packings listed below. The volume flow rate through this packed column was 30 ml/min.

TABLE II. COLUMN PACKINGS FOR PLUG DEPOSITION EXPERIMENTS

Packings	Mass (grams)	Mesh Size	Material
Soil	2.90	35-45	dried top soil
Sand	3.00	45-80	silicone oxide
Silica gel	2.90	10-200	silica/silica hydroxide
Gaschrom-R	2.14	60-70	alumina and silicone oxides

These four packings were chosen to represent natural soils with a range of retentive tendencies. Sand was used to mimic a highly porous material in the environment and silica gel was used to represent a worst case scenario where the adsorption of the hydrocarbon compound to soil would be



Plug Deposition Apparatus Schematic

FIGURE 1

very strong.

Each packing was poured into a graduated cylinder until a predetermined volume was reached. This amount was weighed in a tared container. Then the weight of the packings were divided by the respective volumes in order to determine the bulk densities. To determine the actual densities another 50 ml graduated cylinder was filled with 10 to 20 ml of water. The previously weighed packings were then poured into the water. The final volume of water was read and the weight of the packings were divided by the amount of displaced water to determine the actual densities. The linear velocities of the purge flow gas for each packing were calculated as shown in the Sample Calculations. (See Table III and Section V).

TABLE III. EXPERIMENTAL PARAMETERS FOR COLUMN PACKINGS

Packings	Actual Density (gr/ml)	Bulk Density (gr/ml)	Volume Flow Rate (ml/min)	Linear Velocity (cm/sec)
Soil	2.36	0.946	30.	2.6
Sand	2.49	1.49	30.	3.9
Silica gel	2.20	0.725	30.	2.3
Gaschrom-R	2.72	0.381	30.	1.8

The retention times were studied isothermally in 20 °C increments. The lowest temperatures used were, in general, 40 °C below the boiling point of a specific

compound. If the desorption times were too large, then higher temperatures were chosen. The highest temperatures chosen were those that caused the organic compound to desorb in a few seconds. The highest temperatures ranged from 200 to 260 °C.

Compounds studied in the plug flow deposition experiments were:

Ethyl Benzene
p-Chlorotoluene
o-Xylene
p-Xylene

2. Procedure

One microliter samples of the liquid organic compound was injected into the Shimadzu Gas Chromatograph (GC). The settings on the Shimadzu GC were 1 for attenuation, 10^2 for the range, 250 °C for the injection and detection ports. The temperature of the oven was set at various temperatures in order to optimize retention times. The temperature range of the oven was set at approximately ± 40 °C of the boiling point of the compound being studied. The desorption rates were studied from 80 °C to 260 °C at an increment of 20 °C for each run.

3. Mathematical Modeling

From previous experiments it was observed that the natural logarithm of the retention time of the selected organic compounds was directly proportional to the inverse temperature(1)(2). From this observation, the following

empirical equation was developed to describe the relationship between retention time and temperature. The equation is:

$$R_t = A_i e^{B_i * 1/T}$$

where:

- i. R_t is the retention time in minutes.
- ii. A_i is a fitted constant dependent on the mass.
- iii. B_i is the slope of the linearized equation and it is dependent on the Temperature, Dipole Moment, Heat of Vaporization, Purge Gas Flow Rate and Soil Properties.
- v. T is temperature in degrees Kelvin

Graphing was done with a computer software package, Plotrax. Graphs are shown and illustrated in Appendix 7: Figures Concerning Plug Flow Results.

4. Results

The retention times in this experiment were observed to increase exponentially with increases in the inverse temperature.

The following results were obtained:

TABLE IV. RESULTS FOR THE SOIL COLUMN

Compound	A_i (10^3)	B_i (Slope)	(Correlation Factor) ² r^2
o-Xylene	3.125	2474.0	0.999
p-Xylene	2.629	2510.7	0.999
p-Cl Toluene	2.904	2544.6	0.985
Ethyl Benzene	5.184	2194.6	0.992

TABLE V. RESULTS FOR THE SAND COLUMN

Compound	Ai (10 ³)	Bi(Slope)	(Correlation Factor) ² r ²
o-Xylene	3.907	2267.8	0.991
p-Xylene	1.386	2798.9	0.931
p-Cl Toluene	2.092	2545.9	0.980
Ethyl Benzene	5.856	2048.1	0.995

TABLE VI. RESULTS FOR THE GASCHROM COLUMN

Compound	Ai (10 ³)	Bi(Slope)	(Correlation Factor) ² r ²
o-Xylene	7.980	1738.8	0.986
p-Xylene	8.964	1611.4	0.981
p-Cl Toluene	8.581	1750.9	0.980
Ethyl Benzene	16.69	1425.8	0.977

TABLE VII. RESULTS FOR THE SILICA-GEL COLUMN

Compound	Ai (10 ⁶)	Bi(Slope)	(Correlation Factor) ² r ²
o-Xylene	2.019	7486.1	0.997
p-Xylene	3.470	7200.9	0.996
p-Cl Toluene	-.---	-----	.---
Ethyl Benzene	4.846	6997.0	1.000

As shown in the previous four tables, the empirical equation fit all the data with the square of the correlation factor greater than 0.931.

5. Discussion of Results

Results show that the mathematical model will fit the data for all compounds with the correlation factor being greater than 0.93. The retention time decreased with increasing temperature according to the equation shown in the mathematical modeling section. The slope could be dependent on the heat of vaporization or on the boiling point of the selected chemicals. However, results were inconclusive due to the closeness of heat of vaporization values of the selected compounds. The linear velocity of purge gas through the packings, and the weight of the packings were different from column to column. As a result, the relative retentiveness of each packing could not be determined. Data is shown in Appendix 2: Plug Flow Data.

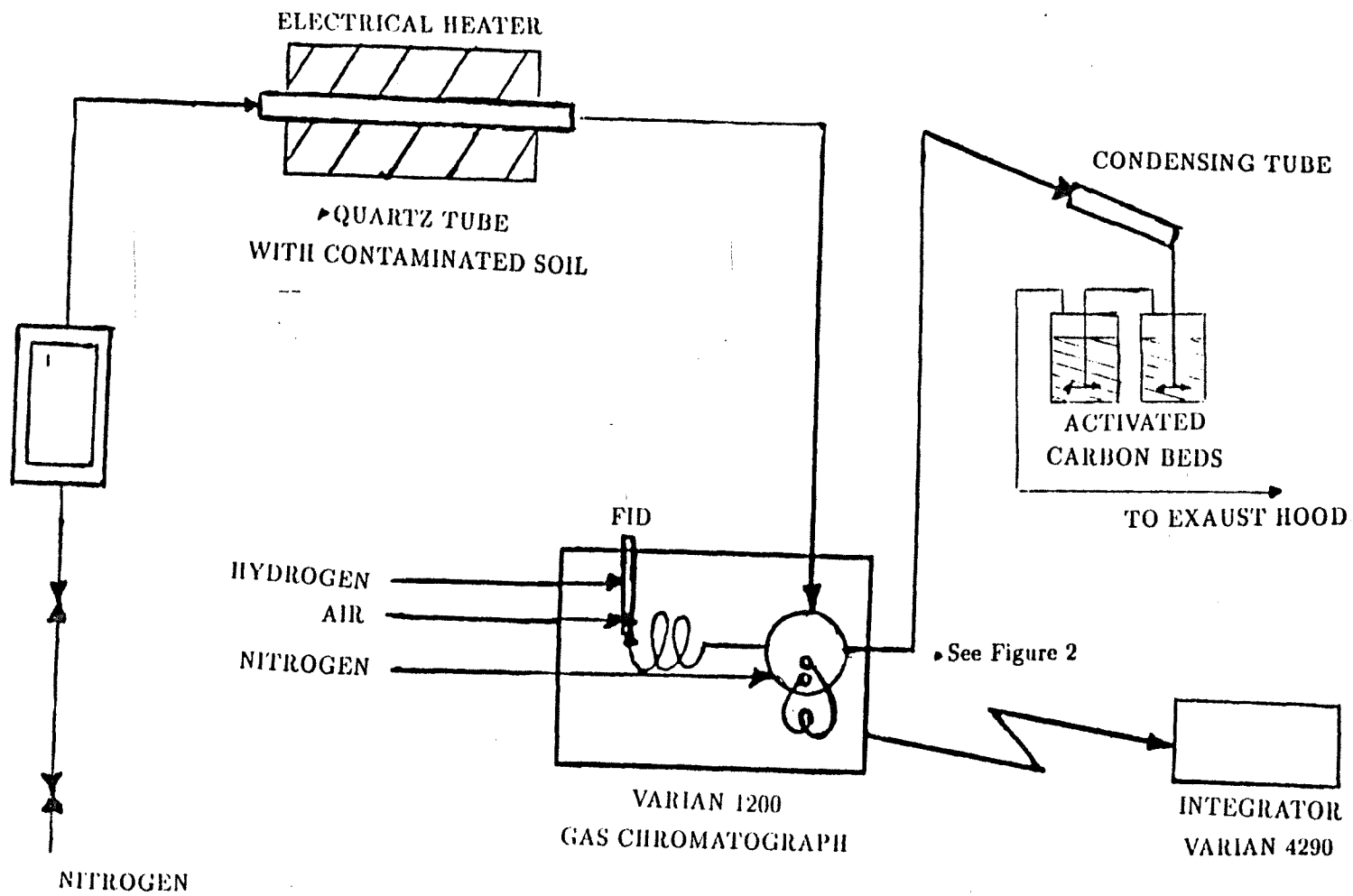
B. Desorption Experiments with Uniformly Contaminated Soil

1. System Description and Operation

The reactor consisted of a quartz tube of approximately 30 cm in length and 10 mm inside diameter. This tube contained the soil sample to be desorbed and which was secured to stainless steel tubing at both ends by 1/2 inch * 1/4 inch stainless reducing union connectors, equipped with 1/2 inch graphite ferrules, stainless steel back ferrules and two polytetrafluoroethylene gaskets (Teflon). The quartz tubing was used because it provides an inert surface and does not act as a catalyst for the decomposition of the target organics.

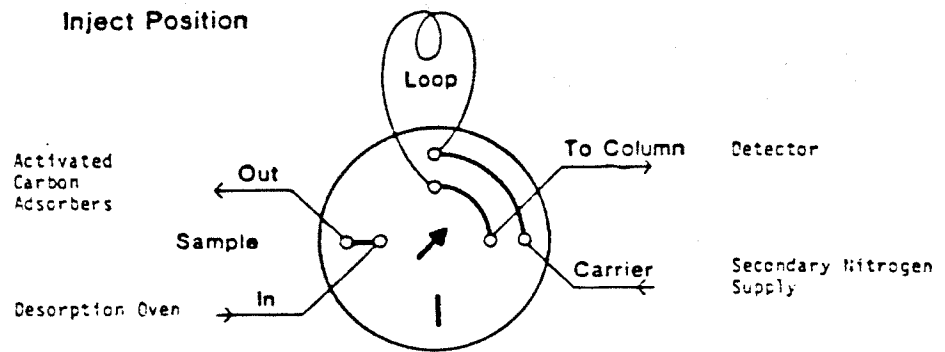
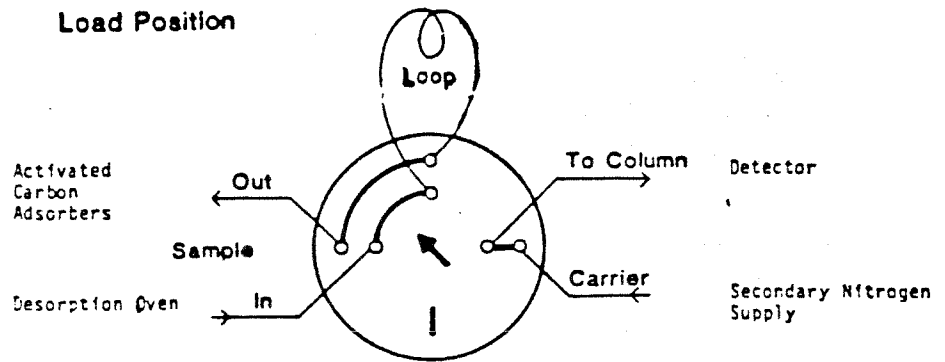
The quartz column was filled with loosely packed soil held inside by quartz wool plugs. The length of the tube occupied by the soil was 15 cm. The quartz tube was placed in the reactor in such a manner that the portion of the tube containing the soil was in the center of the reactor. The temperature of the reactor of the quartz tube was monitored by two chromel/alumel thermocouples at the center of the quartz tube and at the end of the soil matrix. The temperature between the center and the side of the soil matrix varied by as much as 20 °C and as such a corrected temperature was used to determine the constant of the equation.

Desorption was achieved by heating the reactor at



DESORPTION SYSTEM

FIGURE 2



SIX PORT VALVE CONFIGURATION

FIGURE 3

high temperatures (200-380 °C) and purging the reactor with a nitrogen flow of 30 ml/min. The tubing leading into and out of the reactor as well as the tubing leading into the granulated activated carbon collectors was heated with resistance heating tape. This was done in order to prevent the accumulation of anthracene inside the tube.

2. Characterization of Soils

The soil necessary for this experiment was obtained and treated according to the method of Chemburkar⁽¹⁾ and Hornsby⁽²⁾. A sample of thirty kilograms of soil was taken from the grounds of the New Jersey Institute of Technology at Newark. This soil was placed into large aluminum containers and washed with tap water to remove the clay and silt like particles, salts and other polar compounds. After washing, the mixture was decanted, and the soil slurry was spread over plastic bags and allowed to dry at room temperature. The soil was then sieved and separated into six different mesh sizes as shown in Table XVI. Sieving was done by placing two kilograms at a time in a set of standard sieves and then placed in a mechanical shaker for five minutes. Three size ranges were retained for further study as shown in Table XVI.

The soil from those three mesh sizes was re-homogenized and heated overnight at 200 °C to drive off moisture and most organic compounds. A sample of this soil was sent out for analysis by X-ray diffraction and X-ray

scattering. This analysis, as shown in Appendix 3 gave a detailed chemical make-up of the soil, listing all inorganic components and their fractions present in the soil matrix.

To characterize or delist a contaminated soil as an hazardous or toxic waste, the Extraction Procedure (EP) Toxicity Test⁽³⁾⁽⁴⁾ as defined by state and federal regulations is used. This test requires agitation of a 20:1 mixture of waste in aqueous media. The pH is adjusted to 5.0, if possible, with .05 N acetic acid with a maximum addition of 400 ml. The EP extract is analyzed for eight metals (As, Be, Cd, Cr vi, Pb, Hg, Se, and Ag, plus two herbicides [2,3-D and 2,4,5-TP(Silvex)] for which National Interim Primary Drinking Water Standards (NIPDWS) have been established.

3. Preparation of Standards to Create Plots of Area Ratios Versus Concentration (ppm)

A preweighed amount of anthracene was dissolved in toluene to a given concentration of 5000 ppm(w/w). The first solution was made up of 0.8669 grams of anthracene diluted in 200 ml of toluene. Toluene was chosen because it dissolved anthracene easily and it has a relatively high boiling point. The density of toluene is 0.8669 grams per milliliter⁽²⁰⁾.

The 5000 ppm anthracene solution prepared on the first step was further diluted with toluene to the desired

concentrations as shown on Appendix 4. To minimize the wastes produced, care was taken that quantities of chemicals used in the solutions were kept as small as possible, without affecting the accuracy of the standard curves. To each of these solutions, enough 1-chloronaphthalene was added as an internal standard to obtain a solution with 1000 ppm concentration of 1-chloronaphthalene.

The Shimadzu GC was used to analyze these standards, using flow rates of nitrogen, hydrogen, and air set each at 30 ml/min, respectively⁽²¹⁾.

A series of preliminary runs were done to determine the optimum GC column temperature at which the components (toluene and anthracene) of the standard solutions would separate best. These runs were operated at 150 C to 220 C. From these trials it was determined that the standard solutions should be injected in the GC at an oven temperature of 210 °C, an injection and detection temperature of 300 °C, an attenuation of 1, and range of 10⁽²¹⁾. Data was plotted with graphing software and the the graphs were found to be linear for both area ratios versus concentrations, and areas of anthracene versus concentrations of anthracene. For low concentrations the GC was set to full deflection range of 1. Due to the extreme amount of noise at low concentrations, this data was used only to find the limits of detection. Figures 5, 6, and 7 illustrate the resulting standard graphs. Figure 8 is the

computer output of the standard plot of area units versus concentration of anthracene in the standard solutions. The equation is shown in Figure 8:

$$\text{Area Units} = 3664 + 905 \times (\text{Anthracene concentration})$$

This equation was used later on to calculate the concentration in extracted solutions. Figures 5, 6, 7, and 8 are in Appendix 6.

4. Preparation of batches of contaminated soil with the compound to be studied

A ratio of 0.5 ml compound solution (5000 ppm of anthracene dissolved in toluene) per gram of soil was used. There was no special criteria except that the concentration of the contaminant had to be large enough that it could be detected and studied on the GC detector, and that the contamination would resemble values encountered on the field (landfills, hazardous waste sites, etc.).

The solution and soil were mixed in a container and after thorough mixing, the solvent (toluene) was allowed to evaporate in a hood under a vacuum. Vacuum was used because toluene took as long as one week to totally evaporate at atmospheric conditions. Anthracene was not lost in considerable quantity because of its low vapor pressure and high melting (216 °C) and boiling (340 °C) points. Then after drying 48 hours, the soil was transferred to a tightly capped glass.

The quartz tube was filled with 15 grams of

contaminated soil held in place by quartz wool plugs. This tube was then placed in the oven which consisted of resistance heaters insulated with silicon and aluminum oxide ceramics. To monitor the temperature of the desorption tube, two chromel/alumel thermocouples were mounted inside a thin quartz tube and placed alongside the desorption tube at the center and end of the soil plug. These thermocouples were in turn attached to a rotary selector switch and Omega Model 115 KC Digital Thermometer.

Desorption was carried by passing a preheated nitrogen stream through the reactor at 30 ml/min. The flow was monitored by a flowmeter that had been previously calibrated with a soap bubble meter.

Initial collection of effluent organics was achieved by passing the effluent gas through two test tubes filled with 23 grams of granulated activated carbon (GAC). To avoid condensation of the organic contaminants in the tubing just before the GAC adsorbers, heat was applied to these tubes. However, this precaution proved to be useless with anthracene due to its high boiling point; i.e. a very high percentage of anthracene collected inside the copper tubes leading to the GAC adsorbers. To avoid this from happening, a three (3) inch glass tube, open at both ends and with glass wool inside was placed between the effluent copper tubing and the GAC adsorbers as shown in Figure 2. No anthracene broke through the glass tube to the GAC

adsorbers. After the trial runs, extractions were done only on the glass tube as described in the Solvent Extractions and Mass Balances Section. The temperature of the resistance heating tapes was also increased. Unfortunately these measures worked only marginally, and anthracene kept depositing in the effluent tubes forcing regular (every other run) washing with acetone to prevent clogging.

Another problem with this setup was that the soil column was not exposed to uniform temperatures with respect to length. To account for this difference, calculations were done with the average temperature of the oven. Table IX lists desorption temperatures.

5. Solvent Extractions and Mass Balances

In order to do mass balance calculations, solvent extractions were done on the contaminated soil, the desorbed soil, and the collection tube. Additional extractions were also done on some samples of uncontaminated soil. No contaminant peaks were observed in the desorbed soil and uncontaminated soil. Based on a calculated detection limit, the concentration of anthracene in the desorbed soil was less than 6 ppm. (See Sample Calculations).

The extractions were performed by adding 20 ml of toluene to the desired material. After vigorous shaking for five minutes, the solution was allowed to settle. From this solution 8 ml was taken and spiked with 1-chloronaphthalene. This internal standard was later abandoned because average

values of anthracene areas were more consistent than values calculated with the internal standard. The same procedure was done with the collection tubes. The tubes were introduced into a small bottle with 20 ml of toluene. An internal standard was later added to 8 ml of this extract solution. These extracts were then injected into the Shimadzu GC using 1 ul portions. All GC settings for these injections were the same as previously used to obtain the standard curves.

Calculations were done for each run, using the standard curves and data obtained from the extracts mass balance. The percent recovery was very small, but this was mainly due to anthracene being deposited in the effluent copper tubes. Extraction results are shown on Table VIII.

TABLE VIII. SUMMARY OF ANTHRACENE EXTRACTIONS

SAMPLE	SOLVENT STANDARD	AVERAGE ANTHRACENE AREA X 10 ⁻⁵	CONC. IN TOL. PPM	CONC. IN SOIL mg/gr
Contaminated Soil Batch 2C	Toluene	9.93	1090.	1.4
Contaminated Soil Batch 2E	Toluene	39.6	4360.	3.8
Desorbed Anthracene	Toluene			
300		-.--	---	-.-
320		3.36	367	0.42
340*		1.09	116	0.13
360*		7.15	785	0.93
380*		2.11	229	0.27
Desorbed Soil	Toluene			
300		ND	---	-.-
320		ND	---	-.-
340		ND	---	-.-
360		ND	---	-.-
380		ND	---	-.-

Notes:

1. ND stands for non detectable
2. Runs with * were done with soil batch 2C. Remaining runs were done with soil batch 2E.
3. Concentrations of anthracene in soils batch 2C and 2E are not the same because in soil batch 2C a film of anthracene was removed with a spatula. With soil batch 2E the extra film of anthracene was mixed into the contaminated soil with a spatula.

6. Condensed Standard Operating Procedure

The standard procedure for the successful thermal desorption of anthracene was as follows:

- i. A clean collection tube was placed at the end of the effluent tube.
- ii. 15 grams of precontaminated soil were weighed.
- iii. This soil was loosely packed in the quartz tube, and both ends were plugged with glass wool.
- iv. At the same time a similar amount of contaminated soil

from the same batch was weighed for extraction.

v. The pyrex tube was consequently inserted inside a preheated electrical oven. The oven temperature was then allowed to become constant.

vi. Hydrogen and air flow rates to the FID, and nitrogen flow rate to the GC column were set at 30 ml/min.

vii. GC settings were allowed to stabilize.

viii. Nitrogen flow to the reactor was set at 30 ml/min.

ix. The opening of this nitrogen valve corresponded to time zero and the six way valve was allowed to stay in the load position for approximately 250 seconds.

x. Injection was achieved by changing the six way valve from load position into inject position. Valve was allowed to stay in injection position for 90 seconds.

xi. Injections were done at regular time intervals and steps were repeated until the area recorded by the integrator was small and enough data point had been obtained.

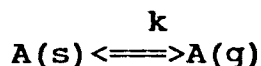
xii. After completion of each run the collection tube, the desorbed soil and the preweighed contaminated soil were extracted with toluene.

xiii. 1 microliter of the extracts were injected in the Shimadzu GC.

xiv. A mass balance was done on anthracene after each run with the Shimadzu GC data.

xv. The previous steps were repeated for each run.

7. Mathematical modeling



For the stoichiometric equation written above the desorption experiments and plug flow data were shown to follow the following equation which represents the instantaneous change in concentration over the change in time.

$$dCa/dt = -kCa$$

Where k is equal a characteristic over all removal rate constant.

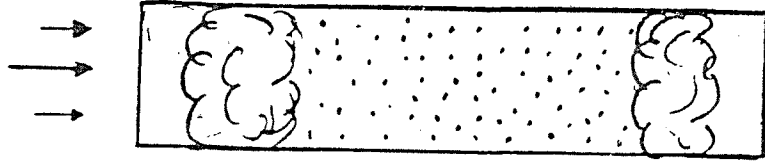
If one assumes a first order reaction this equation can then be integrated to yield:

$$\ln (C(t)/Co) = -kt$$

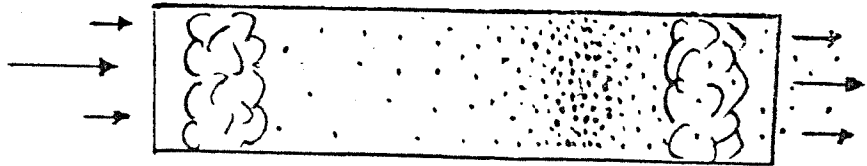
and as a result:

$$C(t)/Co = e^{-kt}$$

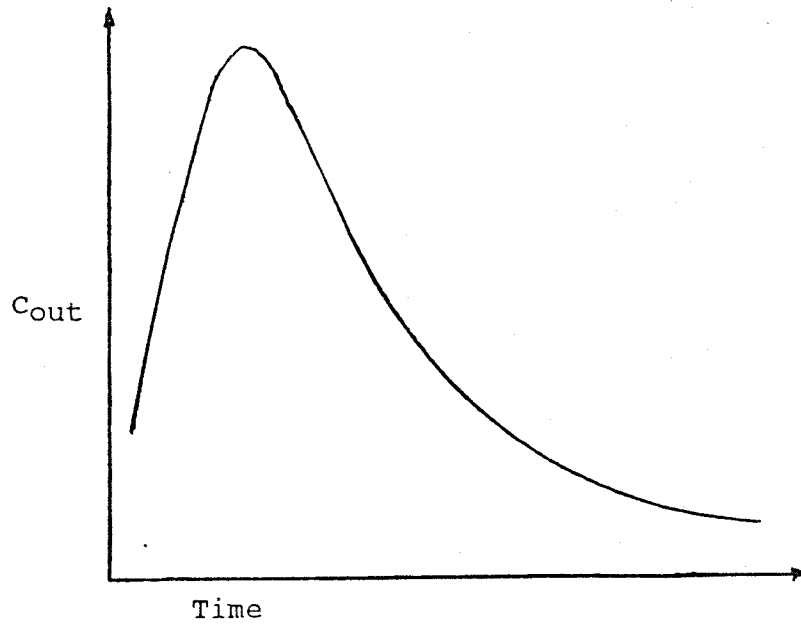
NON-IDEAL THERMAL DESORPTION



a) Uniformly contaminated soil at $t(0)$



b) Contaminated soil at $t(x)$



c) Desorption Profile

FIGURE 4

Where:

- i. $C(t)$ is concentration remaining on soil at time t .
- ii. C_0 is the initial concentration of adsorbed organic contaminant in the soil.
- iii. The coefficient k is a function of Temperature, Dipole Moment, Heat of Vaporization, Purge Gas Flow Rate and Soil Properties.
- iv. t is time in seconds.

At a constant flow rate of a given purge gas:

$$k = A (\text{mass}) e^{-(b \text{ DHvap})/RT} \quad (22)$$

or

$$k = A (\text{mass}) e^{-(b' T_b)/RT} \quad (22)$$

Where:

- i. A , b and b' are fitted empirical constants.
- ii. R is the ideal gas law constant of 1.98 Cal/K*mole
- iii. T is temperature in degrees Kelvin
- iv. DHvap is the change in enthalpy of vaporization.
- v. T_b is the boiling point temperature.

Graphs are shown and illustrated in Appendix 8: Figures Concerning Thermal Desorption Data.

1) Assumptions

In this thermal desorption experiment a few assumptions had to be made in order to use the experimental data.

They were:

- i. The concentration in the soil is uniform throughout the whole runs.
- ii. The concentration in vapor represents concentration effluent from soil at time of measurement.
- iii. These two assumptions in general hold true for the experimental parameters chosen, but in trial runs a rise in effluent concentration followed by logarithmic decay was observed, clearly showing that at certain conditions a better mathematical model is necessary. The proposed physical model for non-ideal thermal desorption is shown on Figure 4. In this more realistic model the organic compound would continuously adsorb and desorb from the soil, and there would be a change in concentration in relation to length of soil matrix. The observed curve is similar to a statistical normal distribution curve and some researchers do use statistics coupled with experimental data to predict the percent of desorption⁽²³⁾.

2) Graphing

The constants A and B were obtained by a least squares computer program, Plotrax, with the computer outputs

and graphs being shown and illustrated in Appendix 8.

3) The Freudlich equation

The Freudlich equation is applicable in dilute solutions and it is represented by the following empirical expression:

$$C^* = k [v (C_o - C^*)$$

Where:

C^* = Final equilibrium concentration value of solution.

C_o = Initial concentration value of solution.

v = Volume of solution.

k = Constant at equilibrium.

This mathematical model describes distribution of a substance in solution (mixed with an adsorbent) between the adsorbent and the solution at equilibrium⁽²⁴⁾. This equation can be used to calculate the concentration of a contaminant in the gaseous phase if the concentration of adsorbed contaminant is known, or vice versa. The Freudlich equation would not be able to predict the concentration over time in the thermal desorption experiment.

8. Results

The results from this experiment confirmed that the mathematical modeling will predict the desorption rate within the chosen parameters. The constants of the equation discussed in the mathematical modeling section are shown in Table IX:

TABLE IX. SUMMARY OF PARAMETERS FOR MATHEMATICAL MODELING OF ANTHRACENE DESORPTION

CENTER TEMP. (°C)	AVG. TEMP. (°C)	AVG. TEMP. (°K)	Cao (ppm)	k(10 ⁴) 1/sec	(Correlation factor) ² , r ²
300	282	555	4800.	4.256	0.890
320	300	573	4800.	9.234	0.785
340	315	588	1400.	11.96	0.677
360	335	608	1400.	13.90	0.782
380	350	623	1400.	18.61	0.548

The equations correlated well except the linear equation obtained for desorption at 380 °C.

The constant of the reaction were plotted versus the inverse temperature and the following equation was obtained:

$$k = 7.86 \times 10^3 \text{ (min}^{-1}\text{)} e^{-2.0 \text{ DHvap/RT}}$$

The correlation factor for this fitted equation was 0.95.

9. Discussion of Results

The results obtained in this experiment demonstrated that within the experimental parameters chosen, the mathematical model will be followed.

The constants in the equation $k = A e^{-(b \text{ DHvap/RT})}$, obtained from previous research(1)(2)(22), were found to be $4.51 \times 10^{10} \text{ min}^{-1}$ and 2.35 for A and b, respectively. From the experiments done in this research, A and b were found to be $7.86 \times 10^3 \text{ min}^{-1}$ and 2.0 respectively. The constant b agreed within 15 % with the previous values but A differed

by several orders of magnitude. (See Appendix 9: Previous Research Data). Summary of parameters from previous experiments and this thesis are shown below in Table X.

TABLE X. SUMMARY OF PARAMETERS FOR MATHEMATICAL MODELING OF THERMAL DESORPTION

COMPOUND	Ai min-1	Bi	Correlation Factor r
Trichlorobenzene	4.86×10^{10}	2.30	0.99
Hexachlorobenzene	7.29×10^{10}	2.48	0.96
Chloronaphthalene	1.39×10^{10}	2.28	0.99
m-dichlorobenzene	1.28×10^5	2.00	0.99
Anthracene	7.86×10^3	2.0	0.95
AVERAGE	2.71×10^{10}	2.2	N/A

N/A = Not Applicable

Due to condensation of anthracene in the effluent copper tubes, the percent recovery obtained from the mass balances was very poor. This problem may be solved by increasing the temperature of the heating resistance tapes around the copper tubing. Another problem with the apparatus was that the temperature profile changed along the oven containing the quartz tube filled with soil. Calculations were done using the average temperature between the center of the soil tube and the side.

III. CONCLUSIONS

1. Thermal treatment will reduce organic contaminants of volatility similar or higher than compounds studied in this and previous work in this laboratory from soil to non-detectable levels.

2. Thermal desorption of anthracene at the chosen experimental parameters will follow the proposed simple mathematical model:

$$C(t)/C_0 = e^{-kt}$$

The equation for constant k is:

$$k = 2.71 \times 10^{10} \text{ (min}^{-1}\text{)} e^{-2.2 \text{ DHvap/RT.}}$$

3. Desorption rates increase as the temperature increases.

4. Thermal desorption of organic contaminants is feasible and as discussed in the Previous Research Section, it has been proven to be both economical and reliable in field testing.

5. Preliminary experiments have shown that desorption rate is influenced by the flow rate of the purge gas.

6. Plug flow data suggests that desorption rate depends on the packing material.

7. The plug flow empirical equation fit all data with a square of the correlation factor greater than 0.93.

IV. SUGGESTED AREA FOR FURTHER STUDY

1. Further study should be done where only the gas phase is heated, and the temperature of the soil surface is monitored.
2. The effect of various purge gas flows and increased temperatures upon the desorption rate should be determined.
3. Construction and testing of a bench scale rotary kiln. Continuous feed would probably be necessary to treat large quantities of soil.
4. Studies will have to be done with soil matrices that more closely resemble field conditions. This is because adsorption of organic compounds to soil may increase as the total organic, oxygen and sulfur content in the soil increases(25).
5. A new mathematical model for the plug flow experiments must be developed. The present simplified model does not yield enough information.

V. SAMPLE CALCULATIONS

1. Bulk and Actual Densities Calculations:

Soil:

Weight of 20 ml of soil = 23.65 grams

Bulk Density = Weight / Total Volume = 1.18 grams/ml

This amount of soil displaced 10 ml of water.

Actual Density = Weight / Displaced Water Volume =
= 2.36 grams /ml

2. Linear Velocity Calculations:

Vl = Linear velocity through the soil (cm/sec)

Fr = Air flow rate as measured with bubble meter
(30 cm³/min)

Av = Cross sectional void area in quartz tube where soil is
(cm²)

At = Cross sectional area area of quartz tube (cm²)

As = Cross sectional area occupied by the soil (cm²)

r = Inside radius of quartz tube (cm)

Vt = Volume of tube in 1 cm length of tube (cm³)

Vs = Volume of soil in 1 cm length of tube (cm³)

Db = Bulk density of soil (gm/cm³)

Da = Actual density of soil (gm/cm³)

Vl = Fr / Av

Av = At - As

Av = At (1 - Vs / Vt)

Dividing both Vs and Vt by 1 gr of soil:

Vs / Vt = Da / Db

Av = At (1 - Da / Db)

At = Pi * r² = Pi * .50² = .78 cm²

Av = .78 (1 - .8 / 1) = .16 cm²

Vl = 30 / .16 = 190 cm /min = 3.2 cm/sec.

Linear velocity through soil was 3.2 cm / sec.

3. Initial Standard Solution Calculations:

Calculations to find how much anthracene in a given 5000 ppm solution with 200 ml of toluene as solvent:

A = Anthracene

T = Toluene

(ppm)_w = Parts per million on a weight basis

Gt = Weight of toluene

D = Density of toluene (gr/ml) = .8667 gr/ml

V = Volume of solution (toluene) (ml)

Conc.toluene = Concentration of Anthracene in Toluene (ppm)_w

f = Some conversion factor

$$\text{Conc.toluene} = A / (V \times D \times f)$$

a) The ratio of weight of anthracene to toluene for a 5000 ppm solution was calculated in a mass basis.

Desired concentration = 5000 (ppm)_w of anthracene in toluene
 $5000 \text{ gr A} / 10^6 \text{ gr T} = 5 \times 10^{-3} \text{ gr A/gr T}$

b) The amount of anthracene in a 200 ml initial solution to obtain the desired concentration was determined.

$$Gt = V \times D = 200 \times .8667 = 17.334 \text{ gr T}$$

$$\# \text{ gr A/Gt} = 5 \times 10^{-3} \text{ A/T}$$

$$A = 5 \times 10^{-3} \times 17.334$$

$$A = .8667 \text{ gr}$$

c) .8997 is the amount of anthracene required in 200 ml of toluene, to make a 5000 ppm solution.

4. Calculation of Concentration of Anthracene from Extracted Soil:

13.27 grams of contaminated soil batch 2C were extracted with 20 ml of toluene and extract was injected in GC. The concentration was found with following equation:

$$\text{GC Area} = 3664 + 905 \times (\text{Concentration of Anthracene in ppm})$$

$$\text{GC Area} = 9.93 \times 10^5$$

$$\text{Conc.toluene (ppm)} = (9.93 \times 10^5 - 3664) / 905$$

$$\text{Conc.toluene (ppm)} = 1090 \text{ ppm}$$

$$\text{Conc.toluene} = A / (V \times D \times f)$$

$$A = (\text{Conc.toluene} \times V \times D \times f) = 19.0 \text{ mg of Anthracene in 20 ml of Toluene}$$

$$\text{Conc.soil} = A \times f_1 / W_{\text{soil}}$$

where:

Conc.soil = Concentration of Anthracene in soil in ppm_w

A = Anthracene in mg

f₁ = Some conversion factor = 1000

W_{soil} = Weight of soil in grams

From this equation the concentration of anthracene in soil was calculated:

$$\text{Conc. soil} = 19.0 \times 1000 / 13.27 = 1400 \text{ ppm}$$

Concentration of anthracene in soil batch 2C was 1400 ppm.

APPENDIX 1: PHYSICAL CONSTANTS OF PREVIOUSLY SELECTED COMPOUNDS

TABLE XI. PHYSICAL CONSTANTS OF PREVIOUSLY SELECTED COMPOUNDS

COMPOUND	BOIL. POINT (°C)	FORMULA WEIGHT gr/mole	HEAT OF VAP. cal/mole	DENS. Gr/cm ³	EMPIRICAL FORMULA
Dichloro-methane	40.0	84.9	7572.3	1.4242	CH ₂ Cl ₂
1,1,1-Tri-chloroethane	74	133.4	8012.7	1.3390	C ₂ H ₃ Cl ₃
1,2,4-Tri-chlorobenzene	214	181.4	11425.1	1.4542	C ₆ H ₃ Cl ₃
Tetrachloro-methane	77	153.8	8271.5	1.5940	CCl ₄
Chlorobenzene	132	112.6	10098.0	1.1058	C ₆ H ₅ Cl
1-Chloro-naphthalene	263	162.6	13570.5	1.9834	C ₁₀ H ₇ Cl
Acetone	56	58.1	7641.5	0.7899	CH ₃ COCH ₃
Toluene	111	92.0	9368.5	0.8669	C ₇ H ₈

APPENDIX 2: PLUG FLOW DATA

TABLE XII. RETENTION TIMES IN THE GASCHROM COLUMN

Temperature (°K)	Retention Times in Minutes			
	p-Xylene	o-Xylene	p-Cl toluene	Ethyl Benzene
353	---.---	1.17	---.---	---.---
363	---.---	---.---	---.---	0.89
373	0.705	0.85	1.00	0.78
383	0.615	---.---	---.---	---.---
393	0.525	0.62	0.75	0.60
403	0.47	---.---	---.---	---.---
413	0.44	0.50	0.56	0.505
423	0.39	---.---	---.---	---.---
433	0.365	0.45	0.45	0.43
443	0.34	---.---	---.---	---.---
453	0.335	0.375	0.40	0.38
473	---.---	0.33	0.355	0.37
493	---.---	---.---	0.32	---.---

---.--- means no data collected

TABLE XIII. RETENTION TIMES IN THE SAND COLUMN

Temperature (°K)	Retention Times in Minutes			
	p-Xylene	o-Xylene	p-Cl toluene	Ethyl Benzene
373	3.60	1.68	2.28	1.35
393	1.24	1.18	1.17	1.06
413	1.07	0.97	0.93	0.88
433	0.87	0.83	0.72	0.70
453	0.64	0.58	0.61	0.54
473	0.54	0.46	0.45	0.45
493	0.42	0.38	0.37	0.36
513	0.35	0.32	0.31	0.31

---.--- means no data collected

TABLE XIV. RETENTION TIMES IN THE SOIL COLUMN

Temperature (°K)	Retention Times in Minutes			
	p-Xylene	o-Xylene	p-Cl toluene	Ethyl Benzene
373	4.66	5.87	---	---
393	1.58	1.71	2.10	1.29
413	1.17	1.23	1.24	1.09
433	0.85	0.94	1.01	0.86
453	0.66	0.75	0.79	0.68
473	0.52	0.58	0.62	0.54
493	0.43	---	0.50	0.44
513	0.36	---	0.44	0.36

--- means no data collected

TABLE XV. RETENTION TIMES IN THE SILICA-GEL COLUMN

Temperature (°K)	Retention Times in Minutes			
	p-Xylene	o-Xylene	p-Cl toluene	Ethyl Benzene
453	---	32.35	---	12.96
473	14.79	14.38	---	6.96
493	7.18	7.44	---	4.11
513	2.62	2.66	---	2.43

--- means no data collected

APPENDIX 3: SOIL CHARACTERISTICS

TABLE XVI. EMISSION SPECTROGRAPHIC ANALYSIS REPORT

SAMPLE	RANGE OF PERCENT BY MASS
Al	>10%
B	0.001-0.01%
Ba	0.01-0.1%
Ca	1.0-10%
Cr	0.001-0.1%
Cu	0.001-0.01%
Fe	1.0-10%
K	1.0-10%
Mg	0.1-1.0%
Mn	0.1-1.0%
Na	>10%
Ni	0.001-0.01%
Si	>10%
Sr	0.001-0.01
Ti	0.1-10%
V	0.001-0.01%
Zn	0.01-0.1%
Zr	0.001-0.1%

NOTES:

1. Results are semiquantitative. Accuracy and sensitivity are element and matrix dependent.
2. Analysis date 2/11/87.

X-RAY DIFFRACTION RESULTS

The x-ray diffraction analysis was made on the sample after grinding to less than 270 mesh. It showed the major compound is silicon dioxide (sand), with some feldspar, and possibly iron oxides. The feldspars albite, andesine, anorthite, anorthoclase, and labradorite all have same diffraction patterns and any one of them or all are possibly in the sample. These are sodium aluminum silicates or

calcium aluminum silicates (some of which are potassium rich) or mixtures of the two. Lines that may be hematite (Fe_2O_3) and magnetite (Fe_3O_4) are present, but there is too much interference from the lines of the other components in the pattern to verify their presence.

The Philips x-ray diffraction unit used for the work is calibrated every three months with a silicon standard obtained from Philips. The particle size of the standard is 1 micron or less.

Note: Analysis and report done by Labtech Corp.,
1275 Bloomfield Avenue, Fairfield, New Jersey 07006.

TABLE XVII. SOIL PARTICLE SIZE DISTRIBUTION

Particle Diameter (inches)	Mesh Size	Mass Grams	% of Total	% of Useable
-	>100	2000	6.9	-
-	100	2910	10.0	-
.0086	70	480	1.7	4.5
.0098	60	3200	11.0	29.8
.0165	40	7060	24.3	65.7
-	<40	13,400	46.1	-
Total Mass	29,050 grams			
Useable Mass	10,740 grams			
Bulk Density	1.0 gram/cc			
Actual Density (by water displacement)	0.8 grams/cc			

APPENDIX 4: STANDARD SOLUTIONS OF ANTHRACENE

TABLE XVIII. STANDARD SOLUTIONS OF ANTHRACENE

Anthracene Conc. in Toluene Solution (ppm)	Initial Volume (ml)	Final Volume (ml)	Final (ppm)
5000.	1.0	10.0	500.
5000.	10.0	20.0	2500.
5000.	2.0	10.0	1000.
2500.	1.0	10.0	250.
500.	1.0	5.0	100.
250.	2.0	10.0	50.
50.	2.0	10.0	10.
50.	1.0	10.0	5.
5.	1.0	5.0	1.

APPENDIX 5: THERMAL DESORPTION DATA

TABLE XIX. ELAPSED TIME AND UNIT AREAS
FOR ANTHRACENE DESORPTION

Elapsed Time (seconds)	Areas	Elapsed Time (seconds)	Areas
Temperature 300 °C		Temperature 320 °C	
Run Number 1		Run Number 1	
800	139440	122	1055366
1250	71068	500	321550
1600	62050	800	146155
2710	28410	1250	73615
4400	21256	1600	61228
4900	17058	2120	33321
5405	17216	2500	39471
6040	14794	3000	19134
6560	13194		
Run Number 2		Run Number 2	
120	341200	122	1517805
500	113100	500	64113
800	115600	800	150629
1260	79371	1250	208683
1600	65876	1600	174235
2100	48425	2100	93320
2500	40502	2500	39171
3000	32343	3020	25393
3500	26854	3600	18829
4000	23473	4100	14036
4500	19207	4500	10273
5000	17684		
Temperature 340 °C		Temperature 360 °C	
Run Number 1		Run Number 1	
120	72272	180	69526
500	15551	660	9712
800	6658	1060	5306
1230	3397	1460	4188
1550	2746		
1900	2141		
2400	1562		
Run Number 2		Run Number 2	
120	935295	180	7364
500	25777	520	10335
800	15224	880	6887
1180	12525	1280	5068
1500	10335	1680	4011
1850	9159	2100	3160
2300	5406		
2800	3210		

TABLE XIX. ELAPSED TIME AND UNIT AREAS
FOR ANTHRACENE DESORPTION (CONTINUATION)

Elapsed Time (seconds)	Areas	Elapsed Time (seconds)	Areas
Temperature 380 °C			
Run Number 1			
800	18081		
1180	9194		
1500	8046		
1850	6930		
2300	4310		
Run Number 2			
500	12776		
800	8013		
1180	4719		
1500	3067		

APPENDIX 6: FIGURES CONCERNING THE STANDARD CURVES

Area vs Concentration

Anthracene (10-500 ppm)

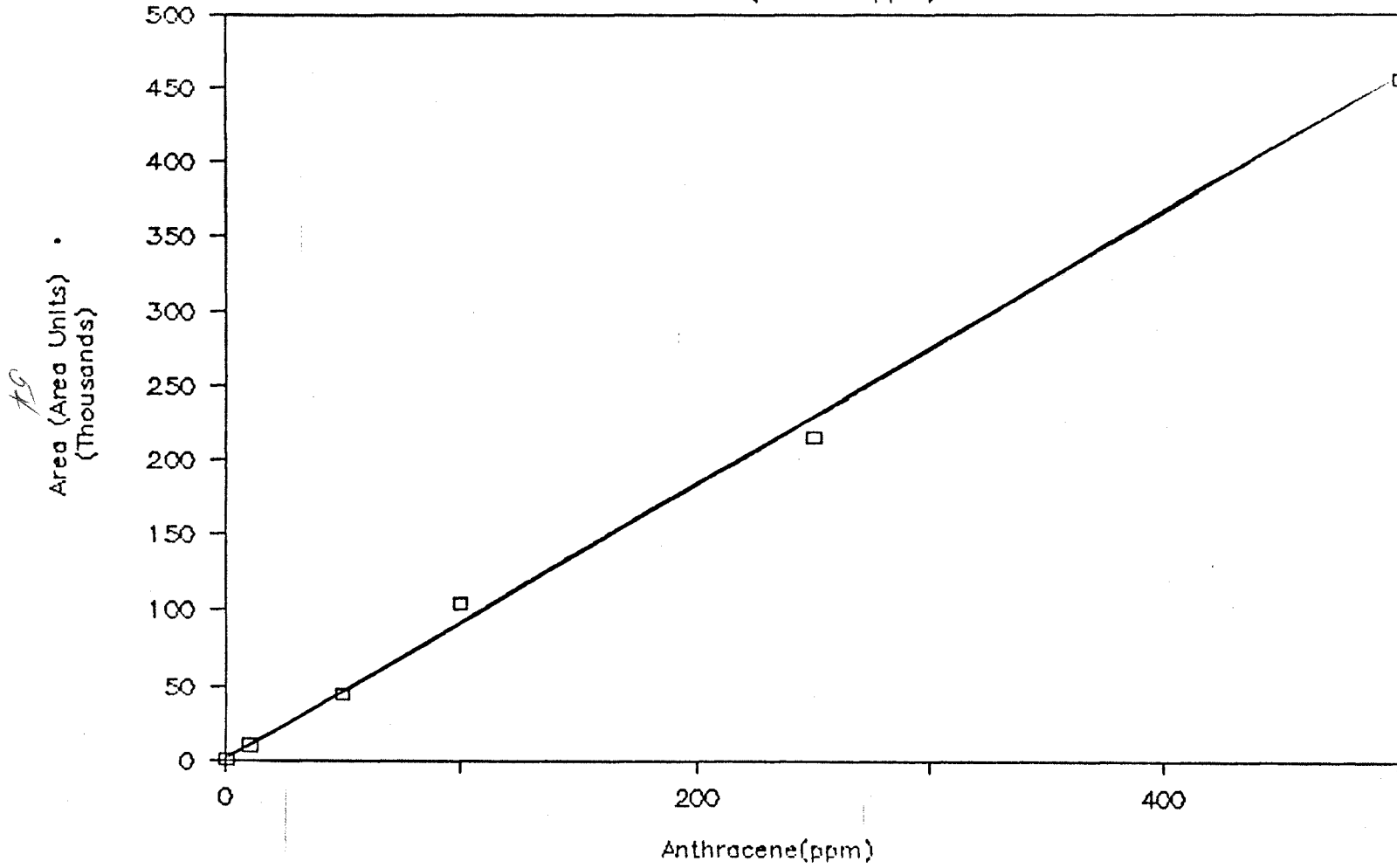


Figure 5. Standard Curve to Measure Anthracene Concentration (10-500 PPM)

Area vs Concentration

Anthracene (250-5000 ppm)

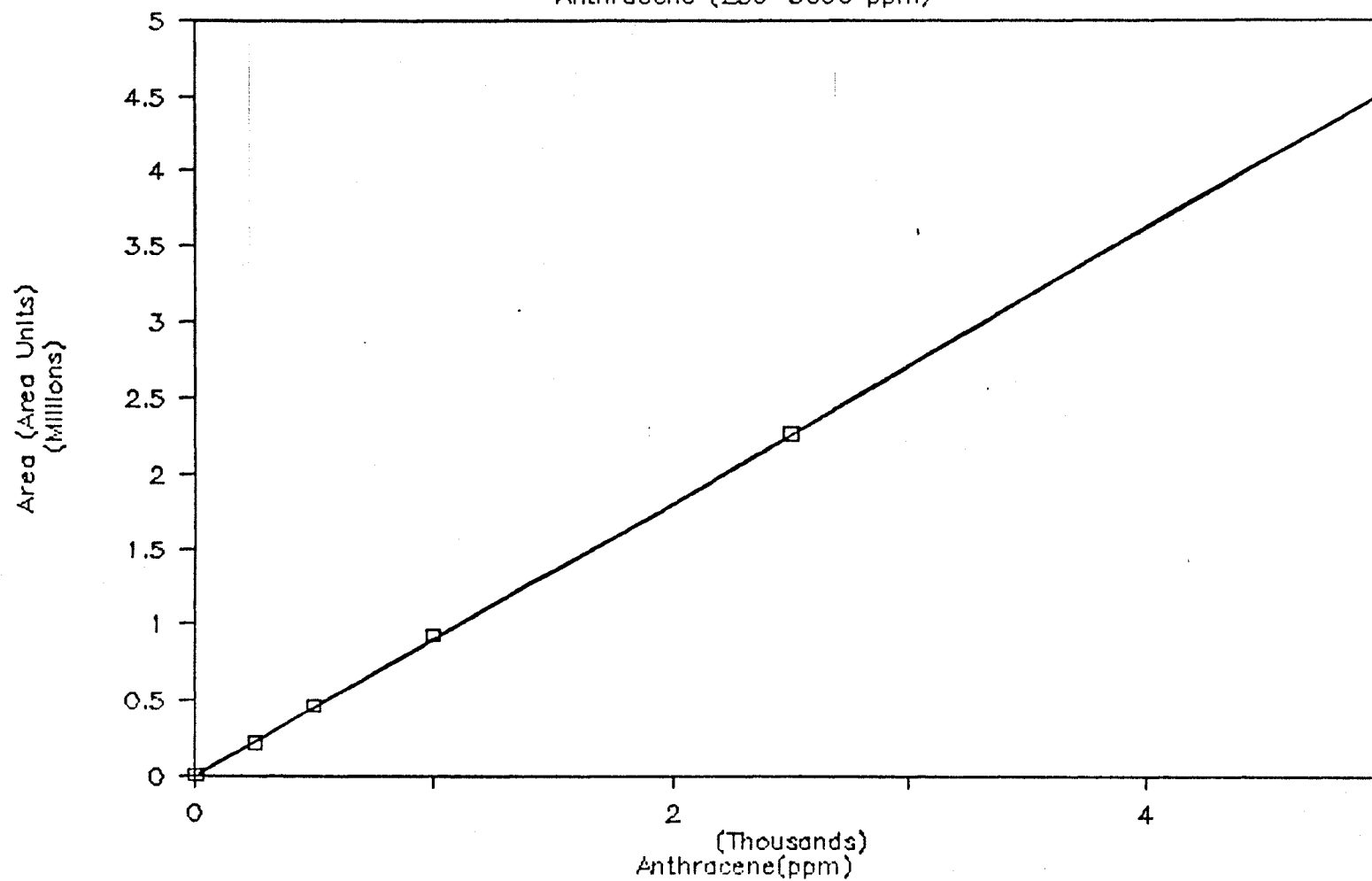


Figure 6. Standard Curve to Measure Anthracene Concentration (250-5000 PPM)

Ratio of Areas vs Concentration

[Anthracene]/[1-Chloronaphthalene]

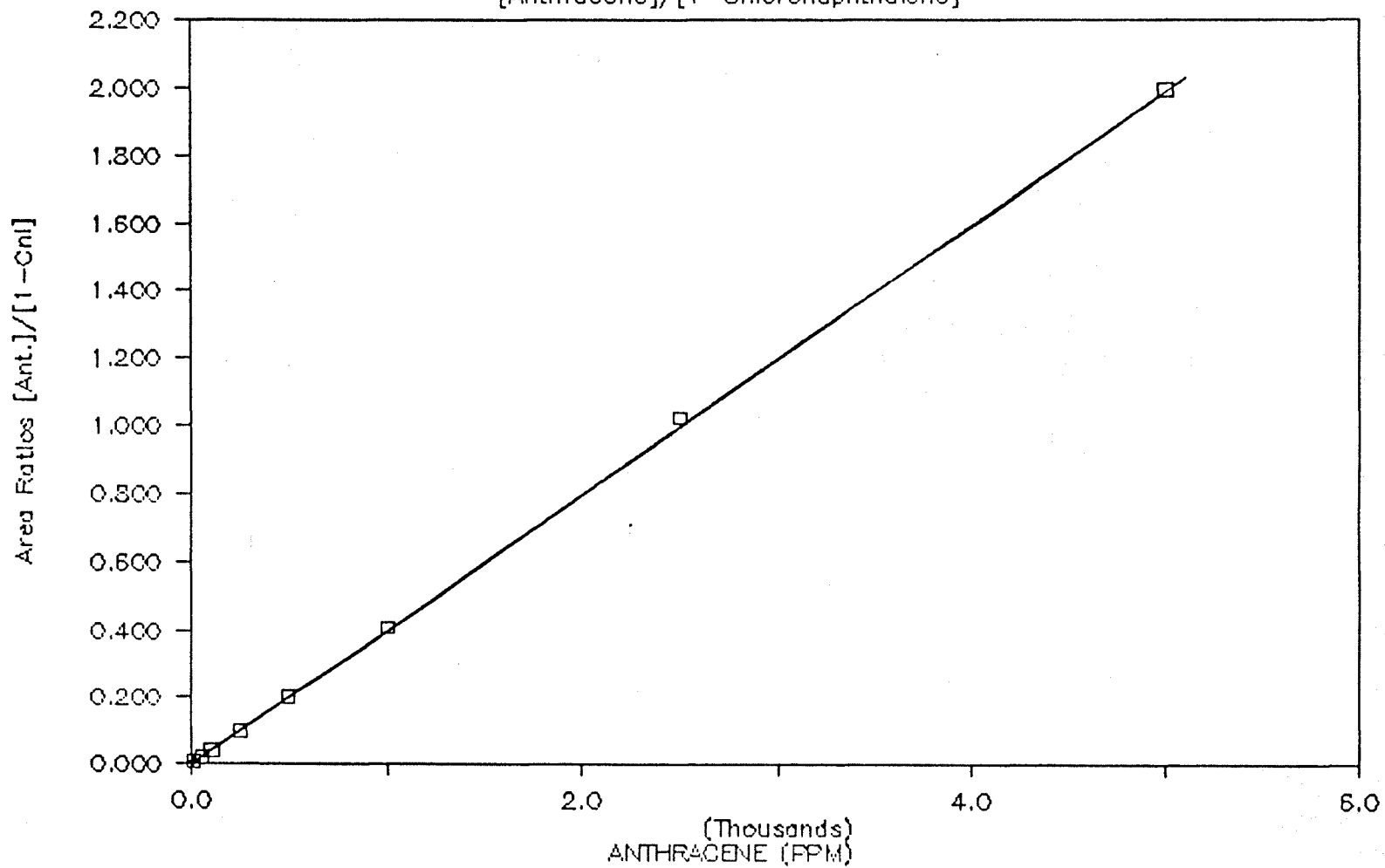


Figure 7. Standard Curve to Measure Anthracene Concentration (10-5000 PPM)

Figure 8. Area Units Versus Anthracene Concentration (PPM) Computer Output

```

Regression coefficients ***** ANALYSIS OF VARIANCE *****
b0= 3664.333 *
b1= 905.9921 *
* SOURCE OF SUMS OF DEGREES OF MEAN *
* VARIATION SQUARES FREEDOM SQUARE *
* * * * *
* regression 1.765308E+13 1 1.765308E+13 *
* error 6.949535E+08 6 1.158256E+08 *
* total 1.765377E+13 7 *
* * * * *
* f test= (MSR/MSE)= 152410.9 *
* coefficient of determination r^2= .9999607 *
* adjusted r^2= .999954 *
* coefficient of correlation r= .9999803 *
* number of data points= 8 *
*****

```

EQUATION: $y = 3664.333 + 905.9921 x$
y = Area Units
x = Anthracene Concentration (PPM)

APPENDIX 7: FIGURES CONCERNING PLUG FLOW RESULTS

Plug Flow

Soil Column

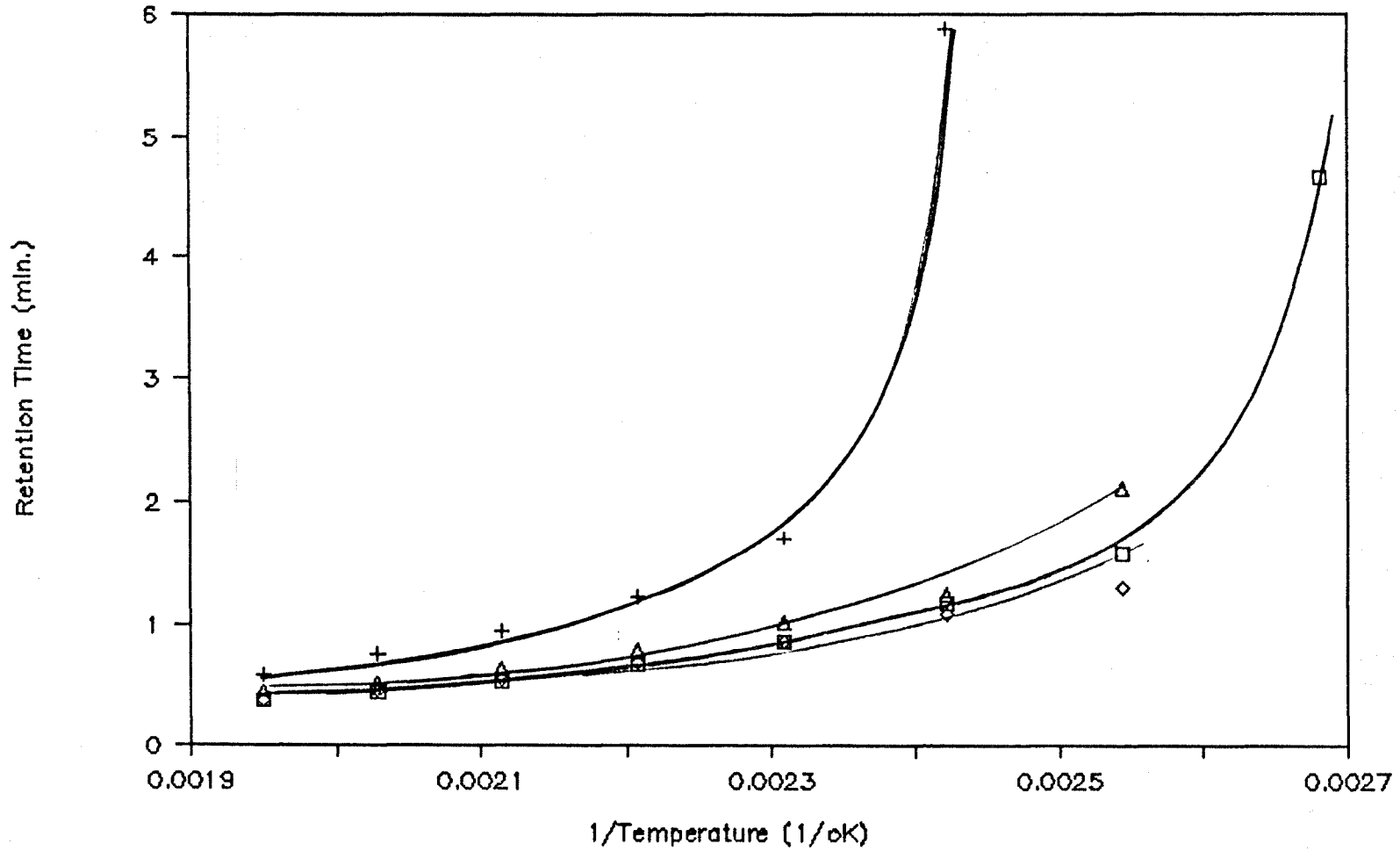


Figure 9. Graph of Retention Times (min.) Versus Inverse Temperature (1/K) for the Soil Column

Plug Flow

Sand Column

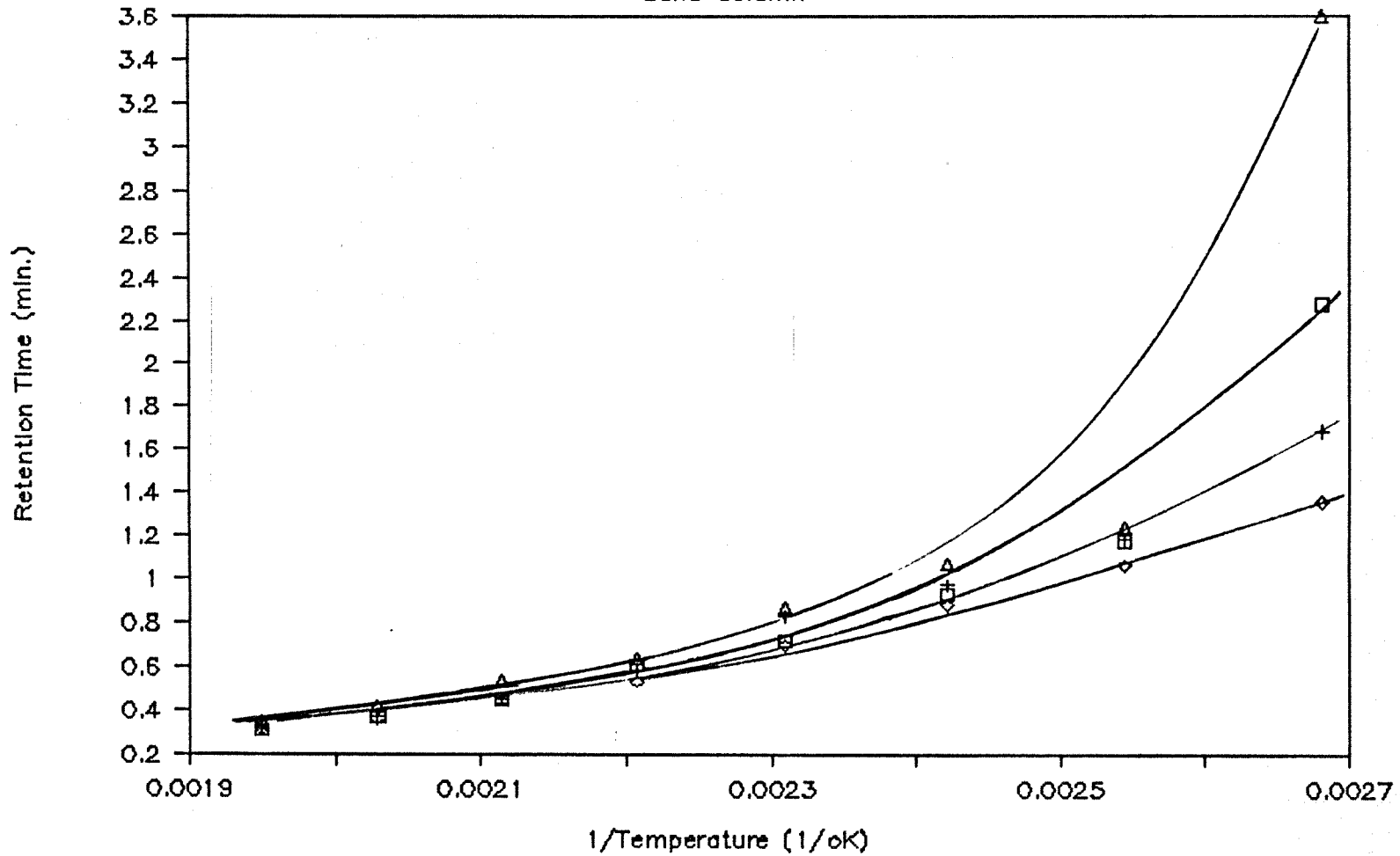


Figure 10. Graph of Retention Times (min.) Versus Inverse Temperature (1/K) for the Sand Column

Plug Flow

Gaschrom-R

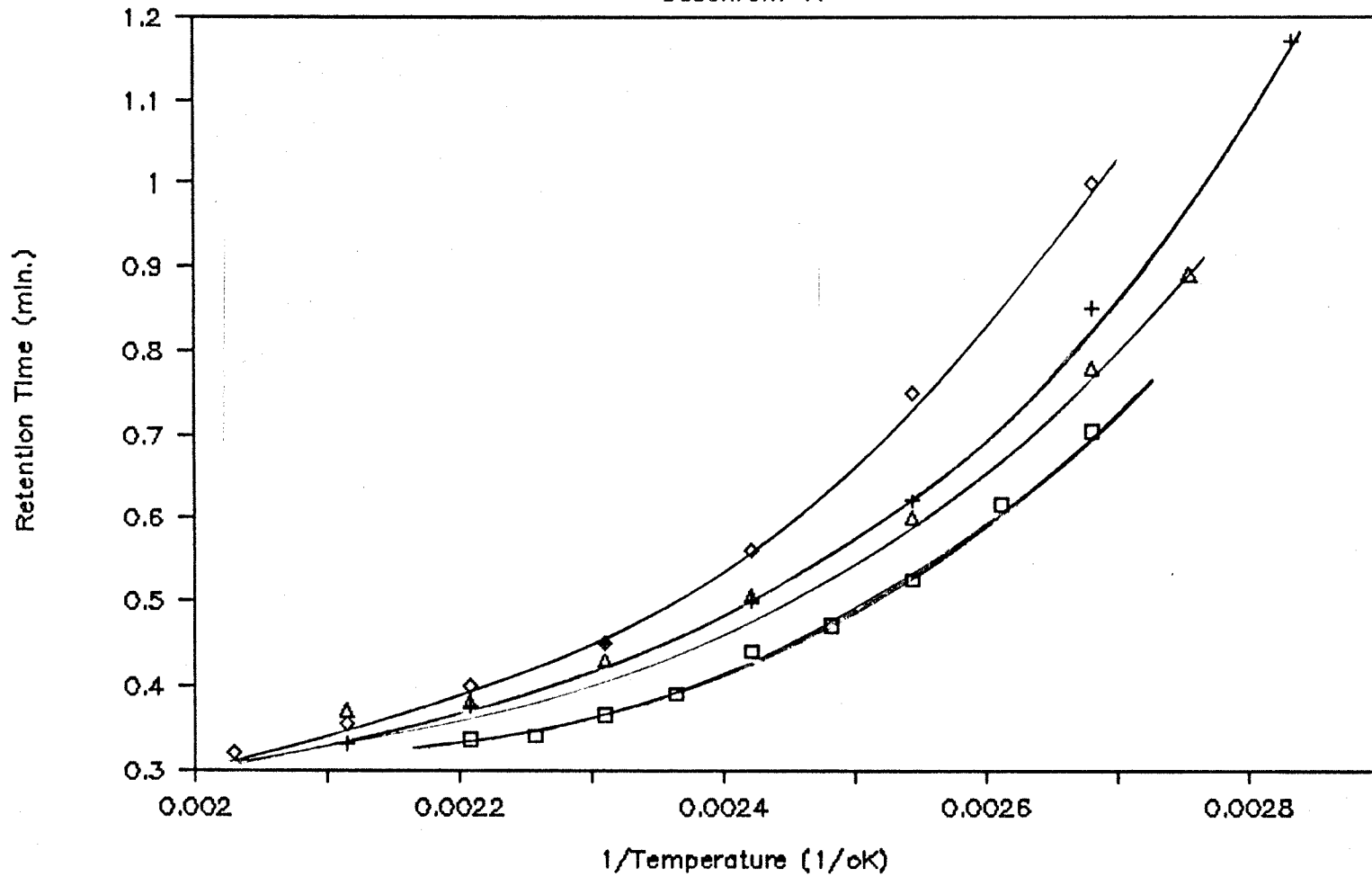


Figure 11. Graph of Retention Times (min.) Versus Inverse Temperature (1/K) for the Gaschrom-R Column

Plug Flow

Silica-Gel Column

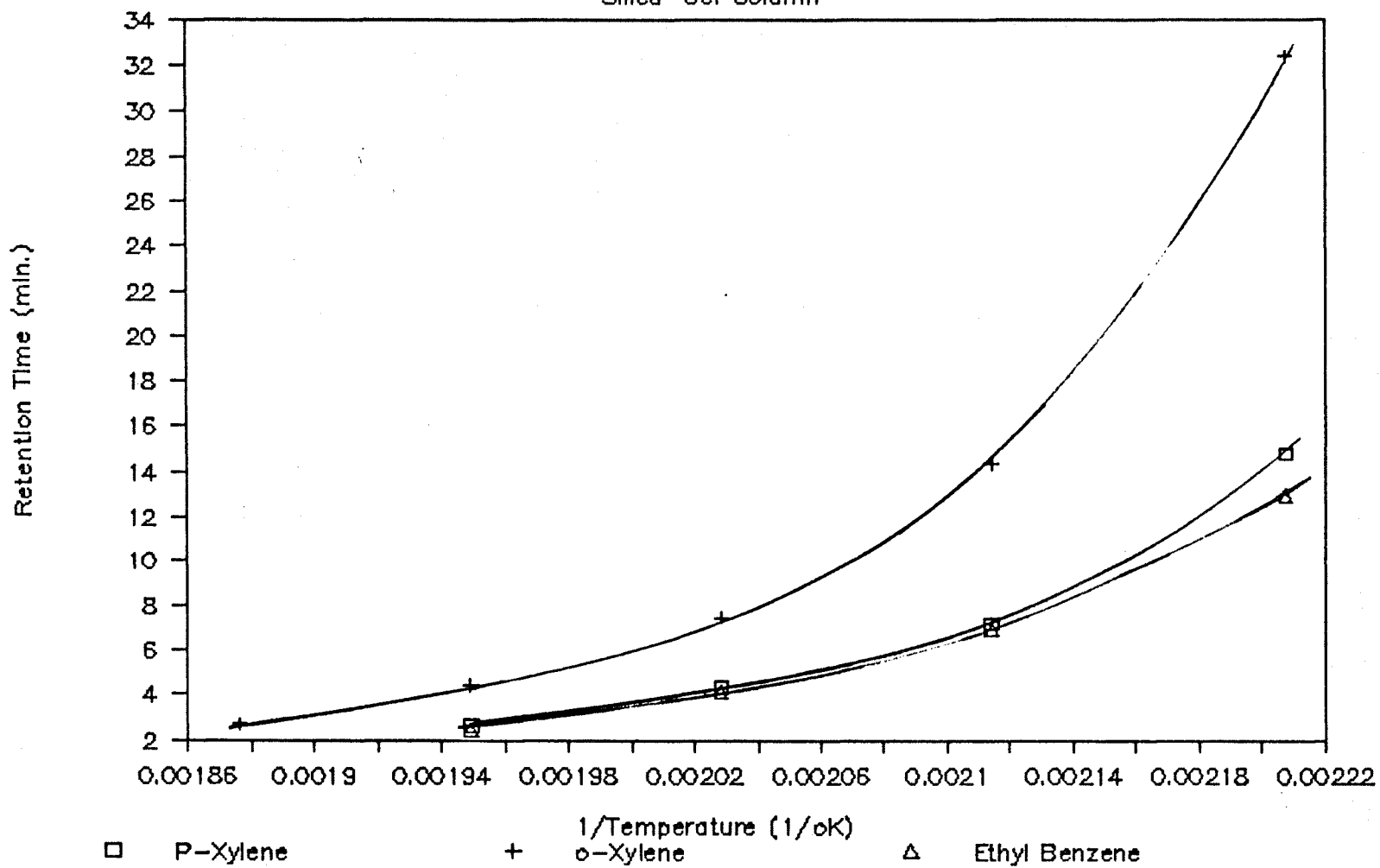


Figure 12. Graph of Retention Times (min.) Versus Inverse Temperature (1/K) for the Silica-Gel Column

APPENDIX 8: FIGURES CONCERNING THERMAL DESORPTION RESULTS

Conc. of Anthracene vs Time

At 300 °C

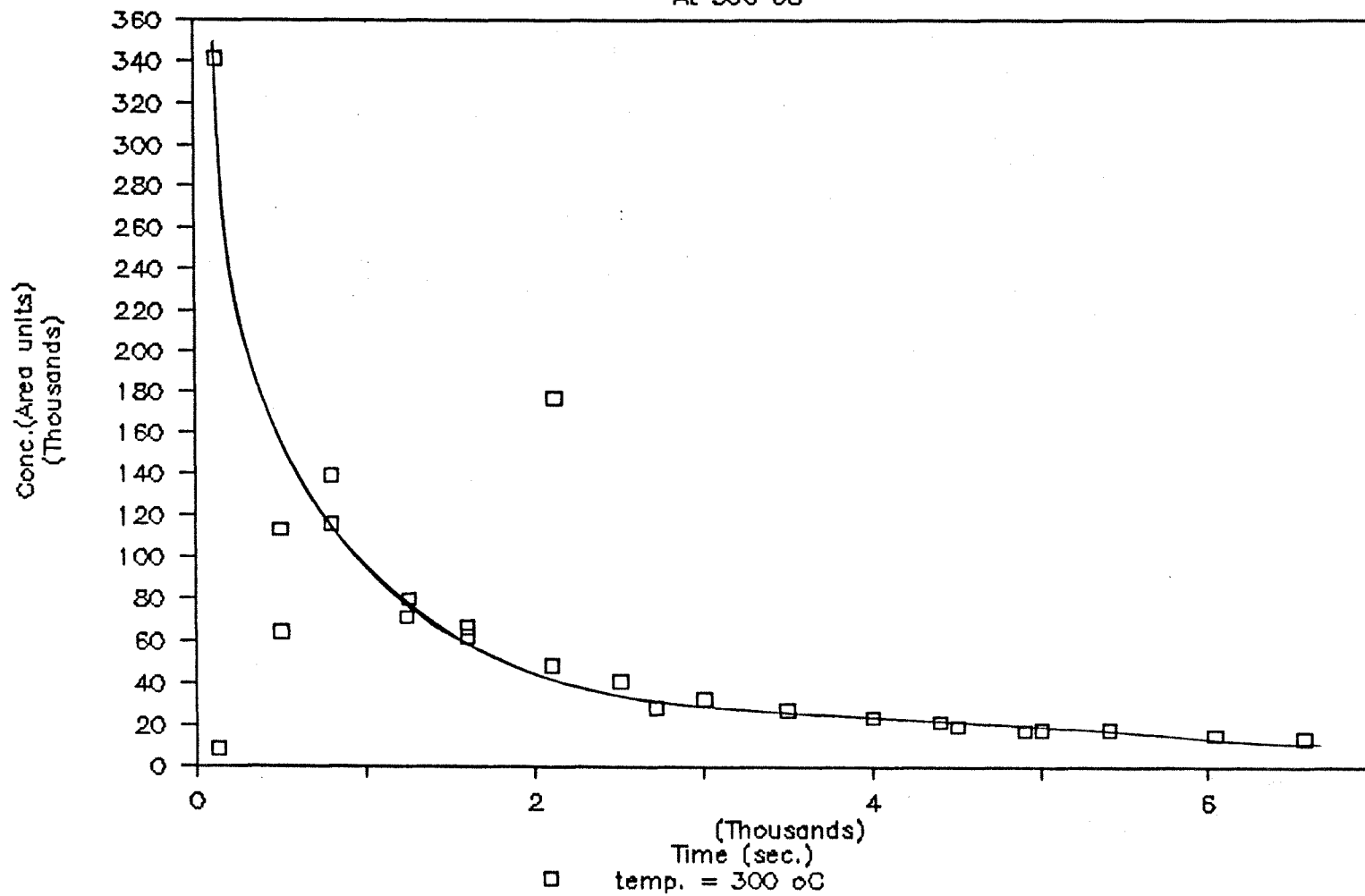


Figure 13. Graph of Thermal Desorption of Anthracene at 300 °C

Figure 14. Thermal Desorption of Anthracene at 300 oC Computer Output

```

Regression coefficients ***** ANALYSIS OF VARIANCE *****
b0= 145012 *
b1= -4.256024E-04 *
* SOURCE OF SUMS OF DEGREES OF MEAN *
* VARIATION SQUARES FREEDOM SQUARE *
* * * * *
* regression 13.79142 1 13.79142 *
* error 1.705004 19 8.973707E-02 *
* total 15.49643 20 *
* * * * *
* f test= (MSR/MSE)= 153.687 *
* coefficient of determination r^2= .8899743 *
* adjusted r^2= .8841835 *
* coefficient of correlation r= -.9433845 *
* number of data points= 21 *
*****

```

EQUATION: $y = 145012 e^{-4.256024E-04 x}$

y = Concentration of Anthracene in Area Units
x = Time in Seconds

Conc. of Anthracene vs Time

At 320 °C

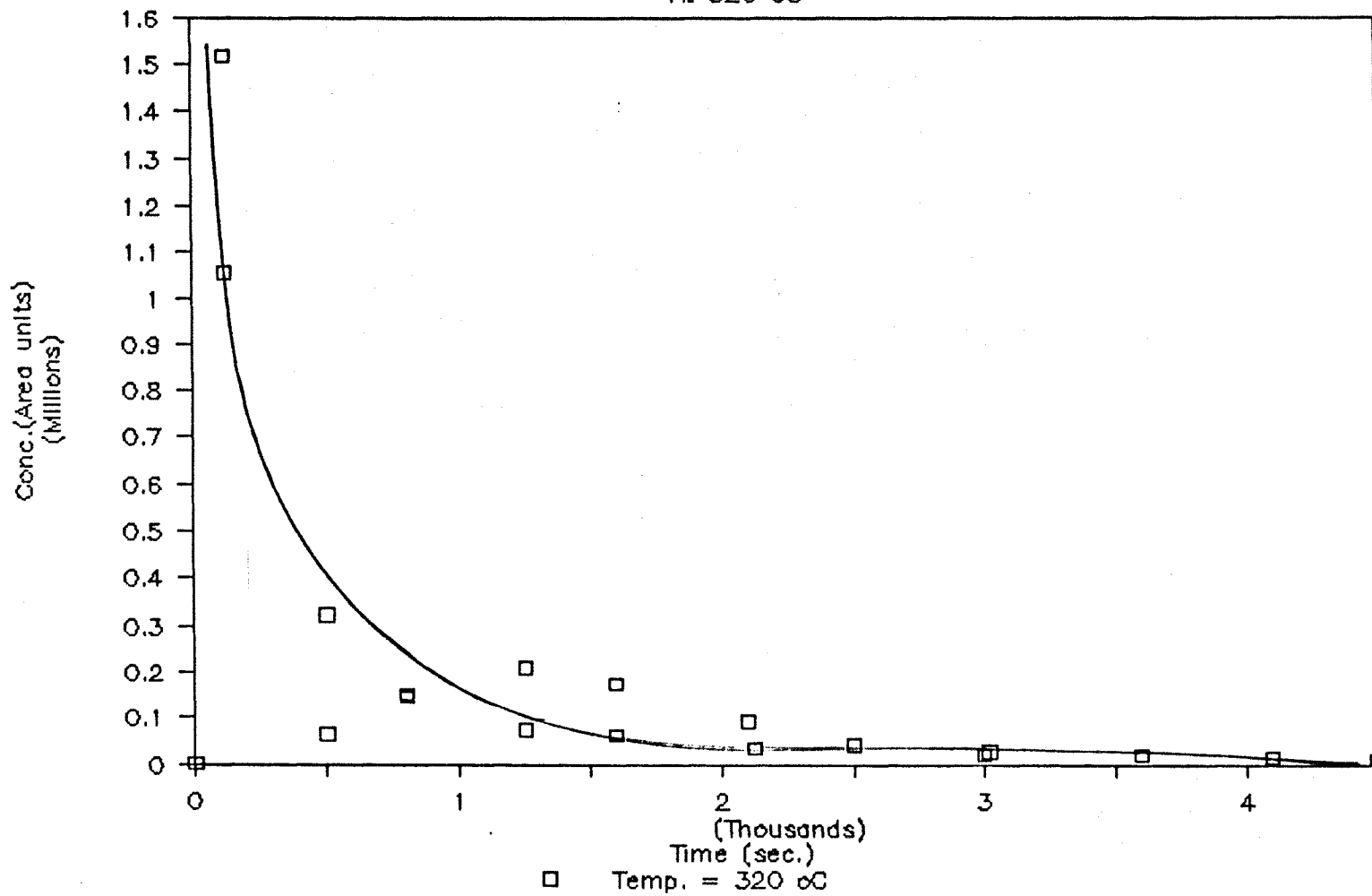


Figure 15. Graph of Thermal Desorption of Anthracene at 320 °C

Figure 16. Thermal Desorption of Anthracene at 320 oC Computer Output

```

Regression coefficients ***** ANALYSIS OF VARIANCE *****
b0= 444412.4 *
b1= -9.233521E-04 *
* SOURCE OF SUMS OF DEGREES OF MEAN *
* VARIATION SQUARES FREEDOM SQUARE *
* * * * *
* regression 26.81333 1 26.81333 *
* error 7.326725 17 .4309838 *
* total 34.14006 18 *
* * * * *
* f test= (MSR/MSE)= 62.21425 *
* coefficient of determination r^2= .7853921 *
* adjusted r^2= .7727681 *
* coefficient of correlation r= -.8862235 *
* number of data points= 19 *
*****

```

EQUATION: $y = 444412.4 e^{-9.233521E-04 x}$
y = Concentration of Anthracene in Area Units
x = Time in Seconds

Conc. of Anthracen vs Time

At 340 °C

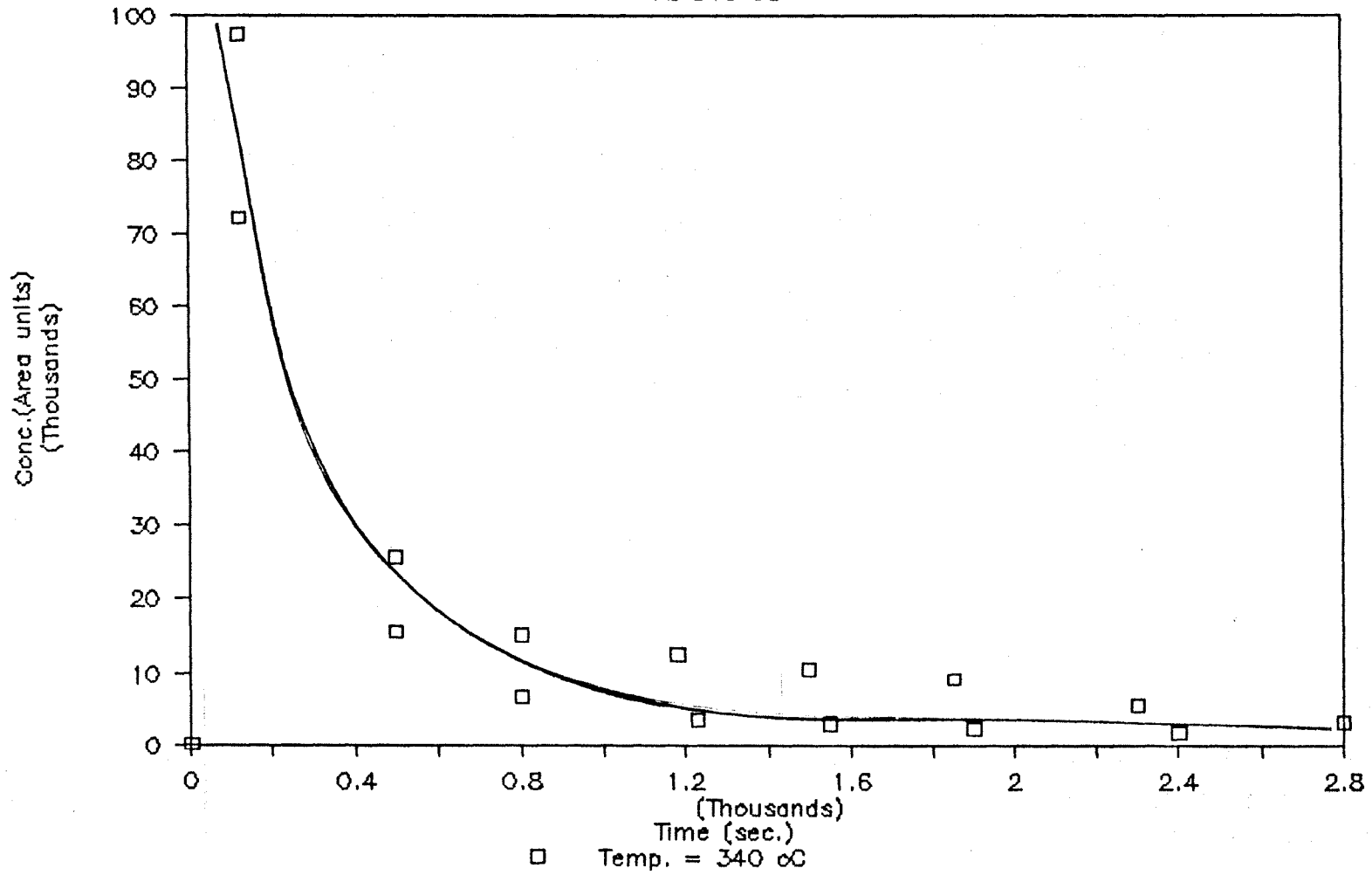


Figure 17. Graph of Thermal Desorption of Anthracene at 340 °C

Figure 18. Thermal Desorption of Anthracene at 340 oC Computer Output

```

Regression coefficients ***** ANALYSIS OF VARIANCE *****
b0= 42465.38
b1= -1.196271E-03
*
* SOURCE OF VARIATION SUMS OF SQUARES DEGREES OF FREEDOM MEAN SQUARE
*
* regression 14.0372 1 14.0372
* error 6.684126 13 .5141636
* total 20.72133 14
*
* f test= (MSR/MSE)= 27.30104
* coefficient of determination r^2= .6774277
* adjusted r^2= .6526145
* coefficient of correlation r= -.8230599
* number of data points= 15
*****

```

EQUATION: $y = 42465.38 e^{-1.196271E-03 x}$

y = Concentration of Anthracene in Area Units
x = Time in Seconds

Conc. of Anthracene vs Time

At 360 °C

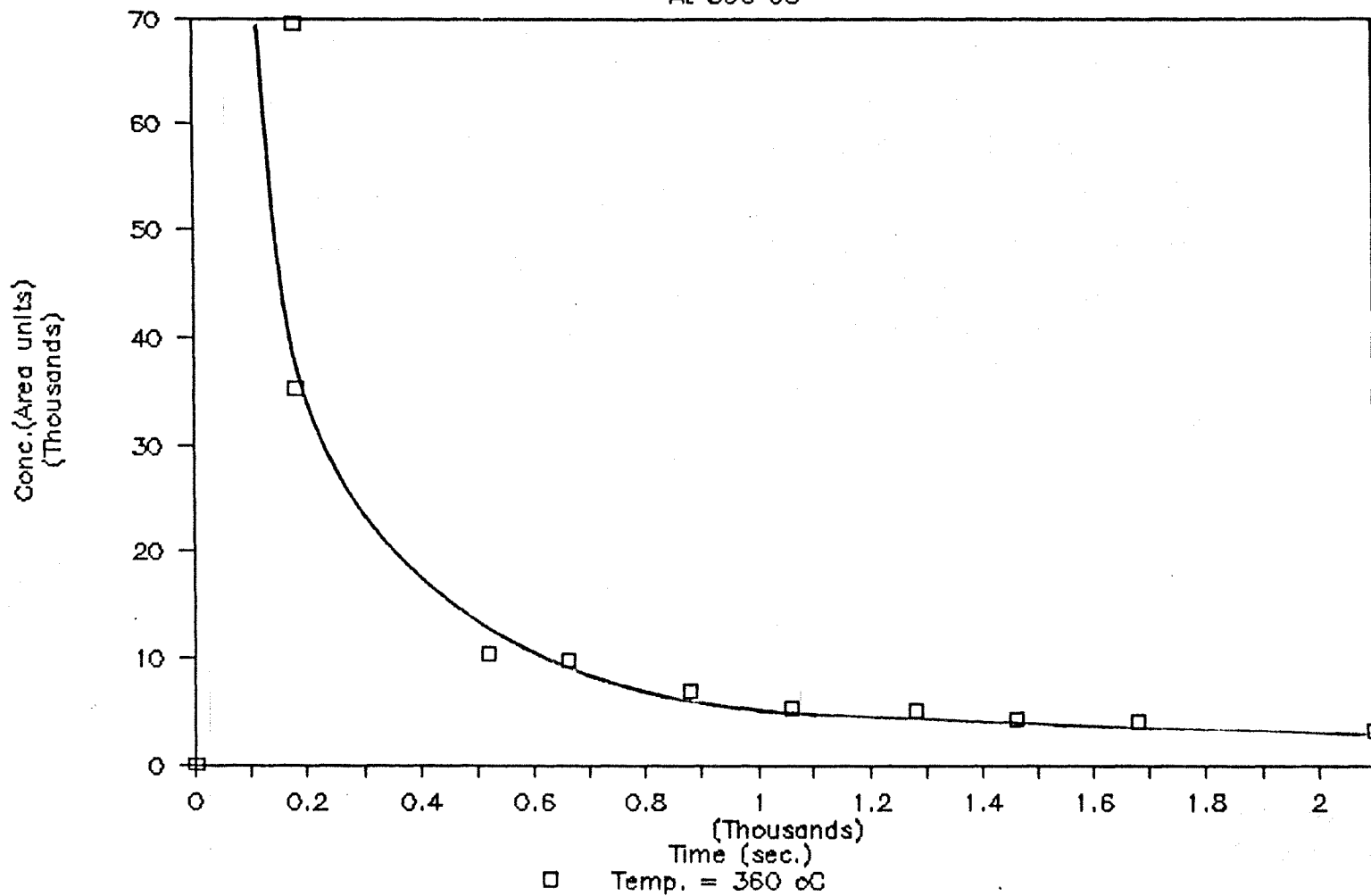


Figure 19. Graph of Thermal Desorption of Anthracene at 360 °C

Figure 20. Thermal Desorption of Anthracene at 360 oC Computer Output

```

Regression coefficients ***** ANALYSIS OF VARIANCE *****
b0= 34835.1
b1= -1.389865E-03
* SOURCE OF VARIATION SUMS OF SQUARES DEGREES OF FREEDOM MEAN SQUARE
* regression 7.091754 1 7.091754
* error 1.981616 8 .2477019
* total 9.07337 9
*
* f test= (MSR/MSE)= 28.63019
* coefficient of determination r^2= .781601
* adjusted r^2= .7543011
* coefficient of correlation r= -.884082
* number of data points= 10
*****

```

EQUATION: $y = 34835.1 e^{-1.389865E-03 x}$
y = Concentration of Anthracene in Area Units
x = Time in Seconds

Conc. of Anthracene vs Time

At 380 °C

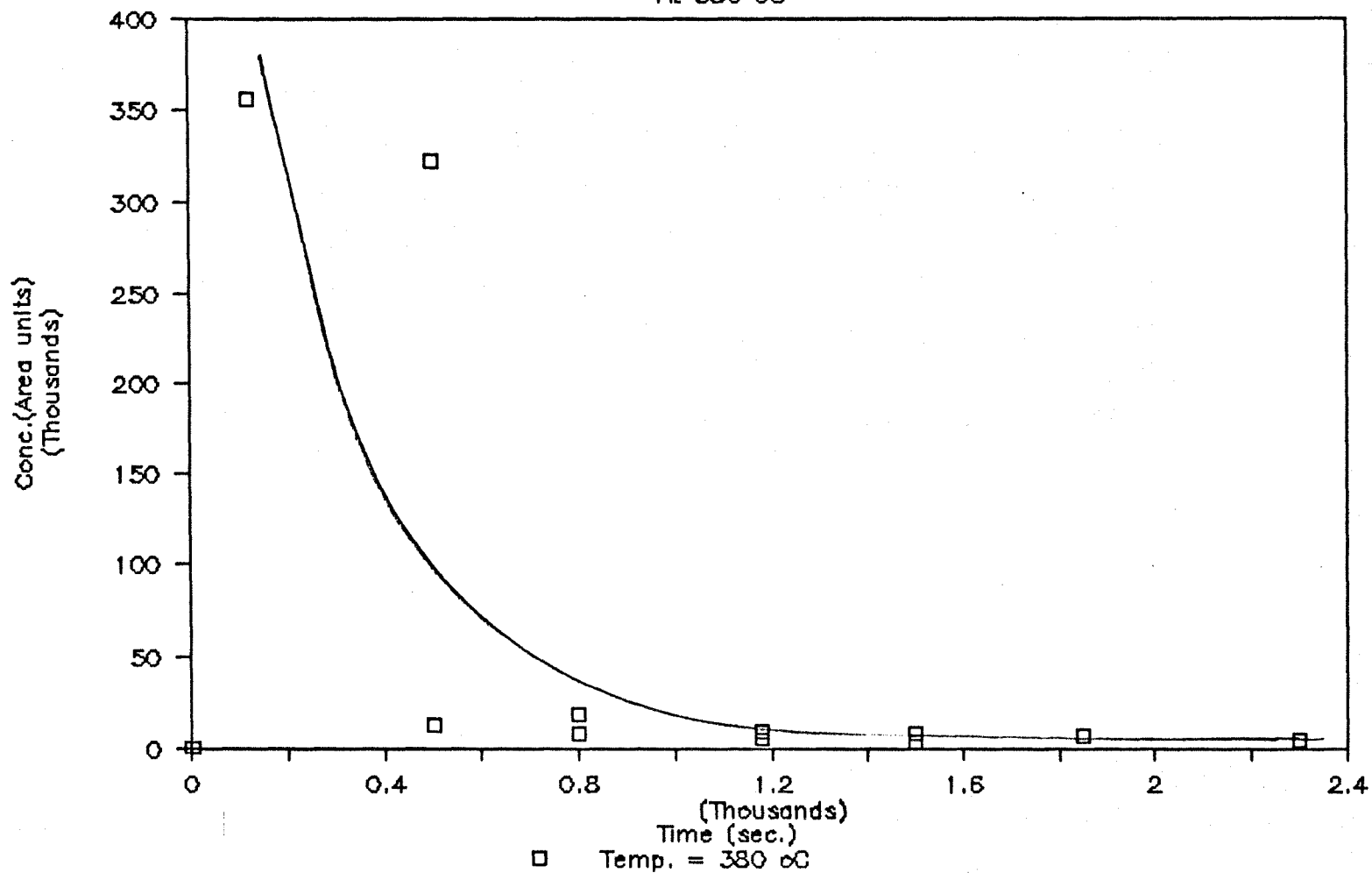


Figure 21. Graph of Thermal Desorption at 380 °C

Figure 22. Thermal Desorption of Anthracene at 380 oC Computer Output

```

Regression coefficients ***** ANALYSIS OF VARIANCE *****
b0= 116251.4 *
b1= -1.861306E-03 *
* SOURCE OF SUMS OF DEGREES OF MEAN *
* VARIATION SQUARES FREEDOM SQUARE *
* * * * *
* regression 14.53049 1 14.53049 *
* error 12.01025 9 1.334472 *
* total 26.54074 10 *
* * * * *
* f test= (MSR/MSE)= 10.88857 *
* coefficient of determination r^2= .5474789 *
* adjusted r^2= .4971988 *
* coefficient of correlation r= -.7399181 *
* number of data points= 11 *
*****

```

EQUATION: $y = 116251.4 e^{-1.861306E-03 x}$
y = Concentration of Anthracene in Area Units
x = Time in Seconds

Graph of k vs 1/T

Anthracene

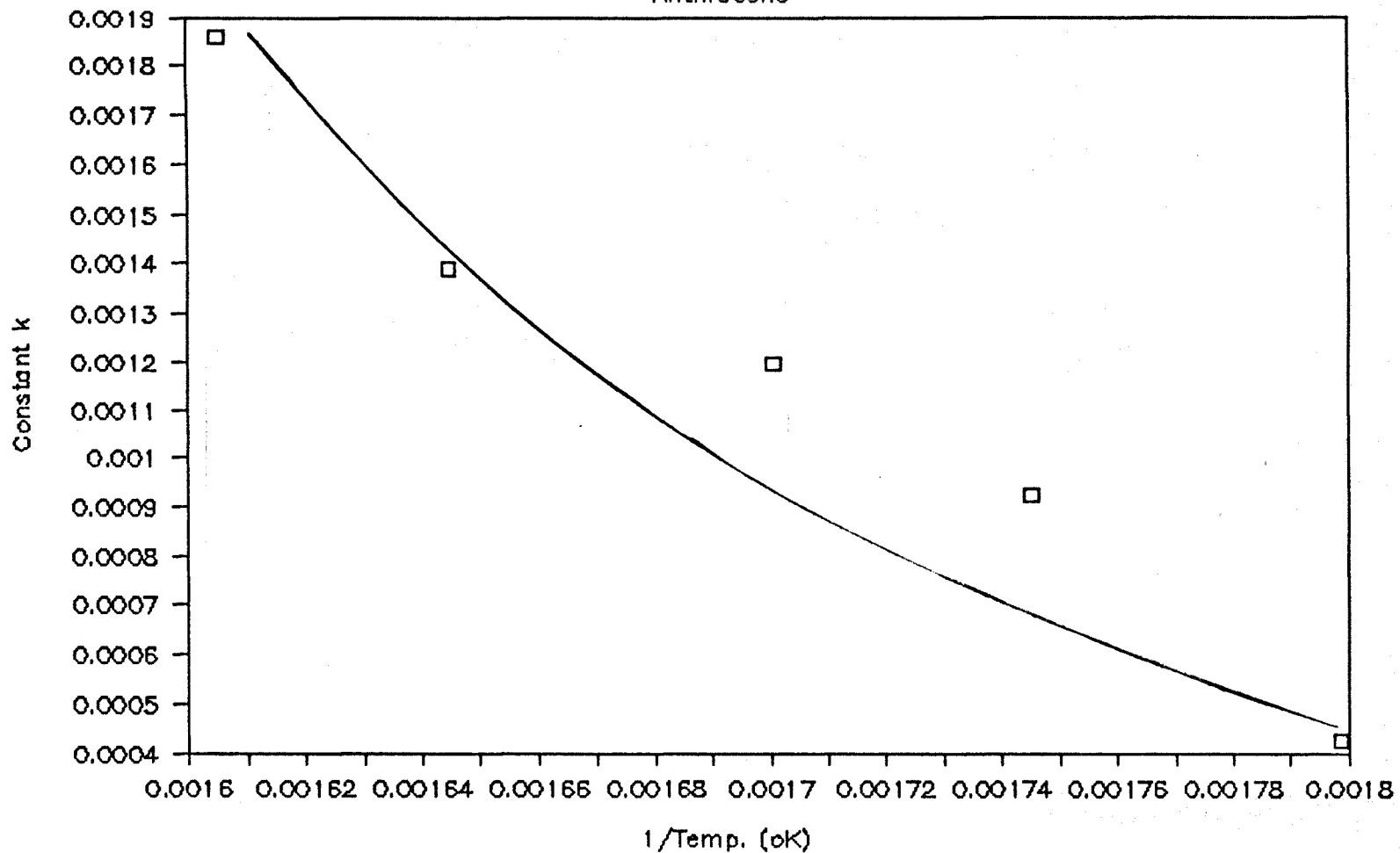


Figure 23. Graph of Constant k Versus Inverse Temperature

APPENDIX 9: PREVIOUS RESEARCH DATA (1) (2)

Figure 24. Regression Coefficients of the Proposed Mathematical Model

```

Regression coefficients ***** ANALYSIS OF VARIANCE *****
b0= 131.2872 *
bl= -6914.038 *
* SOURCE OF SUMS OF DEGREES OF MEAN *
* VARIATION SQUARES FREEDOM SQUARE *
* * * * *
* regression 1.137953 1 1.137953 *
* error .1169688 3 3.898959E-02 *
* total 1.254922 4 *
* * * * *
* f test= (MSR/MSE)= 29.18607 *
* coefficient of determination r^2= .906792 *
* adjusted r^2= .8757227 *
* coefficient of correlation r= -.9522563 *
* number of data points= 5 *
*****

```

EQUATION: $y = 131.2872 e^{-6914.038 x}$
 $y = k$, Constant of anthracene Desorption
 $x =$ Inverse Temperature, in $1/oK$

TABLE XX. ELAPSED TIME AND UNIT AREAS
FOR TRI-CHLOROBENZENE DESORPTION(1)

Elapsed Time (minutes)	Areas 10^{-3}	Elapsed Time (minutes)	Areas 10^{-3}
Temperature 140 °C		Temperature 180 °C	
3	284	3	986
38	141	13	842
54	126	32	421
68	81	52	60
84	49	66	30
98	44	74	18
154	23	--	--
Temperature 160 °C		Temperature 200 °C	
3	276	1	1800
25	333	15	514
47	51	39	27
78	22	59	12
92	13	69	8
112	7	--	--
140	5	--	--
158	5	--	--

TABLE XXI. ELAPSED TIME AND UNIT AREAS
FOR HEXACHLOROBENZENE DESORPTION(1)

Elapsed Time (minutes)	Areas 10^{-3}	Elapsed Time (minutes)	Areas 10^{-3}
Temperature 140 °C		Temperature 180 °C	
5	100	5	97
23	79	18	32
59	44	39	2
307	1.2	--	--
Temperature 200 °C			
1	125		
57	49		
70	32		
90	17		
102	8		

TABLE XXII. ELAPSED TIME AND UNIT AREAS
FOR 1-CHLORONAPHTHALENE DESORPTION (2)

Elapsed Time (minutes)	Areas 10^{-3}	Elapsed Time (minutes)	Areas 10^{-3}
Temperature 180 °C		Temperature 220 °C	
13	580	1	1513
25	539	7	941
50	462	16	458
70	409	22	298
361	69	38	115
377	60	53	33
--	--	70	27
Temperature 220 °C			
1	714		
8	647		
15	452		
22	291		
29	245		
37	196		
46	165		
57	95		
68	88		
80	59		
126	31		

TABLE XXIII. SUMMARY OF PARAMETERS FOR MATHEMATICAL MODELING OF 1,2,4-TRICHLOROBENZENE DESORPTION

CENTER TEMP. (°C)	AVG. TEMP. (°C)	CENTER TEMP. (°K)	Cao (Area units x 1000)	k 10 ² 1/sec	(Correlation factor), r
140	--	413	384	1.73	0.981
160	---	433	500	2.98	0.958
180	---	453	1520	6.03	0.984
200	---	473	2060	8.23	0.979

TABLE XXIV. SUMMARY OF PARAMETERS FOR MATHEMATICAL MODELING OF HEXACHLOROBENZENE DESORPTION

CENTER TEMP. (°C)	AVG. TEMP. (°C)	CENTER TEMP. (°K)	Cao (Area units x 1000)	k 10 ² 1/sec	(Correlation factor), r
260	--	533	31.4	1.47	0.999
280	---	553	59.1	2.56	0.964
300	---	573	264.	11.56	0.994

TABLE XXV. SUMMARY OF PARAMETERS FOR MATHEMATICAL MODELING OF 1-CHLORONAPHTHALENE DESORPTION

CENTER TEMP. (°C)	AVG. TEMP. (°C)	CENTER TEMP. (°K)	Cao (Area units x 1000)	k 10 ² 1/sec	(Correlation factor), r
180	--	453	36.5	.62	0.999
200	---	473	151.	2.65	0.975
220	---	493	334.	6.13	0.985

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