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Organics in ambient air by collection on tenax, thermal desorption,and capillary chromatography with parallel FID/ECD analysis

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Abstract

Title of thesis : Organics in Ambient Air By Collection On Tenax, Thermal Desorption, and Capillary Chromatography with Parallel FID/ECD Analyses

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Master of Science in Environmental
Engineering, 1989.

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Volatile organics in ambient air were regularly collected on Tenax cartridges at high (10 ml/min) and low (5 ml/min) intake flows. These Tenax samples were analyzed by using a Tekmar Automatic Thermal Desorber and capillary GC system with parallel flame ionization (FID) and electron capture detectors (ECD). A series of quality assurance procedures has been established to improve and assure the accuracy and precision of sample collecton and analysis. These procedures relate to performance of the Tekmar thermal desorber, recovery efficiency from the Tenax, and include studies on breakthrough and co-elution. The results demonstrate (1) that from ambient air data in East Central

New Jersey, chlorocarbons, including chloroform, 1,1,1-trichloroethane, carbon tetrachloride, trichloroethylene and perchloroethylene, are present at lower levels than the hydrocarbons: hexane, benzene, toluene and p,m-xylene. Among the chlorocarbons, chloroform and trichloroethylene are present at very low trace levels (within 0.5 ppb) at sampling sites if compared to the other compounds; (2) the ECD is a preferred detector to accurately quantitate the chloro compounds, while hydrocarbons are bset quantitated by FID; (3) The accuracy and precision of Tenax sampling for most of the target compounds warrants its use as judged from studies of breakthrough as well as agreement between high and low flow samples; and (4) Tenax adsorbent is unable to efficiently trap light chlorocarbons such as methylchloride and dichloromethane. A statistical analysis of air data is presented based upon the above studies.

ORGANICS IN AMBIENT AIR BY COLLECTION ON TENAX, THERMAL DESORPTION, AND CAPILLARY CHROMATOGRAPHY WITH PARALLEL FID/ECD ANALYSES

BY

CHIH-HSIANG LIAO

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 Master of Science in Environmental Science
1989

APPROVAL SHEET

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My wife, Michelle, and my son, Steven, have cheerfully waited for this "homework" to be finished. I am deeply gratefully to them for their patience.

Finally, I am indebted to my parents in Taiwan, and I would like to present this paper to them.

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I. Introduction

A. Staten Island/New Jersey Urban Air Toxics Assessment Project (SI/NJ UATAP)

The Urban Air Toxics Assessment Project for Northeastern New Jersey and Staten Island is a three year project encompassing indoor as well as outdoor air sampling and analysis. The overall objective is to characterize the air quality in the target region for selected toxic air contaminants. Ultimately, data collected during the study period will be used to develop human exposure assessments. The groups involved represent both academic communities and governmental agencies: University of Medicine and Dentistry of NJ (UMDNJ) - Robert Wood Johnson Medical School; New Jersey Institute of Technology (NJIT); College of Staten Island (CSI); NY Department of Environmental Conservation (NYDEC); NJ Department of Environmental Protection (NJDEP); NY Department of Health (NYDOH); and US Environmental Protection Agency (EPA). Four ambient pollutant monitoring sites were selected in Northeastern New Jesey, two sites in Union County and two in Middlesex County. NJIT has serviced the two sites at Elizabeth and Carteret for two years with sampling once every 6 days. Selected ambient volatile organic compounds (VOCs) include:

MeCl --- methylchloride; DCM --- dichloromethane;
C6 --- hexane; CFor --- Chloroform;
1,1,1--- 1,1,1-trichloroethane;

Bz --- benzene; CCl4 ---carbon tetrachloride;
Tric --- trichloroethylene; Tol---toluene;
Perc --- perchloroethylene; pmX--- p,m-Xylene; and
oX --- o-xylene.

They are on the EPA list of priority pollutants. Seven of the above target compounds are chloro compounds (C_{1-2}). Quarterly and annual reports of experiments data are submitted to both NJDEP and EPA for comparison with the other analytical laboratories involved in this project. To assure data quality produced by individual laboratory or agent, an intensive sampling campaign called a "shoot-out"- a project for interlaboratory comparison study-was held once a year with sample collection in Staten Island, New York. Before the "shoot-out", separate media for sample collection were sent to EPA for spiking with standards. Subsequent analytical results were reported to EPA with separate quality assurance analyses on Tenax samples also.

B. Tenax Sampling of Ambient Air

Volatile organic compounds in ambient air or water are present in low concentrations (ppm-ppb range). Thus preconcentration of VOCs is necessary for quantitative or qualitative gas chromatographic analysis. This preconcentration can be achieved by either loading a large-volume air sample collected in a canister into a cryogenic trap or drawing a required air volume through a cartridge containing a sorbent medium, which retains the organic compounds while allowing the bulk constituent (air, H_2O, CO_2...) to pass through, then desorbing the sample for analysis.

In selecting a solid sorbent for sampling, we have considered the influence of temperature and humidity on the collection efficiency. The sorbent's affinity for water vapor in ambient air can be a limiting factor especially when a sample is to be analyzed by capillary gas chromatography. One liter of air at 50% relative humidity and 25^{o} C will contain approximately 10 mg of water that appears as ice in the cryogenic focus area of the capillary trap. This ice can present a large problem because of the possibility of altering or plugging the carrier flow. Carrier flow variation or temporary stoppage will cause variability in retention times and may cause incomplete cryotrap desorption [1].

The ability of Tenax to collect and retain hazardous vapors from ambient air is nearly unaffected by humidity conditions commonly encountered in field sampling [2,3]. In addition to the sorption power of Tenax,(adsorbing compounds up to C_{16} (B.P 290^{o} C) [4]), it can easily be regenerated for repeated re-use [3]. A disadvantage, however, is the relatively small breakthrough volumes for light hydrocarbons and low molecular weight polar compounds that limit sample volumes for these species [5].

In the SI/NJ UATA project, we have used Tenax for sampling. The parameters for Tenax cartridge and sampling conditions are listed below :

stainless steel tube	0.64 cm (o.d) x 18 cm
weight of adsorbent	350 mg
adsorbent particle size	60/80 mesh
adsorbent bed	0.60 cm (i.d) x 7 cm
bulk density in tube	0.18 g/cm^3
sampling flow rate	
low flow tube	5 ml/min or 7 liter/day
high flow tube	10 ml/min or 14 liter/day
sampling flow rate/cross area	
low flow tube	17.7 cm/min
high flow tube	35.4 cm/min

C. Analytical Method

After sampling, the concentrated compounds are released by thermal desorption and introduced via a carrier gas system into a cryofocusing capillary trap at the head of the GC capillary column, followed by rapidly heating this trap and injection onto the capillary GC column. The effluent of the column is split into two GC detectors- a flame ionization detector (FID) and an electron capture detector (ECD).

To quantitate the sample, a standard gas mixture containing target compounds was prepared and analyzed in the same manner as the samples. The concentrations of unknown sample are then calculated according to the formula :

$$C_1/C_2 = A_1/A_2$$

where C_1 is standard concentration;

C_2 is sample concentration;

A_1 is peak area of detector response of standard;

A_2 is peak area of detector response of sample.

D. Evaluation and Statistical Analysis of Ambient Air Data

A careful evaluation of ambient air data obtained from Tenax sampling is a difficult and lengthy process containing much uncertainty. This paper will describe several criteria for interpretation of the precision of the entire sampling and analytical system.

In addition to the basic requirement of correct identification of target compounds, specific criteria are necessary for judging data quality statistically. These include both laboratory and field studies such as :

(1) performance of desorber/analytical system;

(2) recovery efficiency and reproducibility of spiked Tenax;

(3) bias of entire system;

(4) duplicate sampling; and

(5) agreement between high and low flow tubes.

II. Instrumentation

A. Description of the Analytical System

As shown in Figure 1, the analytical system incorporates a Tekmar Model 5000 Automatic Desorber and a Varian Model 3700 gas chromatography equipped with an IBM PC based data acquisition system (Interactive Microware, Inc.). The Tekmar 5000 automatic on-line desorber is a microprocessor- based instrument designed for the thermal desorption and gas chromatographic injection of volatile orgranic compounds from adsorbent traps for use with capillary G.C. The interface provides direct on-column injection,by cryogenically trapping the desorbed sample into a narrow band on an uncoated inert fused silica precolumn (0.32 mm i.d). This then flash-heated for injection into the G.C. Both desorber and GC were interfaced so that the entire analysis was automated. All transfer components (tubing, valves) were heated to minimize sample loss by physical adsorption.

A 50 m x 0.21 mm i.d. cross-linked methyl fused silica column (Hewlett Packard) with 0.5 micron film thickness of OV 101 was used to resolve the target compounds. The column flow was maintained at a volume flow of 1 ml/min by using a flow controller. Zero-grade helium served as the carrier gas, and zero-grade nitrogen (28 ml/min)was used as the make-up gas to provide electron capture operation. Optimum analytical results were achieved by temperature programming the GC oven from 35 to 195^{o} C at 6^{o} C/min. At the end of the column, the carrier gas flow is split to the FID and ECD, with 90% of the effluent going to the FID.

Besides, two six-port valves (shown in Figure 1) with 2 ml sampling loops were also used in order to have the flexibility to :

(1) run standard gas either by the flow routing of sample analysis or by direct injection onto the GC column; and

(2) spike the standard gas mixture onto Tenax for diverse experiments.

B. Performance of Tekmar Automatic Desorber

The Tekmar Model 5000 Automatic Desorber [6] provides both fully automatic desorption of the Tenax cartridge and fully automated interface to the G.C. Once a Tenax trap has been inserted into the furnace, it is first swept with helium before it is heated. This prepurge mode serves dual functions. It allows any water present to be purged out of the tube before the internal trap is cooled; this is particularly helpful for taking samples under high humidity or rainy conditions. The second advantage of this mode is that it removes oxygen from the tube before heating, thus increasing the life time of the reuseable

Tenax material and reducing the formation of artifacts. After prepurge the trap is heated and swept with helium into a cryogenic trap cooled by liquid nitrogen. After the desorption step is complete, an eight-port switching valve is rotated to cause the carrier gas to backflush the cold trap, which is heated rapidly to a preset temperature, and the sample is then transfered to the second cryogenic trap before the sample is injected into the G.C. After sample injection, the Tenax trap can be baked to regenerate it. All the sampling traps were analyzed under the following desorption conditions :

Prepurge 1 : 3 min at 10 ml/min; Desorb : 12 min at 210^{o} C into a cold trap (Cryo-1) at -150^{o} C; Transfer: 12 min at 1 ml/min and 210^{o} C of transfer line into a second cryofocus (Cryo-2) at -150^{o} C; Inject : 2 min at 210^{o} C; and Bake: 15 min at 225^{o} C.

To ensure the reliability of the Tekmar desorber, an experiment was made to compare a standard gas mixture run through the desorber and GC (Cryo-1 and 2) versus the same standard directly injected into the GC (Cryo-2 only), which are termed Method 1 and Method 3, respectively, and were automatically controlled by desorber, upon their selection.

Table 1 presents results for a standard gas mixture analyzed on different dates using Method 1. For any given compound, the reproducibility is acceptable, since the coefficient of variation (CV) ranges from 2.4 to 10.38 %.

Similarly, Table 2 shows CV ranging from 1.37 to 6.96 % by Method 3. Comparing the CV value of a given compound, as one might expect due to the comparative simplicities, the dispersion is lower by Method 3 than by Method 1 with the exception of C6 and CFor, which are eluted at nearly the same retention time.

A criterion for judging Tekmar desorber performance under the mentioned desorption conditions is to investigate the difference between both methods(1 and 3 only). The difference was computed and shown in Table 3, which indicates that the percent difference between Method 1 and 3 ranges from -6.44 to 1.71. It thus implies that complete recovery can be obtained under the preset conditions of desorber; for any of the target compounds, the -150^{o} C temperature of Cryo-1 and 2 is low enough to trap all target compounds at a desorption flow rate of 10 ml/min in Cryo-1 or transfer flow rate of 1 ml/min in Cryo-2. Another point is that the temperature of the transfer line and the transfer time, which are 210^{o} C and 12 min, can be also accepted. However, we have investigated various transfer line temperatures to check the recovery efficiencies of target compounds. Table 4 presents the result of the transfer line temperature versus weight detected, and Figure 2 and 3 show the graphs plotted from Table 4 for comparison of low and high boiling points. As we can see, the quantities of pmX and oX decrease a little at 90^{o} C but significantly decrease at 60^{o} C, while all the other compounds are not affected by temperatures as low as 60^{o} C. This shows us a very important concept that the water-plugging problem in the capillary column could be solved by controlling the transfer line temperature at an optimum value, so as to retain the water vapor and water- soluble polar compounds while allowing the target compounds to be transferred to Cryo-2. Using modification on the Tekmar desorber, we can also analyze the VOCs collected by the canister method or by other solid sorbents which tend to adsorb water vapor under such analytical system.

Another experiment to test the performance of the entire analytical system including the desorber and capillary GC was made by spiking standards into 8 clean Tenax traps and then desorbing the traps. The recovery values of 8 replicates were computed in Table 5. It shows that complete recovery of target compounds can be obtained with only one exception MeCl, which will be explained later on.

Before high performance was achieved in the analytical system, we had found that a hot spot (around 80 ^{o} C) at the far end of desorber furnace existed with temperature high enough to liberate the sample adsorbed on the most concentrated end of the Tenax tube. Thus we would lose the sample during three-minute prepurge mode, even though the displayed furnace temperature was 40^{o} C. Due to this, we decrease the quantity of Tenax adsorbent, 500 mg to 350 mg, allowing a shorter Tenax bed (7 cm long) in the sampling tube with the bottom region (10 cm long) of the furnace empty to avoid effects of the hot spot.

C. Comparison of FID and ECD GC Detector

The FID [7] is highly sensitive to most organic compounds with the exception of certain functional groups such as carbonyl, hydroxyl, halogen, and amino. These can sometimes yield fewer ions or none at all. On the other hand, the ECD [7] is selective in its response, being highly sensitive toward molecules containing electron-negative functional groups such as halogens, peroxides, quinones, and nitro groups. ECD is insensitive toward molecular classes including amines, alcohols, and hydrocarbons. It will be shown in this work that knowing the response intensity relationships for chloro compounds between FID and ECD is of significant benefit since 7 of 12 target compounds are chloro compounds. We will show that these chloro compounds can be easily interfered with (co-elution) by other hydrocarbons. This co-elution or interference is amplified further because the FID response to some of the chlorinated compounds is very small. Inaccurate quantitation may result if these are determined only by FID response. Due to this potential problem we show that one should consider the quantitation of chlorocarbons by ECD, instead of by FID, if good reproducibility and linear response can be achieved.

Different amounts of standard were injected into the desorber and GC using six-port valve with 2 ml sampling loop, by changing the pressure in the sampling loop (eg 1 atm, 28", 20", 10", 5", 2"). The sampling loop was set at temperature of 165 ^{o} C. Table 6 shows the reproducibility comparison of the two GC detectors. The coefficients of variation (CV) of FID and ECD response indicate the dispersion of a group of replicate data. As for FID, all the CV values are within 10%, while they show larger variations with ECD (within 17%). However, the precision of quantitation can be better by ECD than by FID when one considers the described co-elution problem of chloro compounds with atmospheric HC's. From this experiment, sets of data on FID and ECD responses with several different amounts of standard gas were obtained. These data are shown in Table 7. The gas law (PV=nRT) shows that P is proportional to (n/V)=C (concentration) when T is constant. If linear response of both detectors is assumed, then the following expression is obtained:

$$\frac{P_1}{P_2} = \frac{C_1}{C_2} = \frac{A_1}{A_2}$$

where P_i is pressure

C_i is concentration of compound

A_i is peak area of compound.

Using the experimental data in Table 7, a number of regression lines of response area vs pressure are shown in Figure 4-1 through 4-12. It is apparent that high linear correlation is achieved for each of target compounds. Table 8 shows the correlation coefficients of R squared between FID/ECD and pressure for each of the twelve compounds and also the weight range being detected. In general, a high linear response exists for all target compounds with both FID and ECD. The correlation coefficients of chloro compounds from FID and ECD indicate that higher correlation is obtained for the ECD. Hence, to eliminate the interference from co-eluting peaks, we recommend quantitation of chlorinated compounds directly by ECD since this method has the higher linear correlation.

III. Characteristics of Tenax Adsorbent and Adsorbate

A. Basic Parameters

Tenax is a relatively polar porous ploymer based on 2,6-diphenyl-p-phenylene oxides. It was developed by AKZO Research laboratories and introduced as a GC column material by Van Wijk in 1969-70 [8]. Typical characteristics of Tenax TA are :

specific surface area	35 m^2/g;
pore volume	2.4 cm^3/g;
average pore radius	200 nm; and
density	0.16 g/cm^3.

With a prepared standard gas mixture containing about 5 ppm of each of the target compounds, the following series of experiments can be performed:

(1) calibrating the concentration of air sample;
(2) spiking Tenax trap for adsorption/desorption study; and
(3) spiking Tenax trap for recovery study.

Table 9 shows relevent information for the standard gas mixture and the properties of the target compounds, which are listed in the order of elution in chromatogram.

B. Adsorption/Desorption of Adsorbate

This experiment was mainly designed to understand the adsorption/desorption efficiency for all adsorbates prior to field sampling. Using the analytical system shown in Figure 1, we can connect the blank Tenax trap between the six-port valve and Tekmar furnace. The breakthrough compounds in the effluent were directed to the Tekmar desorber and detected by GC when loading 2 ml standard gas mixture into trap under the conditions of 10 ml/min carrier flow, temperature 170^{o} C and 1 atm. After these breakthrough compounds are measured, the spiked Tenax trap is desorbed into the GC. By noting the results presented in Table 10, it is clear that most of the MeCl breaks through the Tenax trap under the above loading conditions with a total volume of carrier gas of 120 ml. Relating the conditions to field sampling, it seems that the Tenax trap is unable to retain MeCl efficiently. As for the other compounds, the average adsorption/desorption efficiencies ranged between 95 and 124.5 %.

C. Retention Tendency of Adsorbate

The real mechanism of the adsorption process on a solid sorbent is very complicated in field air sampling. It is generally determined by such synergistic parameters as the concentration of the investigated compounds, temperature, co-adsorption, sorbent particle size, gas flow-rate, gas humidity, properties of the adsorbent and adsorbate, and the number of adsorption-desorption processes[9]. To grasp some idea about retention tendency of adsorbate under the current sampling conditions, we designed an experiment by first spiking a known amount of standard Tenax trap blank at a carrier flow 10 ml/min for time periods of 12 minutes. We then purged the traps with zero grade nitrogen (same flow direction as air sample collection) at approximate purge flow rates of 5 and 10 ml/min, respectively, for 24 hours. This is similar to routine sampling conditions.

The results are shown in Table 11. As we can expect, MeCl instead of being purged out will break through immediately at the very beginning when spiking the blank traps. We also see that DCM can be completely purged out at either low or high flow rate. CFor is purged only at high purge flow rates. With a high purge flow rate of 9.8 ml/min, approximately 60 % of C6, 1,1,1, and CCl4 were purged out. However, by purging at low flow rate of 4.8 ml/min, only 54 % of CFor was purged out.

Although this experiment does not reflect the real state of routine field sampling, which actually varies the inlet concentration with time, it demonstrates that Tenax seems unable to effectively adsorb light chloro compounds, especially MeCl and DCM. If necessary, another active sorbent can be used in series with Tenax for collection of low molecular weight compounds which break through on Tenax.

IV. Two Studies of Ambient Air Analysis

A. Co-elution Study of Chloro Compounds

There is a problem of co-elution in the chromatographic separation of several chloro compounds when analyzing routine Tenax air samples collected from both Elizabeth and Carteret, New Jersey. First, the peaks of interest in the chromatogram of a spiked air sample are identified and then chromatograms of the air sample are compared with the spiked air sample. Then the ratio of FID vs ECD response of spiked sample is compared to that of a Standard Gas Mixture to demonstrate that the use of ECD response solves the problem of co-elution in quantitating these chlorocarbons. The experimental procedures are described as follows:

(1) Prepare two air samples collected in Tenax traps from a field site.

(2) Spike one of the above samples with 2 ml Standard Gas Mixture under the conditions of 1 atm and temperature 165° C. Figure 1 shows the apparatus used to spike Standard gas into Tenax trap by using a six-port valve and a 2 ml sampling loop.

(3) Analyze each of samples listed below in Tekmar Desorber/GC :

a. 2 ml Standard gas at 1 atm and 165° C;

b. spiked air sample; and

c. plain air sample.

The results are discussed in the four points below : Figure 5 plots the relationship between temperature program and retention time for each standard in the gas mixture. The temperature program sets initial temperature at 35° C for 12 min, then, increases temperature to a final temperature of 195° C at the rate of 6° C/min. The final temperature is maintained for 5 min. The last target compound, o-Xylene, is eluted at 126° C and retention time 27.1 min.

Paired chromatograms of FID and ECD response on the standard gas, spiked air sample and air sample are shown in Figure 6-1 through 6-4, 7-1 through 7-4 and 8-1 through 8-4, respectively.

(1) Analyzing the chromatogram of spiked air sample: first, chloroform elutes on the shoulder of a hydrocarbon peak next to it. Quantitation is difficult unless high concentration is present. No separation could be expected, therefore, if its concentration is much lower than spike, as is usually found in ambient air. Secondly, trichloroethylene comes out between two hydrocarbon peaks which are so close to it that they may result in interference. Third, perchloroethylene is poorly separated in a two peak set. Again, the same result as for chloroform may be expected. Fourth, 1,1,1-trichloroethane and carbon tetrachloride seem to show good separation.

(2) In the chromatogram of the air samples, no shoulder exists for chloroform and only one peak stands at the retention time of perchloroethylene, indicating significant hydrocarbon interference and lack of good quantitation for this chlorocarbon.

(3) If one assumes that no co-elution occurs, when 5 peaks of chloro compounds (4, 5, 7, 8, 10) are picked by simply matching with the standard chromatogram, their ratios of FID vs ECD response should approximate those of standard gas. Table 12 lists the comparison of ratios. If the ratio for the spiked sample is much greater than that of the standard, it demonstrates that co-elution is occurring. Peak 5 has the least co-elution, peaks 7 and 8 have about 54 and 44% interference, respectively, and peaks 2,4 and 10 have even more interference. As for DCM, it is difficult to quantitate due to described co-elution as well as its relatively insensitive ECD response compared to the other chloro compounds.

(4) This study indicates that the ECD is a preferred and more accurate method to quantitate the chloro compounds, since its good linear response in the appropriate concentration range has been mentioned earlier.

B. Breakthrough Study of Target Compounds

Concerning the sampling system, the flow rates of current air sampling are set approximately at 5 and 10 ml/min in each sampler. The air volumes of low and high flow rate total up to 7 and 14 liters, respectively. The sampling traps, as mentioned, are packed with 7 cm long bed of 350 mg Tenax-TA. To understand the breakthrough problem under field conditions, both front and backup traps were put in series when sampling at both Elizabeth and Carteret on Sep. 30, 1988. The results are described as below:

(1) Due to the interference of hydrocarbons with chloro compound, the concentrations of the following chloro-compounds, chloroform, 1,1,1-trichloroethane, carbon tetrachloride, trichloroethylene and perchloroethylene, are obtained by ECD peak response, where there is no response from HC's. The concentrations of all other compounds are directly obtained by FID peak response. The methyl chloride and dichloromethane do not have good sensitivity on the ECD detector and the peaks from FID detector may have background interference from hydrocarbons.

(2) Table 13 and 14 show that the air concentrations of front and backup tubes at high and low flow rates sampled in Carteret and Elizabeth on September 1988. Table 15 and 16 shows the average concentration percentage being trapped by both front and backup traps in each site. As shown in Table 15 and 16, breakthrough percentage of each compound is within 10% except for the light compounds, Methyl chloride and Dichloromethane. As for the term of relative average error(R.A.E), it explains the agreement between

high and low flow rates. Again MeCl and DCM show larger values of R.A.E. Poor agreement is due to poor adsorption for these light compounds.

(3) The results indicate that the current sampling conditions are acceptable for the required quantitation of this project.

V. Quality Control and Assurance

A. Reproducibility of Spiked Tenax

In this experiment, we spiked 8 Tenax blank traps with known quantities of standard and then desorbed them into the analytical system. The result of 8 replicates is shown in Table 17. The quantity of spike is approximately equivalent to the ppb level in ambient air. The reproducibility can be noted by the relative dispersion value(i.e., CV), which is indicative of the spread in a set of observations. As shown in Table 17, the coefficients of variation of target compounds are within 10 %, excluding MeCl due to its very low breakthrough volume, described before.

B. Bias experiment audited by EPA

There are two different types of errors that may be present in statistical measurements, namely, systematic errors and random errors. Systematic errors (or bias), as the term implies, caused a measurement to be incorrect in some systematic way. These are errors involved in the procedures of a statistical investigation and may occur after the processes of collection and analysis. Reproducibilty experiments can demonstrate the performance of spiked Tenax and the analytical system. However, it cannot assure that precise quantitation has been achieved because bias can be introduced by the standard gas mixture or other factors.

The Environmental Monitoring Systems Laboratory of EPA loaded four cleaned Tenax traps with known standards, including one background trap. The result of audit is presented in Table 18, which records spiked and reported amount of the compounds, and Table 19 shows the bias percentage. Among those compounds, the bias of CCl4, Tol and Perc range from +30 to +60 %, while that of Bz and Tric range up approximately to +20 %, and the others are within +/- 10 %.

C. Agreement Between High and Low Flow Tenax Samples

The agreement of high vs low flow traps was statistically analyzed by employing the data generated from Carteret (March 4 ~ December 29, 1988) and Elizabeth (June 14 ~ December 29 1988). A useful aid in studying the agreement between high and low is to plot a scatter diagram of paired point data. This allows visual examination of the extent to which the data follow the ideal line (slope=1); it roughly gives an understanding of (1) whether breakthrough or inefficient adsorption occurs ; (2) how much agreement is achieved; and (3) abnormal discrepancies in the data. Figure 9-1 through 9-22 present scatter diagrams for a given compound and site. From visual examination, the paired points for most of the diagrams are below the ideal line, it indicates that larger values of air concentrations result from the low flow tube. To further understand the strength of the linear relationship and the difference between the two flows, that is, how well the two flows are correlated and how well the regression slopes approach the ideal line, we calculated the coefficients of determination (R squared) and the slope by linear regression analyses, as summarized in Table 20. To assure data quality, both large values of correlation coefficients and slopes near unity are preferred. As a general principle, if an absolute value of R is larger than 0.7 (if R squared is larger than 0.49), there is significant correlation. As shown in Table 20, all R squared values are larger than 0.49, which demonstrates that one of the two requirements is statisfied. From the slope column, the values larger than 0.9 are those compounds of C6, Bz, Tric, and Tol in Carteret, and CCl4, Tric in Elizabeth. the others are between 0.70 and 0.90 except for MeCl in both sites and CCl4 in Carteret. Further comparing the slopes, half of the paired slopes are close to each other excluding those of C6, Bz, 1,1,1, CCl4 and Tol.

Because all the slope values except Bz in Carteret are smaller than 1, and air concentration is determined by the two factors of weight/volume of target compound and total sampling air volume, it indicates that either breakthrough or system bias has been involved. However, as discused earlier, the safe sampling flow rate has been assured in the breakthrough study. Should it happen, the reason may be that Tenax has gradually deteriorated from frequent re-use. Therefore, the system bias, excluding the less dominant bias from analytical system as has been described, may be contributed by the factor of total sampling air volume, which subsequently may lead to bias resulting from flow-rate calibration curve of a rotameter calibrated at room temperature.

Another way to study the agreement can be obtained by computing the mean of relative average error (M.R.A.E) and its standard deviation. The lower the mean is, the higher precision and agreement are acheived. Table 21 shows the M.R.A.E of each compound sampled during a long time series mentioned above. As may be seen, it seems that the precisions of hydrocarbons, especially those in Carteret, are better than chloro compounds, which were generated by ECD response. To determine the precision of entire system, the two M.R.A.E columns in Table 19 were averaged, giving the 14.54% +/- 8.49%.

D. Results of Staten Island Interlaboratory Comparison Study

This study -- "Shootout" #2 of SI/NJ UATP -- was held July 25 ~ 29 1988 at Susan Wagner High School, Staten

Island, New York. Duplicate Tenax samplers were employed to collect air samples at low and high flow rates. The precision, in terms of R.A.E between high and low flow by each compound, of air data generated by NJIT was calculated in Table 22. Again the precision of entire system, as noted in the preceding section, was obtained from a six measurements of data in this study and the calculated value is 19.18% +/- 11.29%.

The interlaboratory data evaluation[10], released by SI/NJ UATAP Management/Steering Committee meeting February 8 1989, was digested in Table 23. The results show the % difference and its absolute value from the mean for all compounds/all organizations. The shootout data allowed the following conclusions:

(1) most organizations [CSI, NJSDEC, PEI, EPA/RTP, NJIT Tenax] has results within +/- 40% (NJIT: -15.67 %) of the mean, as measured by difference from the mean;

(2) precision, as measured by absolute value of the differences from the mean, was within 45% (NJIT: 25.33%) for the organizations measured above;

(3) greatest factor responsible for variation of results was the organization performing analysis, not sampling method;

(4) there was a high correlation between the results of the performance evaluation samples (i.e., bias audit by EPA) and the results of the "shootout".

VI. Statistical Analysis of Ambient Air Data

A. Concentrations Trend of Target Compounds

The ambient air data generated was based on a weekly time series of VOC collection and analyses and it is a set of analytical measurements arranged in chronolgical order. In general, the raw weekly data contains trends of cyclical and weekly variations as well as other irregular factors. In this section, our interest is centered on the two elements of the trends or cycles. A practical method [11] has been employed to obtain a series of moving averages that smooth, but still illustrate roughly the trend and cycle components. These are computed using the following procedures: the first moving average figure contains the first four weekly data. Then the first weekly data point is dropped, and the second through fifth weekly figures are averaged. The computation proceeds this way until the last moving average is calculated, containing the last four weekly data of the original series. In the actual calculation an adjustment was made in order to center the moving average figures so their timing corresponds to that of the original data.

The moving averages along with the original raw data points have been plotted in Figure 10-1 through 10-16 by site and given target compound. As may be expected by visual examination, the moving average, which runs through the original data, can be observed to follow the cyclical fluctuations. If the series were long enough, we would be able to see how the moving average describe long term trend movements as well. To further visually compare the relationship between compounds in each site, we combine the curves of several target compounds moving averages and present them in Figure 11-1 through 11-4.

In summary, the concentration levels of the moving averages in this geogrphical area of Carteret and Elizabeth can be divided into four groups. Among them, one includes the highest level target compound of 5 ppb of Tol; the second group, near 2 ppb, are Bz, C6 and pmX; the third group such as 1,1,1 is near 1 ppb; and the group of lowest level, which is near 0.5 ppb, are CCl4, Perc, and oX. The trend of the moving average for most compounds in Carteret site can be roughly described as follows: First, an upward movement occurred in early April, this gradually increased to a maximum point in early June. After early June, there was one-month downward trend with a valley point in early July. Again, two months of upward trend happened through July and August. After maximum point in late August, a stable level was maintained from early October till early November, then moved upward to another peak in early December. It seems that Perc and oX moved steadly along the level of the moving average of 0.5 ppb. For the Elizabeth site characterized from June 14 to December 29 1988, the same trends occurred with a little difference for C6, which had a peak at mid August ahead of the others.

B. Site Relationship of Target Compounds

The comparison of moving average between sites for a given compound was shown in Figure 12-1 through 12-8. In general, there is no significant difference between sites for any given compound, however, several deviations are described. As a generality, there was a obvious distinction between sites in that the Perc level (moving average) in Elizabeth was always larger than that in Carteret. For the other compounds, the higher levels occurred in Elizabeth between June and late August or early September (summer time); then higher level were observed in Carteret between late August or early September and mid/late November(autumn). The higher levels in Elizabeth again appeared between mid/late November and late December. The oscillations seem to be associated with the change of seasonal weather condition (i.e., from hot weather to mild then cold).

VII. Conclusion

1. This paper describs the aspects of Tenax sampling of ambient VOCs - methylchloride, dichlomethane, hexane, chloroform, 1,1,1-trichloroethane, benzene, carbon tetrachloride, trichloroethylene, toluene, perchloroethylene, p,m-xylene and o-xylene. It covers the analytical method - Tekmar Automatic Thermal Desorber and capillary GC system with a FID as well as an ECD, and statistical air data analysis. A whole series of quality assurance procedures have been established and experiments performed to evaluate the precision of sample collection and analysis.

2. By parallel field sampling at 5 and 10 ml/min of intake flow with 350 mg Tenax adsorbent, it is assured that no breakthrough of target compounds except methylchloride will occur.

3. Comparison of a direct sample injection into GC (no Tekmar) with indirect injection into GC(with Tekmar), shows percent differences ranging from -6.44 to 1.71, and indicates that satisfactory performance of the Tekmar desorber has been achieved under the selected conditions of time, temperature and system flow.

4. Due to co-elution of chloro compounds with atmospheric HC's as well as high linear responses of both FID and ECD, it is shown that the ECD is clearly preferred

to more accurately quantitate the chlorocarbons, while hydrocarbons are quantitated by FID.

5. The adsorption/desorption efficiencies of Tenax using spiked samples are shown to be between 95 and 125 % for all target compounds with the exception of methylchloride (17 % only). Reproducibilities of analysis on spiked Tenax, in terms of the dispersion with 8 replicates for all target compounds, are within 12 %, except for methylchloride (29.26 %).

6. It is difficult to quantitate both methylchloride and dichloromethane, due to the former's very low breakthrough volume on Tenax, and the latter's relatively insensitivity on the ECD in addition to co-elution of other HC's in the background.

7. The entire system precision, as measured by the mean of relative average error for all target compounds, averages 15% with a standard deviation of 8%. The precision is within 14% for most compounds except carbon tetrachloride (21%) for the Elizabeth site, while, for Carteret , trichloroethylene was quantitated with the precision of 26%, 1,1,1-trichloroethane, perchloroethylene and carbon tetrachloride approximate to 20% and the other compounds are within 9%. As menthioned above, the analytical method performs at higher precision for hydrocarbons than for chlorocarbons.

8. The measured concentrations of moving average,

during a time period in the area of Carteret and Elizabeth, are divided into four levels : (1) 5 ppb for toluene; (2) 2 ppb for benzene, hexane, and p,m-xylene; (3) 1 ppb for 1,1,1-trichloroethane; and (4) 0.5 ppb for carbon tetrachloride, perchloroethylene and o-xylene. In general, the concentrations of hydrocarbons are higher than chlorocarbons. As for the site relationship for a given compound, there appears to exist oscillations in concentration levels which alternate when the seasonal weather conditions change.

Table 1 Peak Area Of Standard Gas Mixture By Method 1 *
(2 ml, 1 atm, 170°C)

Compound	Date					Avg	C.V %
	2/15/88	2/19/88	2/24/88	3/5/88	3/10/88		
1. MeCL	1203	1216	1202	1266	1252	1228	2.40
2. DCM	2895	2959	3065	3127	3116	3032	3.35
3. C6	13572	14112	14520	14206	13994	14081	2.45
4. CFor	2724	3037	2859	2880	3167	2933	5.84
5. 1,1,1	4802	4950	5623	5243	5439	5211	6.49
6. Bz	17785	18842	19064	19382	19110	18837	3.28
7. CCL4	1947	2271	2523	2454	2516	2342	10.38
8. Tric	6097	6023	6957	6487	6603	6433	5.96
9. Tol	18990	20525	21333	19872	21231	20390	4.81
10. Perc	3993	4489	5216	4246	4568	4502	10.17
11. pmX	11470	11996	12716	11680	13258	12224	6.10
12. oX	17224	17671	18380	16902	19950	18025	6.71

Note : C.V = Coefficient of Variation
* --- Run through desorber and GC (Cryo-1 and 2)

Table 2 Peak Area Of Standard Gas Mixture By Method 3 *
(2 ml, 1 atm, 140°C)

Compound	Date					
	1/30/88	2/3/88	2/9/88	2/15/88	2/21/88	2/27/88	3/4/88
1. MeCL	1295	1299	1324	1317	1280	1289	1276
2. DCM	3237	3173	3226	3301	3180	3214	3129
3. C6	15952	15892	15906	15988	15548	14911	15228
4. CFor	3153	3049	3197	3200	3214	3801	3347
5. 1,1,1	5609	5627	5546	5668	5420	5593	5450
6. Bz	20856	20454	20887	20873	20265	20183	19911
7. CCL4	2708	2607	2703	2594	2801	2688	2652
8. Tric	6867	6730	7021	6952	6728	6746	6597
9. Tol	22634	21711	22578	22598	22704	22446	21838
10. Perc	4793	4580	4822	4920	4761	4781	4647
11. pmX	13964	12860	14025	13959	14368	14026	13340
12. oX	20227	18183	20360	20216	20371	20378	18575

Compound	Date 3/10/88	Avg	C.V %
1. MeCL	1279	1295	1.37
2. DCM	3210	3209	1.58
3. C6	15405	15604	2.55
4. CFor	3340	3288	6.96
5. 1,1,1	5474	5548	1.64
6. Bz	20125	20444	1.88
7. CCL4	2730	2685	2.51
8. Tric	6757	6800	2.02
9. Tol	22412	22365	1.69
10. Perc	4798	4763	2.21
11. pmX	14118	13833	3.53
12. oX	20102	19802	4.49

Note : * --- direct injection into GC
(Cryo-2 only)

Table 3 Percentage Difference Between Method 1 and 3

Compound	Average Method 1	 Method 3 *	difference %
1. MeCL	1228	1207	1.71
2. DCM	3032	2991	1.37
3. C6	14081	14547	-3.21
4. CFor	2933	3065	-4.29
5. 1,1,1	5211	5173	0.75
6. Bz	18837	19060	-1.17
7. CCL4	2342	2504	-6.44
8. Tric	6433	6339	1.48
9. Tol	20390	20851	-2.21
10. Perc	4502	4440	1.40
11. pmX	12224	12896	-5.21
12. oX	18025	18461	-2.36

Note : * --- corrected value by a temp. factor of 413/443

Table 4 Weight Detected On Various Transfer Line Temp., ng

Compound	Transfer line temperature, °C							
	60	80	90	100	120	150	180	210
1. MeCL	8.42	8.66	8.80	8.94	9.04	8.92	8.96	8.75
2. DCM	60.41	56.88	56.00	59.75	58.83	55.58	57.58	56.85
3. C6	35.50	37.49	37.51	38.42	34.26	37.55	38.36	37.86
4. CFor	101.10	94.05	94.68	92.47	94.78	93.90	94.17	90.02
5. 1,1,1	69.23	68.80	67.92	69.14	69.18	67.49	69.17	66.82
6. Bz	43.78	43.33	42.92	44.38	45.25	43.78	44.36	43.03
7. CCL4	106.41	100.08	116.59	110.76	96.69	101.89	99.85	101.80
8. Tric	69.72	69.04	67.30	69.19	72.84	70.61	66.52	71.27
9. Tol	45.48	47.08	47.57	48.22	49.71	48.25	48.59	48.21
10. Perc	60.03	61.75	61.33	62.75	64.77	63.78	63.38	62.94
11. pmX	2.67	17.54	17.44	19.48	21.78	21.03	21.51	20.65
12. oX	0.87	22.00	19.43	23.90	26.91	27.44	26.84	26.46

Table 5 Recovery Efficiencies By Analytical System

Compound	No. of replicate	Avg Re. Eff. %	C.V %
1. MECL	8	20.88	28.99
2. DCM	8	103.66	6.64
3 C6	8	99.58	2.42
4. CFOR	8	99.01	7.56
5. 1,1,1	8	97.73	4.80
6. Bz	8	108.15	3.09
7. CCl4	8	103.63	9.81
8. Tric	8	96.30	7.61
9. Tol	8	105.40	4.08
10. Perc	8	120.87	6.11
11. PMX	8	112.38	11.81
12. OX	8	112.70	8.10

Table 6 Reproducibility Comparison of GC Detector Response

		CV	
	Compound	FID (7 replicates)	ECD (11 replicates)
1.	MECL	0.98%	
2.	DCM	1.93%	
3.	C6	2.04%	
4.	CFOR	2.93%	9.12%
5.	1,1,1	2.91%	16.72%
6.	Bz	2.47%	
7.	CCL4	5.33%	12.83%
8.	TRIC	2.75%	12.95%
9.	Tol	2.41%	
10	Perc	4.80%	6.65%
11	PMX	9.75%	
12	OX	8.96%	

Table 7 Experimental Data On FID versus ECD Response Undr Different Pressure (in-Hg) Loading of Standards (2ml, 165^{o} C)

		Peak area of FID				
	Compound	28"	20"	10"	5"	2"
1.	MECL	1140	837	448	284	178
2.	DCM	3599	2332	1124	563	302
3.	C6	13228	9212	4441	2759	1619
4.	CFOR	3656	2270	1072	594	371
5.	1,1,1	4843	3217	1543	987	571
6.	Bz	24574	15433	7250	4346	2510
7.	CCL4	2104	1304	563	406	194
8.	TRIC	9625	5544	2448	1360	772
9.	Tol	36656	21286	9208	5076	2846
10	Perc	9421	5372	2282	1264	654
11	PMX	33999	19954	8438	4608	2516
12	OX	50818	30057	13349	7184	4008

		Peak area of ECD				
	Compound	28"	20"	10"	5"	2"
1.	MECL					
2.	DCM	166	140			
3.	C6					
4.	CFOR	1686	1296	884	522	288
5.	1,1,1	1610	1284	587	355	166
6.	Bz					
7.	CCL4	16784	12214	5554	3434	1715
8.	TRIC	1998	1578	896	497	317
9.	Tol					
10	Perc	19992	14555	6823	3298	1862
11	PMX					
12	OX					

Table 8 Linearity Comparison of GC Detector By Response vs Pressure Loading

	Compound	No of observations	R squared FID	ECD	ng range
1.	MECL	5	1.00		1.26˜8.09
2.	DCM	5	0.99		6.03˜71.91
3.	C6	5	1.00		4.58˜37.79
4.	CFOR	5	0.98	0.99	11.07˜109.05
5.	1,1,1	5	0.99	0.99	7.22˜61.25
6.	Bz	5	0.99		6.04˜59.12
7.	CCL4	5	0.98	1.00	8.10˜87.87
8.	TRIC	5	0.97	1.00	10.07˜125.56
9.	Tol	5	0.97		7.91˜101.91
10	Perc	5	0.97	1.00	11.16˜160.82
11	PMX	5	0.98		6.25˜84.47
12	OX	5	0.98		8.69˜110.21

Table 9 Parameters of The Standard Gas Mixture

	Standard Compound	Conc. ppm	M.W g	B.P C	M.P C
1.	MECL	2.98	50.50	-23.7	-97.7
2.	DCM	11.86	84.00	40.1	-96.7
3.	C6	7.54	86.00	68.8	-95.3
4.	CFOR	12.66	119.50	61.2	-63.5
5.	1,1,1	7.89	133.40	75.0	-30.6
6.	Bz	9.50	78.10	80.1	5.5
7.	CCL4	10.15	153.80	76.7	-22.6
8.	TRIC	9.15	131.40	86.7	-73.0
9.	Tol	9.05	92.00	110.7	-95.0
10	Perc	6.49	165.80	121.0	-19.0
11	PMX	3.51	106.17	138.8	*
12	OX	4.46	106.17	144.0	0.1

Note : p-Xylene -25.2 C
o-Xylene -47.9 C

Table 10 Experiment Data of Adsorbates Adsorption/Desorption

Adsorption/Desorption Efficiencies, %

Compound	Trap I.D. T-307	T-502	T-507	T-309	avg	C.V
1. MECL	16	16	9	26	16.9	36.0
2. DCM	109	94	95	106	101.1	6.4
3 C6	104	97	96	102	100.0	3.3
4. CFOR	110	97	98	101	101.6	5.0
5. 1,1,1	103	101	101	103	102.1	1.2
6. Bz	108	105	103	110	106.8	2.5
7. CCl4	90	98	97	94	95.0	3.5
8. Tric	103	101	102	106	103.2	1.8
9. Tol	108	109	109	111	109.3	1.1
10. Perc	130	120	118	130	124.5	4.5
11. PMX	98	128	129	130	121.1	11.0
12. OX	104	121	125	126	119.1	7.4

Breakthrough Compounds, %

Compound	T307	T-502	T-507	T-309	Avg
1. MECL	98	99	101	100	99.6
2. DCM	0	0	0	0	0.0
3 C6	0	0	0	0	0.0
4. CFOR	0	0	0	0	0.0
5. 1,1,1	0	0	0	0	0.0
6. Bz	3	3	5	3	3.2
7. CCl4	0	0	0	0	0.0
8. Tric	0	0	0	0	0.0
9. Tol	1	0	2	1	0.9
10. Perc	0	3	5	3	2.8
11. PMX	0	1	5	1	1.7
12. OX	0	0	3	0	0.9

Table 11 Purge -out Percentage of Various Purge Flow Rate

Compound	No. of Replicate	Purge-out Percent			
		4.8 ml/min *		9.8 ml/min **	
		Avg	S.D	Avg	S.D
1. MECL	3	97	0.7	95	1.8
2. DCM	3	100	0.0	100	0.0
3 C6	3	15	4.6	57	7.4
4. CFOR	3	54	16.2	100	0.0
5. 1,1,1	3	23	5.9	56	4.6
6. Bz	3	18	2.3	37	7.1
7. CCl4	3	19	5.4	59	6.0
8. Tric	3	5	1.1	18	4.7
9. Tol	3	5	0.9	2	0.8
10. Perc	3	-9	0.9	-14	4.3
11. PMX	3	13	0.6	11	1.7
12. OX	3	7	0.4	3	1.7

Note : * --- Total Flow = 7.01 l
** --- Total Flow = 14.27 l

Table 12 Ratio Comparison of FID vs ECD Response

Compoun	Standard Gas			Spiked Air Sampl			ratio factor
	FID	ECD	F/E(1)	FID |	ECD	F/E(2)	(2)/(1)
1. MECL	1176	70	16.80	830			
2. DCM	2789	129	21.62	7166	155	46.23	2.14
3 C6	12634			41351			
4. CFOR	2833	1310	2.16	9379	1327	7.07	3.27
5. 1,1,1	4514	1404	3.22	15060	4720	3.19	0.99
6. Bz	17491			66340			
7. CCl4	1850	13435	0.14	3663	17243	0.21	1.54
8. Tric	5265	1107	4.76	8867	1297	6.84	1.44
9. Tol	17486			191320			
10. Perc	3660	6406	0.57	15789	9603	1.64	2.88
11. PMX	9708			101400			
12. OX	13977			51714			

Note : | --- Pick up peak by the same retention time as the standard gas

Table 13 Concentrations From Front and Backup Tenax Tubes At High and Low Flow Rates
Total Flow : High -- 15.05 l (10.59 ml/min)
Low -- 7.7 l (5.42ml/min)
(Carteret Site: 9/30/88, ppb)

	Compound	Front Low	Front High	Backup Low	Backup High	Blank
1.	MECL	0.29	0.13	0.36	0.19	0.07
2.	DCM	7.35	3.03	1.29	4.46	0.00
3.	C6	1.35	1.36	0.02	0.01	0.00
4.	CFOR	0.00	0.00	0.00	0.00	0.00
5.	1,1,1	1.23	1.26	0.00	0.00	0.00
6.	Bz	1.94	1.85	0.17	0.10	0.19
7.	CCL4	0.09	0.09	0.00	0.00	0.00
8.	TRIC	0.05	0.07	0.00	0.00	0.00
9.	Tol	9.84	8.88	0.08	0.13	0.04
10	Perc	0.13	0.17	0.00	0.00	0.00
11	PMX	2.45	2.37	0.02	0.03	0.02
12	OX	0.81	0.77	0.00	0.02	0.00

Table 14 Concentrations From Front and Backup Tenax Tubes At High and Low Flow Rates
Total Flow : High -- 12.93 l (9.26 ml/min)
Low -- 6.14 l (4.4 ml/min)
(Elizabeth Site: 9/30/88, ppb)

	Compound	Front Low	Front High	Backup Low	Backup High	Blank
1.	MECL	0.37	0.14	0.27	0.32	0.07
2.	DCM	9.90	5.75	1.38	3.48	0.00
3.	C6	2.17	1.96	0.02	0.01	0.00
4.	CFOR	0.00	0.00	0.00	0.00	0.00
5.	1,1,1	0.80	0.80	0.00	0.00	0.00
6.	Bz	2.23	1.97	0.25	0.17	0.19
7.	CCL4	0.09	0.07	0.00	0.00	0.00
8.	TRIC	0.07	0.07	0.00	0.00	0.00
9.	Tol	7.27	6.65	0.09	0.07	0.04
10	Perc	0.16	0.15	0.00	0.00	0.00
11	PMX	3.01	2.81	0.06	0.05	0.02
12	OX	0.85	0.82	0.01	0.03	0.00

Table 15 Average Concentration Percent of Front and Backup Tubes (Carteret, 9/30/88)

	Compound	Front,%		Backup,%	
		Avg	R.A.E	Avg	R.A.E
1.	MECL	43	38	57	31
2.	DCM	64	42	36	55
3.	C6	99	0	1	20
4.	CFOR				
5.	1,1,1	100	1	0	
6.	Bz	93	2	7	25
7.	CCL4	100	2	0	
8.	TRIC	100	16	0	
9.	Tol	99	5	1	26
10	Perc	100	14	0	
11	PMX	99	2	1	37
12	OX	99	3	1	100

Note : R.A.E --- Relative Average Error

Table 16 Average Concentration Percent of Front and Backup Tubes (Elizabeth, 9/30/88)

	Compound	Front,%		Backup,%	
		Avg	R.A.E	Avg	R.A.E
1.	MECL	46	45	54	8
2.	DCM	76	26	24	43
3.	C6	99	5	1	26
4.	CFOR				
5.	1,1,1	100	0	0	
6.	Bz	91	6	9	19
7.	CCL4	100	11	0	
8.	TRIC	100	1	0	
9.	Tol	99	4	1	6
10	Perc	100	1	0	
11	PMX	98	3	2	12
12	OX	97	2	3	54

Note : R.A.E --- Relative Average Error

Table 17 Reproducibility of Spiked Tenax

Compound	No. of replicate	Avg ng	S.D ng	C.V %
1. MECL	8	1.74	0.51	29.26
2. DCM	8	57.80	3.97	6.87
3 C6	8	35.71	0.85	2.38
4. CFOR	8	82.83	6.13	7.40
5. 1,1,1	8	56.91	2.44	4.28
6. Bz	8	44.38	1.49	3.36
7. CCl4	8	89.51	9.22	10.30
8. Tric	8	64.01	4.53	7.08
9. Tol	8	48.52	1.74	3.58
10. Perc	8	71.92	4.22	5.86
11. PMX	8	23.15	2.65	11.42
12. OX	8	29.50	2.27	7.69

Table 18 Audit Sample Results for Staten Island Study (Tenax Tubes)

Compound	Spiked, ng T-103	Spiked, ng T-109	Spiked, ng T-110	Reported, ng T-103	Reported, ng T-109	Reported, ng T-110
MeCl	---	---	---	---	---	---
CFor	72	72	108	79	85	134
1,1,1	97	97	146	97	103	146
CCl4	116	116	174	163	167	246
Bz	128	128	192	155	155	226
Tric	142	142	213	175	176	256
Tol	147	147	220	233	234	341
Perc	118	118	177	155	162	230
pmX	---	---	---	---	---	---
oX	128	128	192	118	118	177

Tenax-118 (Tenax blank) : Bz 2 ng, Tol 2 ng, oX 3 ng

Table 19 Percent Bias of Audit Results

Compound	Bias. %		
	T-103	T-109	T-110
MeCl	---	---	---
CFor	9.7	18	24
1,1,1	0	6.2	0
CCl4	40	44	41
Bz	21	21	18
Tric	23	24	20
Tol	58	59	55
Perc	31	37	30
pmX	---	---	---
oX	-7.8	-7.8	-7.8

Table 20 Linear Regression Data of Ambient Air Data
By High vs Low Flow

Compound	Carteret			Elizabeth		
	No of Observation	R Squared	Slope	No of Observation	R Squared	Slope
MECL	49	0.6	0.38	33	0.79	0.47
DCM	-	-	-	-	-	-
C6	49	0.94	0.92	33	0.92	0.74
CFOR	49	0.95	0.76	33	0.72	0.73
1,1,1	49	0.59	0.51	33	0.75	0.82
Bz	49	0.94	1.02	33	0.89	0.70
CCL4	49	0.71	0.45	33	0.95	0.93
TRIC	49	0.89	0.95	33	0.92	0.94
Tol	49	0.94	0.95	33	0.94	0.82
Perc	49	0.86	0.78	33	0.94	0.81
PMX	49	0.82	0.85	33	0.93	0.83
OX	49	0.77	0.83	33	0.89	0.82

Note : R Squared --- Coefficient of Determination
Slope --- X axis, Low Flow Tube and
Y axis, High Flow Tube

Table 21 Precision of Ambient Air Data
Carteret : March 4 ~ December 29 1988
Elizabeth : June 14 ~ Dec 29 1988

	Carteret			Elizabeth		
Compound	No of observation	M.R.A.E %	S.D %	No of observation	M.R.A.E %	S.D %
MECL	49	34.52	19.26	31	33.30	12.83
DCM	-	-	-	-	-	-
C6	49	7.42	6.97	33	10.34	8.18
CFOR	-	-	-	-	-	-
1,1,1	49	19.98	24.02	33	13.85	15.67
Bz	49	8.96	7.96	33	9.51	5.38
CCL4	48	20.24	24.25	33	9.56	8.86
TRIC	35	26.08	29.95	24	21.29	31.04
Tol	49	7.58	12.96	33	7.7	5.52
Perc	48	18.45	24.96	33	9.26	14.38
PMX	49	8.3	15.05	33	7.23	5.71
OX	49	8.9	12.8	33	8.35	6.12

Note : M.R.A.E --- Mean of Relative Average Error

Table 22 Precision of Project "Shoot-out" #2 ---
Staten Island Interlaboratory Comparison Stud

Compound	No of Observations	M.R.A.E %	S.D %
1. MECL	6	37.75	8.17
2. DCM	6	42.70	4.89
3 C6	6	15.92	9.71
4. CFOR	6	29.36	12.28
5. 1,1,1	6	21.88	7.82
6. Bz	6	14.36	8.15
7. CCl4	6	20.14	12.79
8. Tric	6	13.11	16.18
9. Tol	6	6.23	4.81
10. Perc	6	8.20	5.14
11. PMX	6	9.54	6.19
12. OX	6	11.00	7.32

Note : M.R.A.E --- Mean of Relative Average Error

Table 23 Results of Staten Island Interlaboratory Comparison Study

% Difference From The Mean for All Compounds/All Organizations

	NJIT Average 2-Tubes	NYDEC Enviro-chem Average 2-Tubes	NYDEC ATD-50 Average 2-Tubes	CSI Average 2-Tubes	UMDNJ Average 2-Tubes	TEXAS A&EPA/RTP 2 Tube Average	 4 Tube Average
Day 1	-38.95	-61.14	N/S	-19.62	118.52	-55.52	-13.49
Day 2	N/S	-9.11	-11.52	11.23	-24.19	-210.99	39.77
Day 3	-16.7	-37.06	-7.26	-10.91	-15.54	-24.96	N/S
Day 4	8.65	-32.85	-30.78	13.06	-5.01	-8.92	N/S
Avg.	-15.67	-35.04	-16.52	-1.56	18.45	-75.10	13.14

Absolute Value Of The % Difference From The Mean for All Compounds/Organizations

	NJIT Average 2-Tubes	NYDEC Enviro-chem Average 2-Tubes	NYDEC ATD-50 Average 2-Tubes	CSI Average 2-Tubes	UMDNJ Average 2-Tubes	TEXAS A&EPA/RTP 2 Tube Average	 4 Tube Average
Day 1	38.95	61.14	N/S	28.4	126.21	55.52	28.89
Day 2	N/S	24.05	23.54	36.71	67.28	210.99	58.2
Day 3	20.86	37.06	35.46	49.79	44.66	42.11	N/S
Day 4	16.17	32.86	43.39	62.62	37.81	24.66	N/S
Avg.	25.33	38.78	34.13	44.38	68.99	83.32	43.55

Figure 1 : Analytical System Configuration

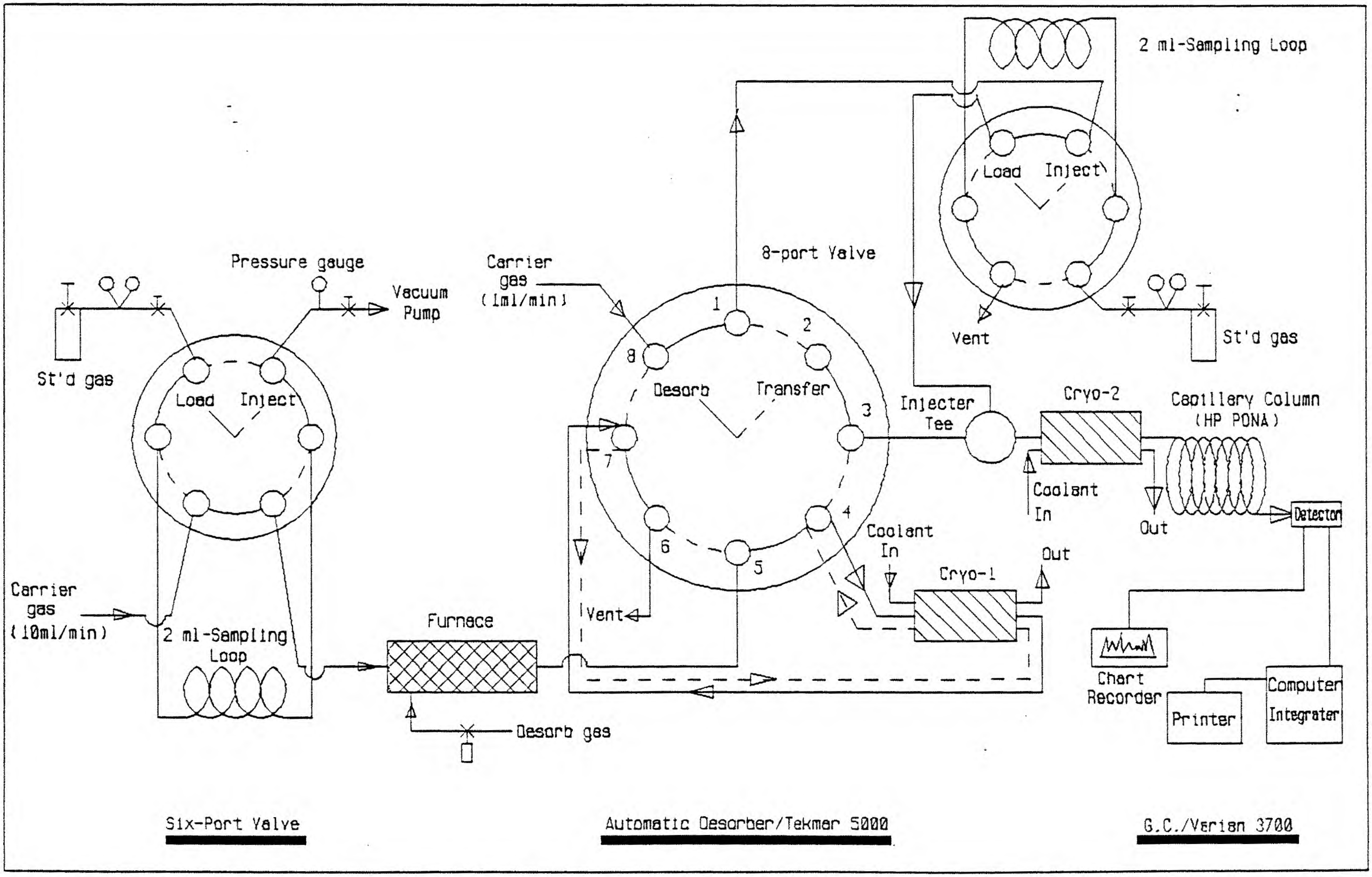


Figure 2 **Temp Effect On Transfer Line**

(Low boiling point compounds)

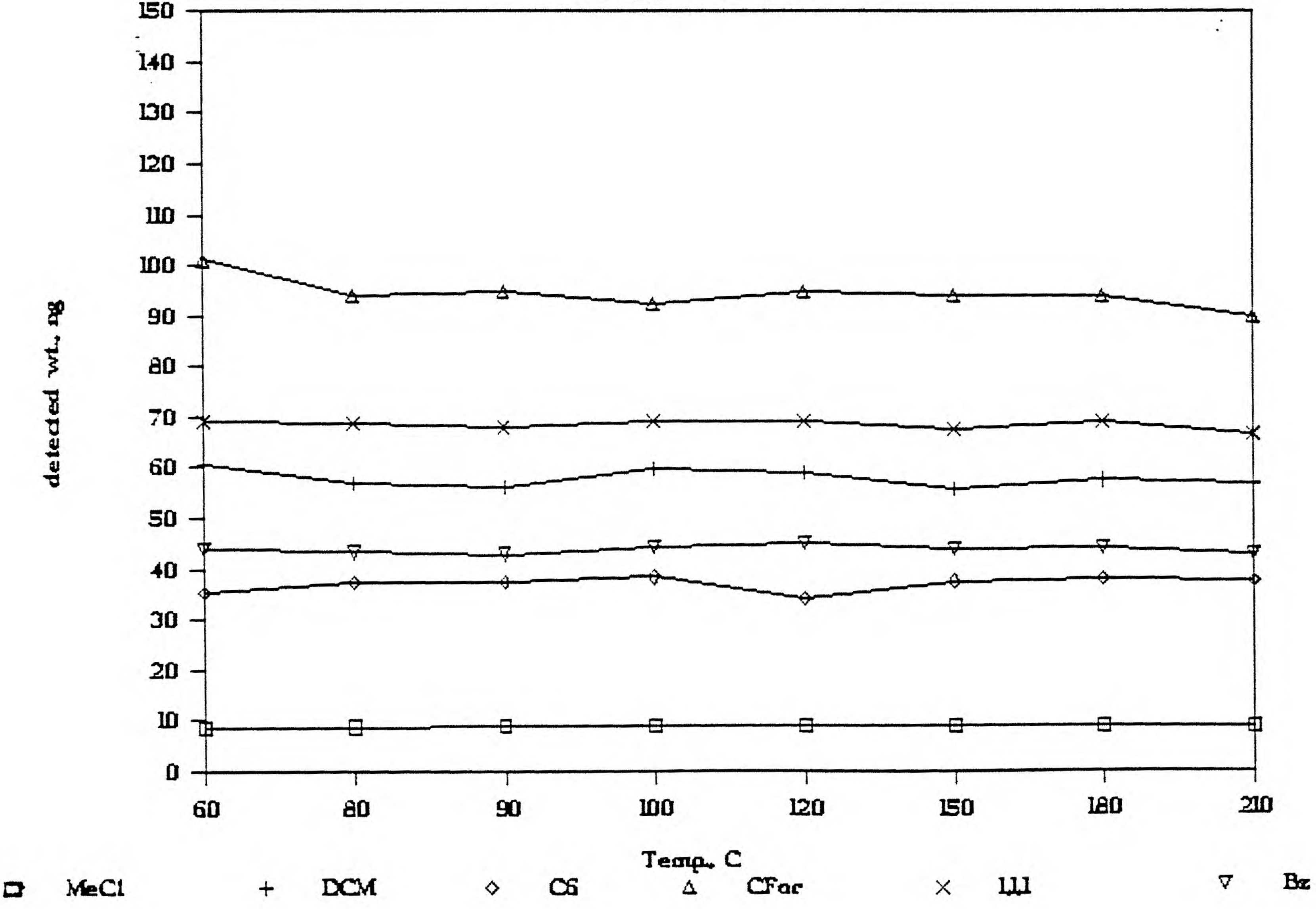


Figure 3 **Temp Effect On Transfer Line**

(High boiling point compounds)

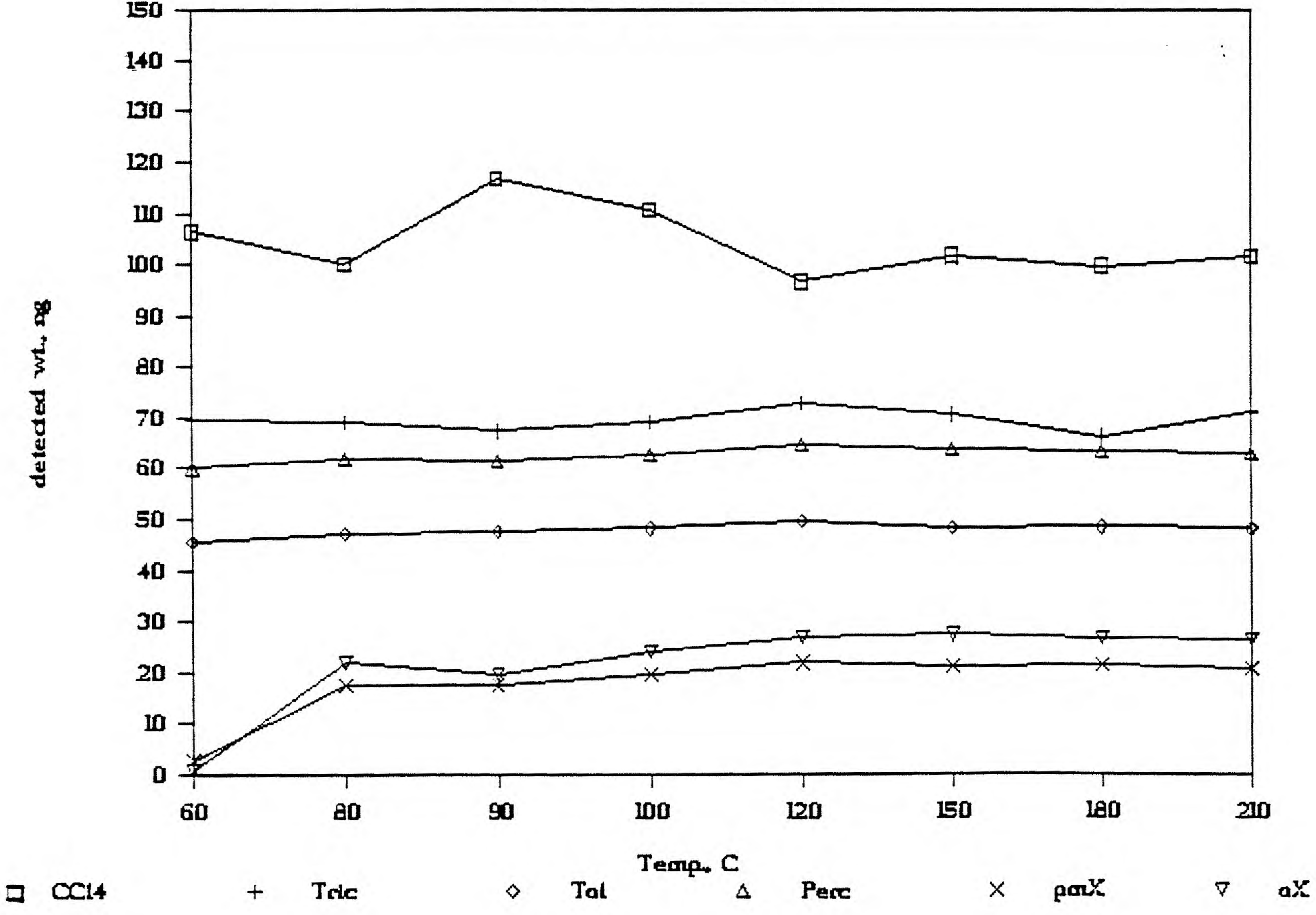


Figure 4-1 Linear Regression of FID Response vs P

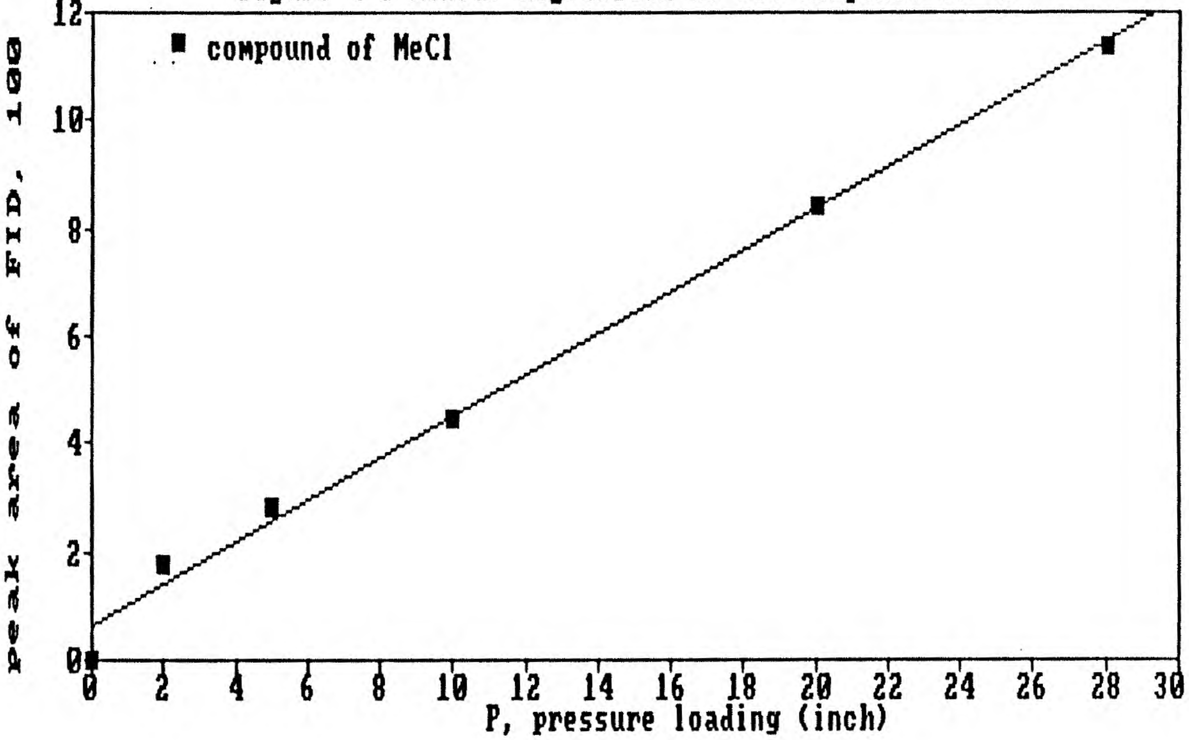


Figure 4-2 Linear Regression of FID Response vs P

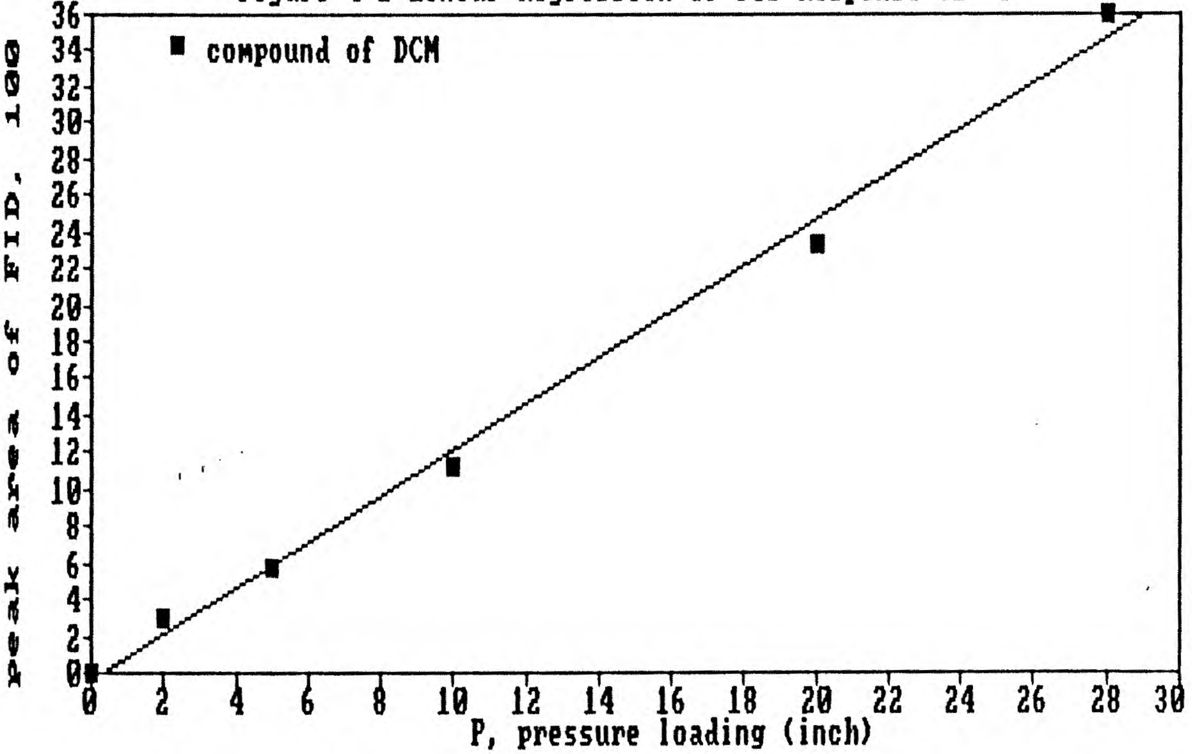


Figure 4-3 Linear Regression of FID Response vs P

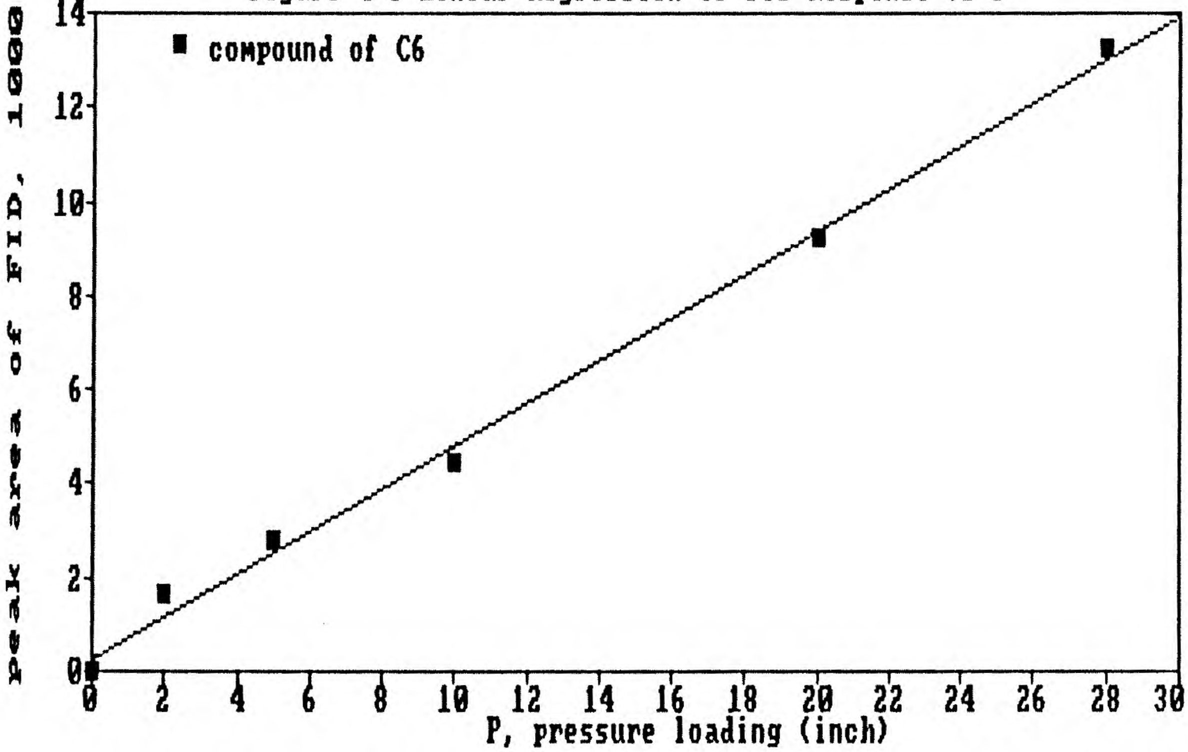


Figure 4-4 Linear Regression of FID Response vs P

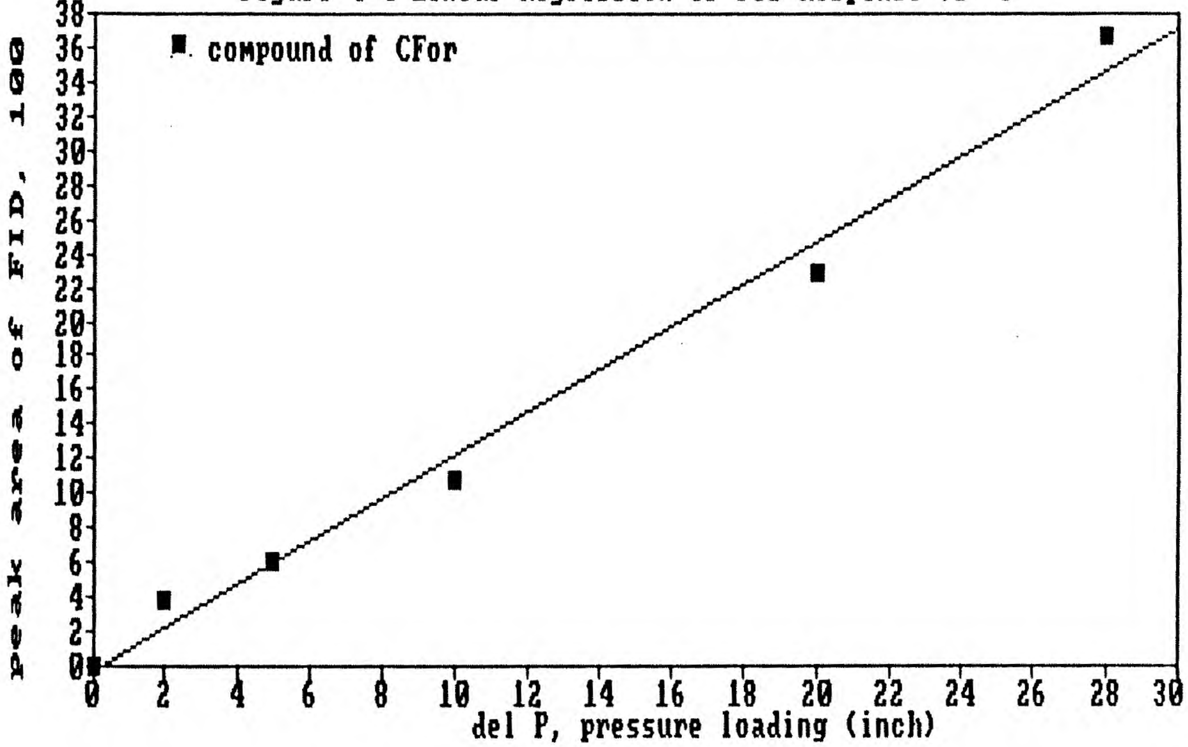


Figure 4-4A Linear Regression of ECD Response vs P

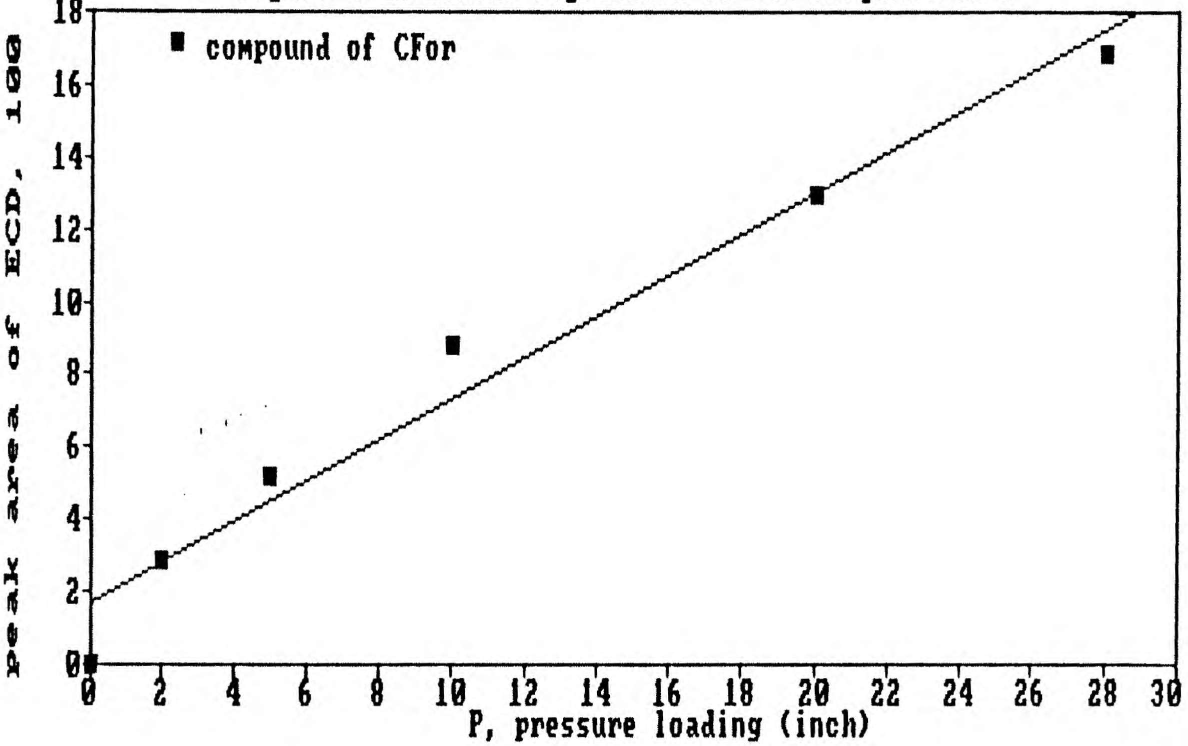


Figure 4-5 Linear Regression of FID Response vs P

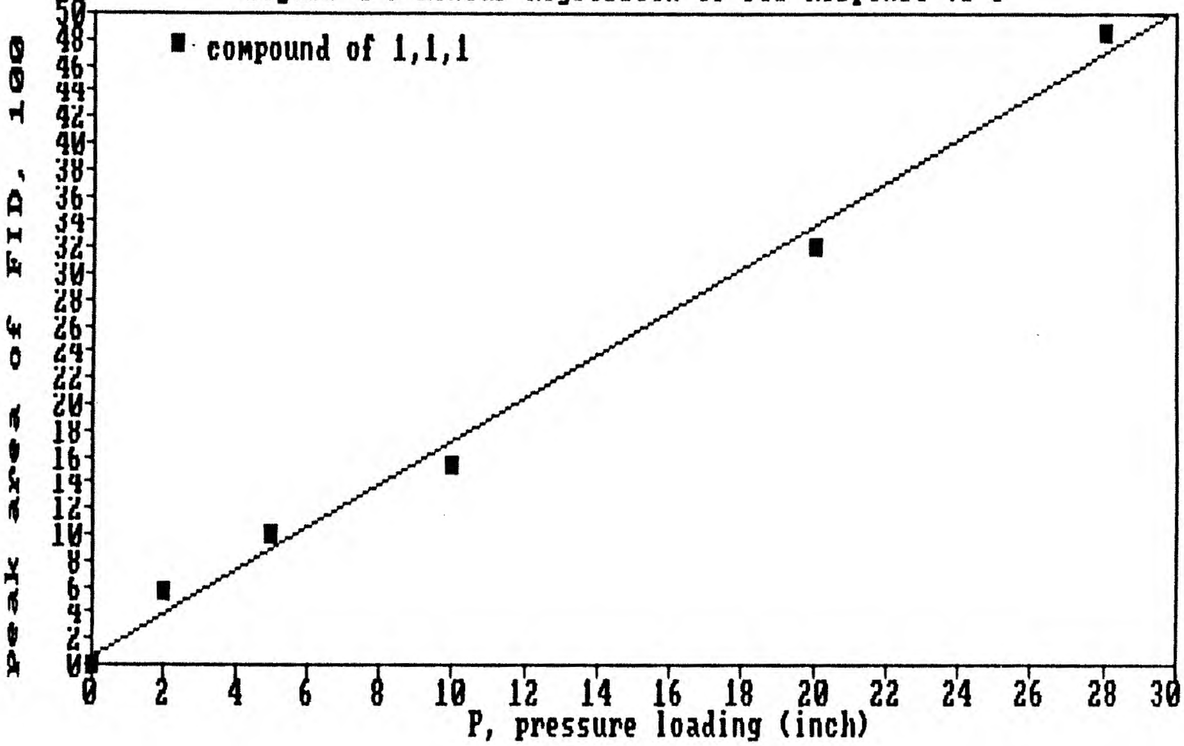


Figure 4-5A Linear Regression of ECD Response vs P

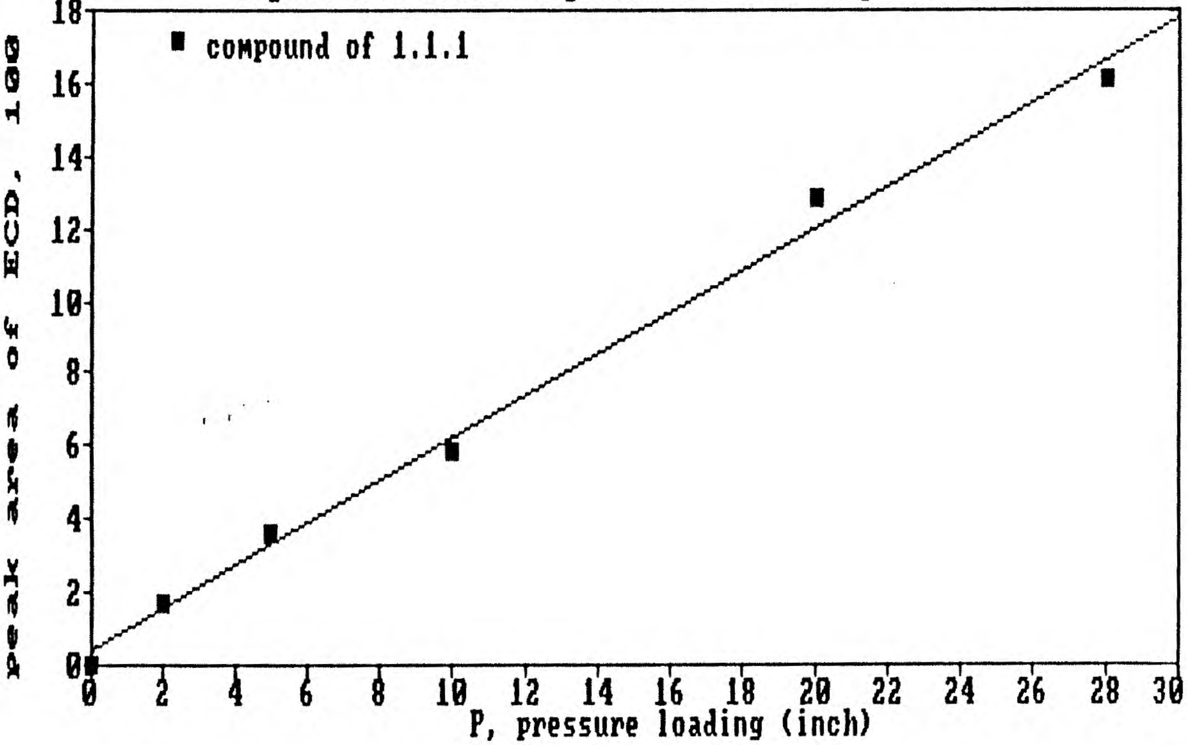


Figure 4-6 Linear Regression of FID Response vs P

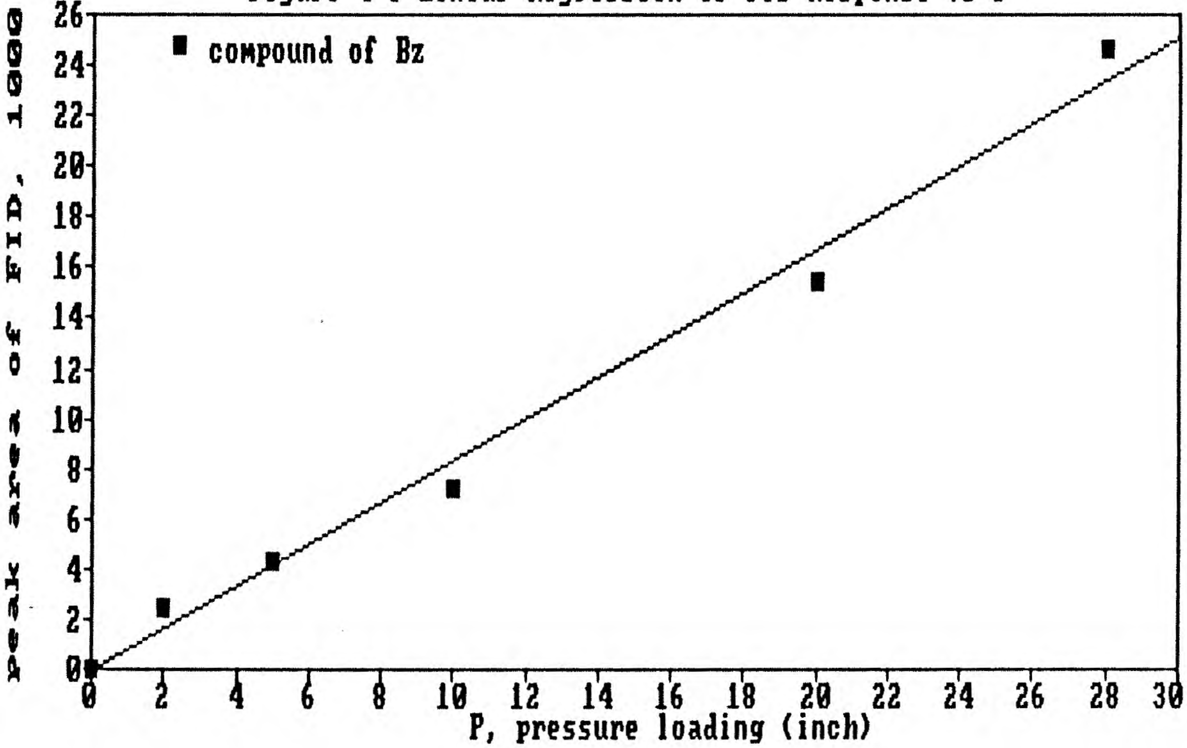


Figure 4-7 Linear Regression of FID Response vs P

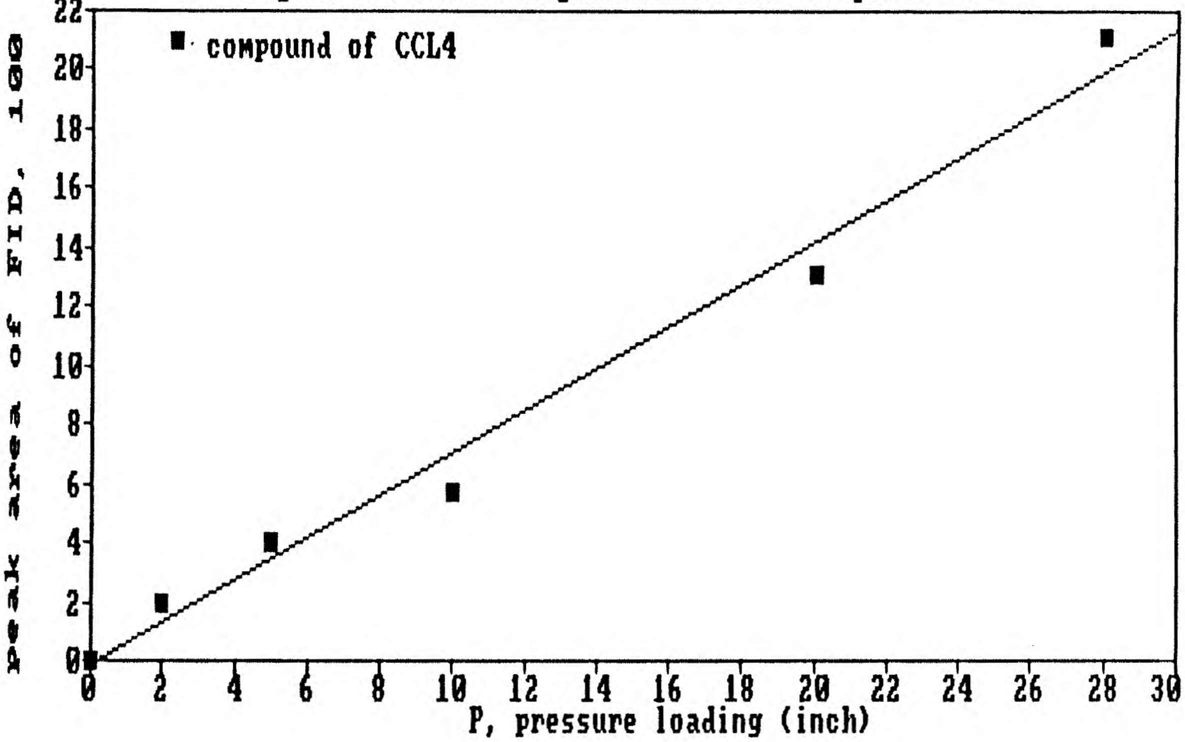


Figure 4-7A Linear Regression of ECD Response vs P

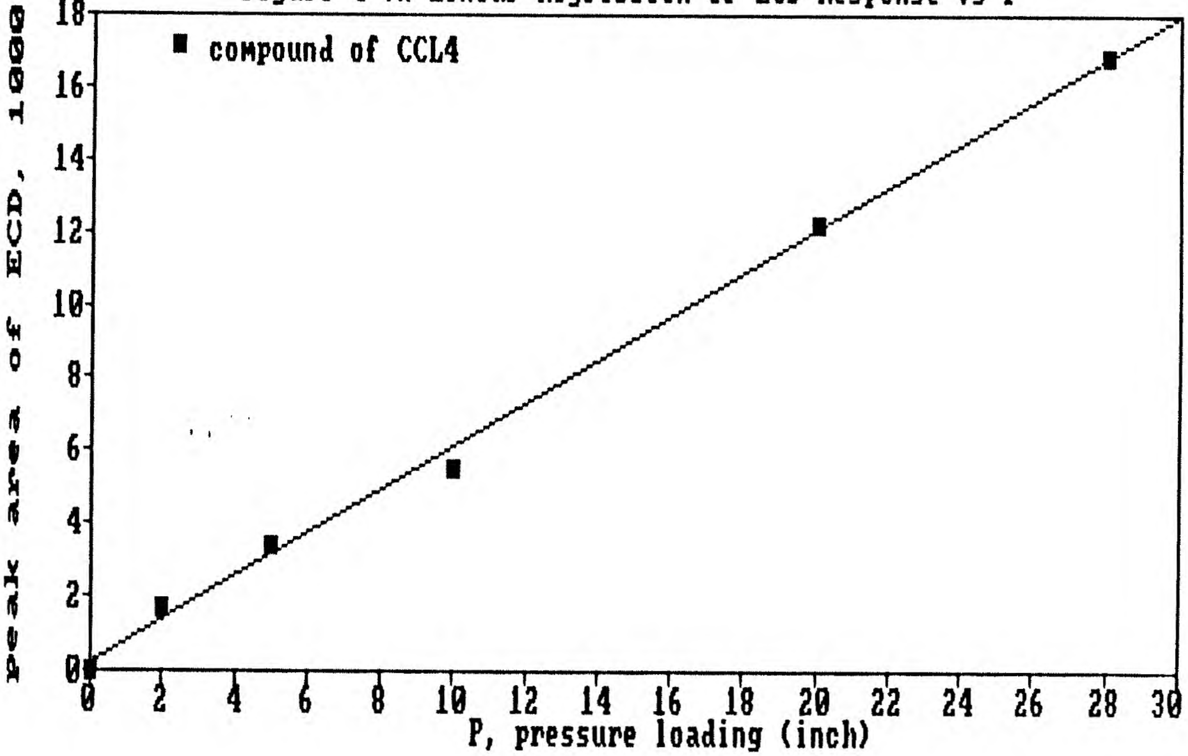


Figure 4-8 Linear Regression of FID Response vs P

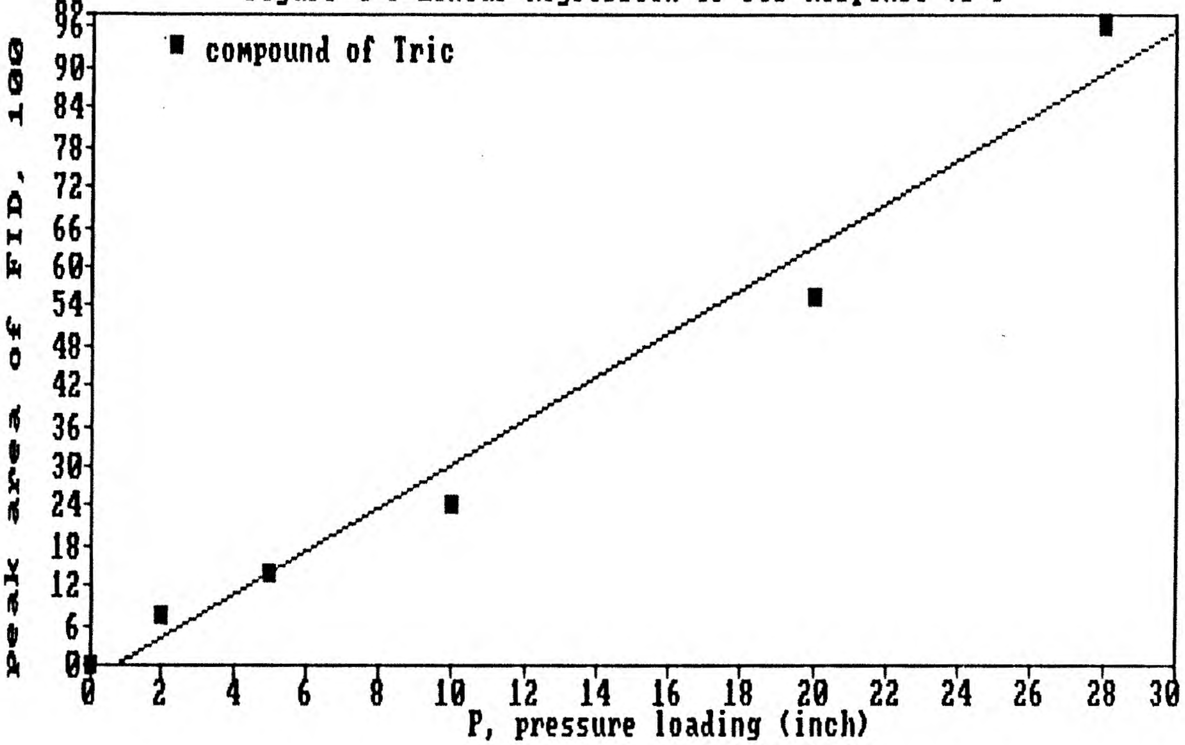


Figure 4-8A Linear Regression of ECD Response vs P

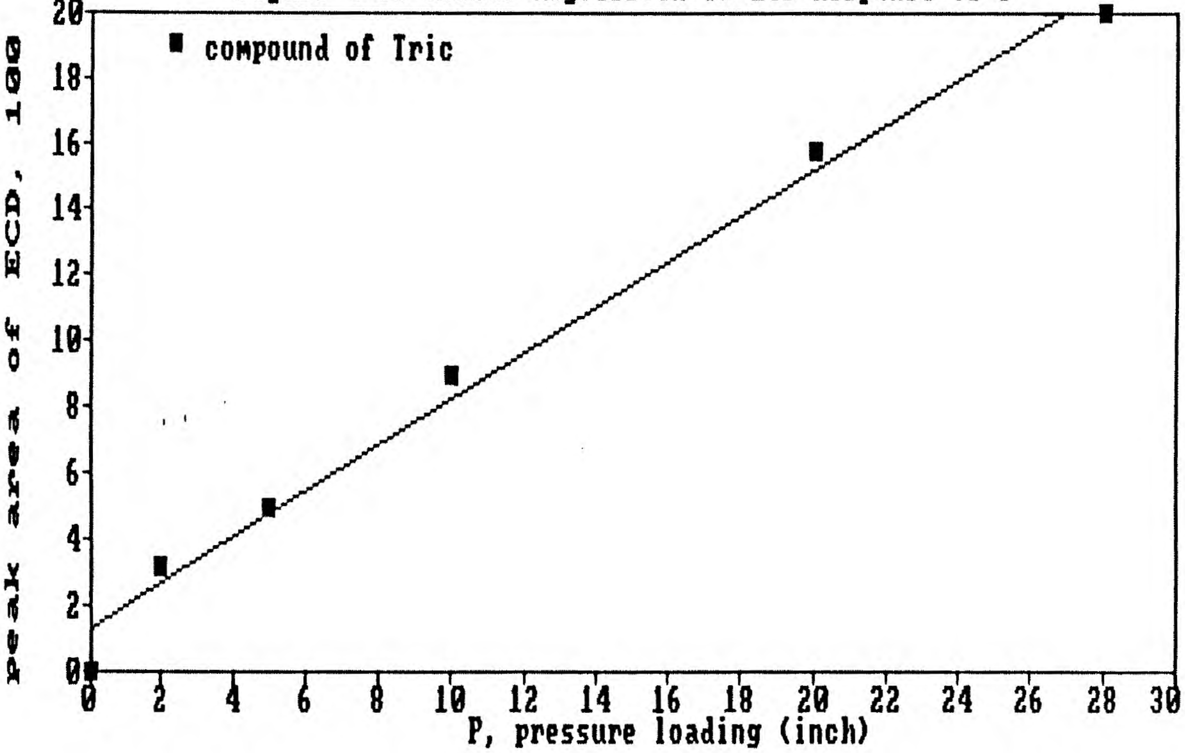


Figure 4-9 Linear Regression of FID Response vs P

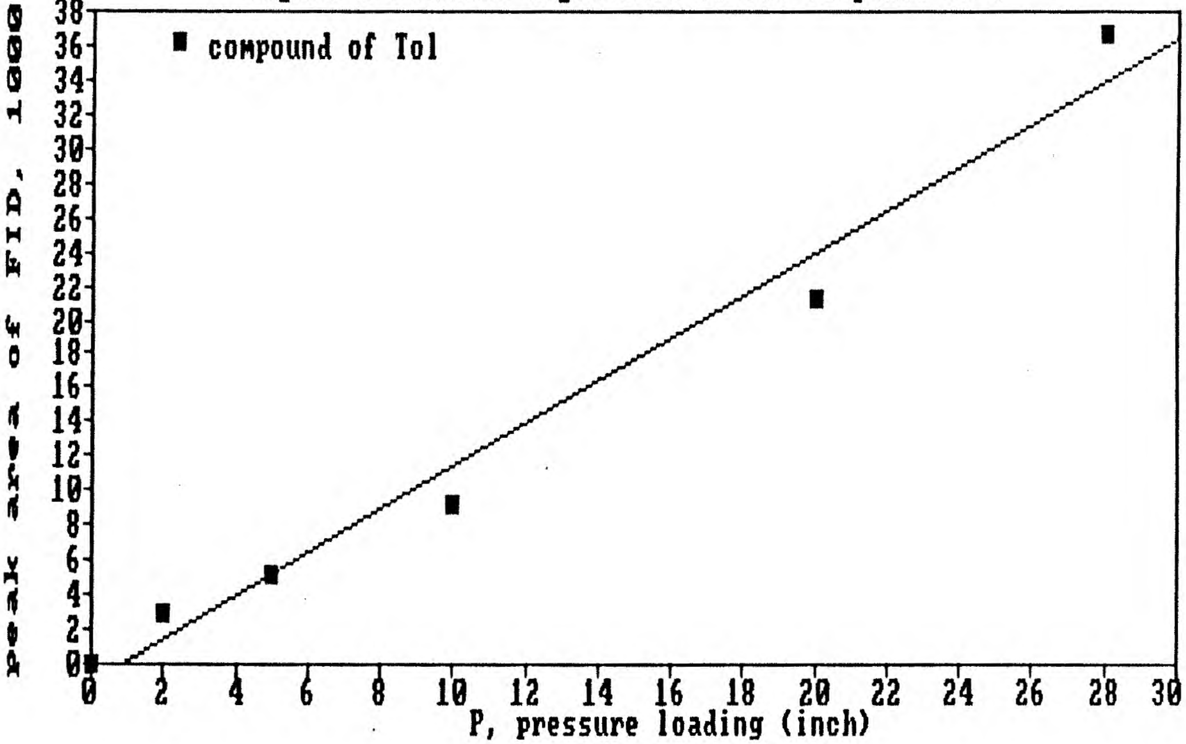


Figure 4-10 Linear Regression of FID Response vs P

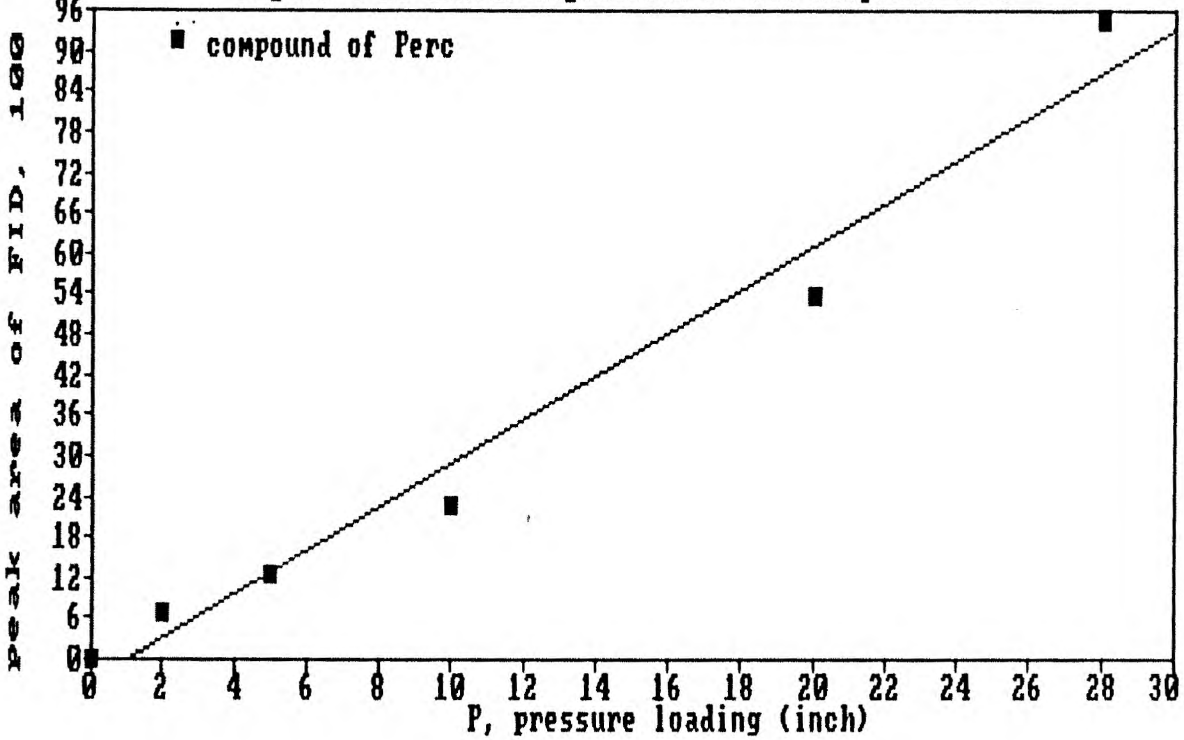


Figure 4-10A Linear Regression of ECD Response vs P

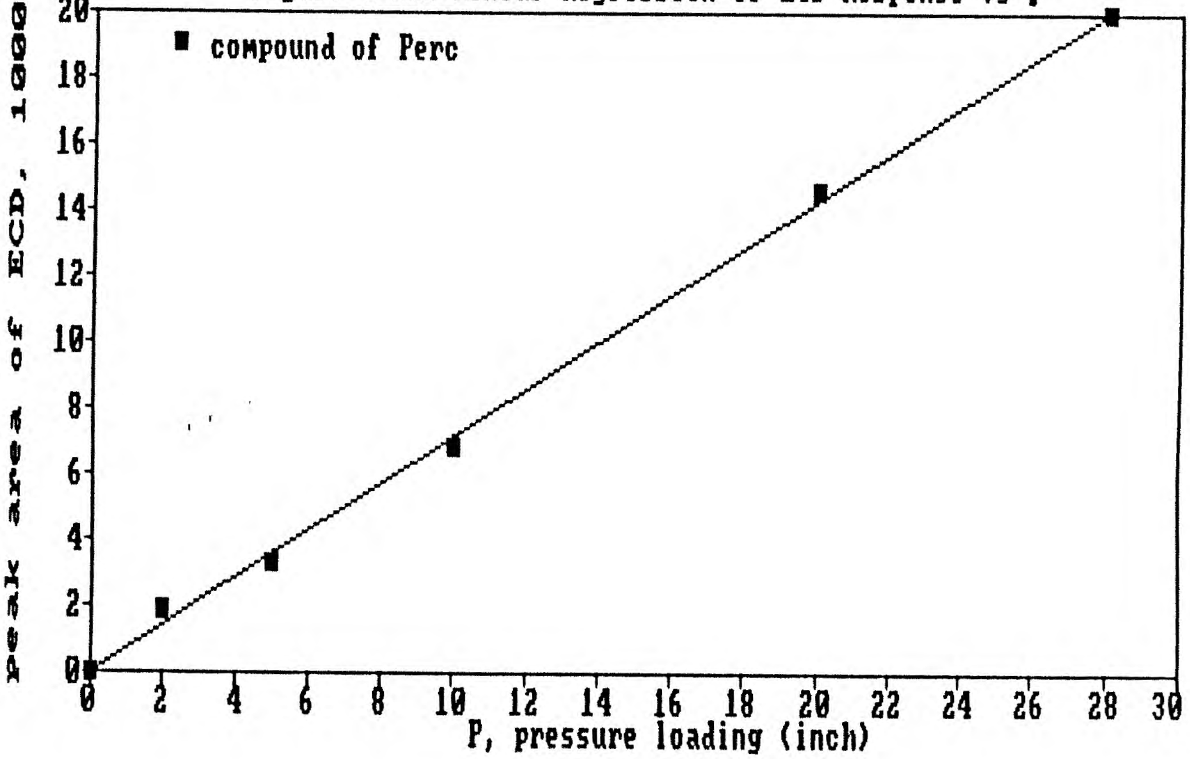


Figure 4-11 Linear Regression of FID Response vs P

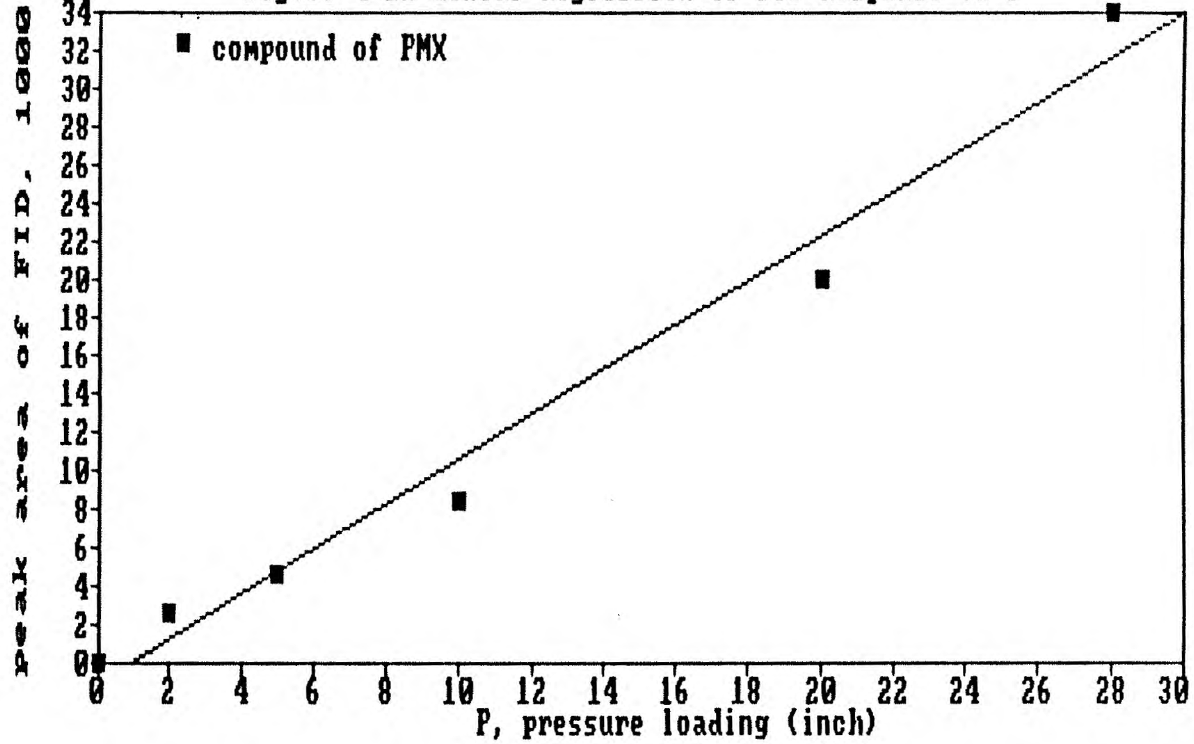


Figure 4-12 Linear Regression of FID Response vs P

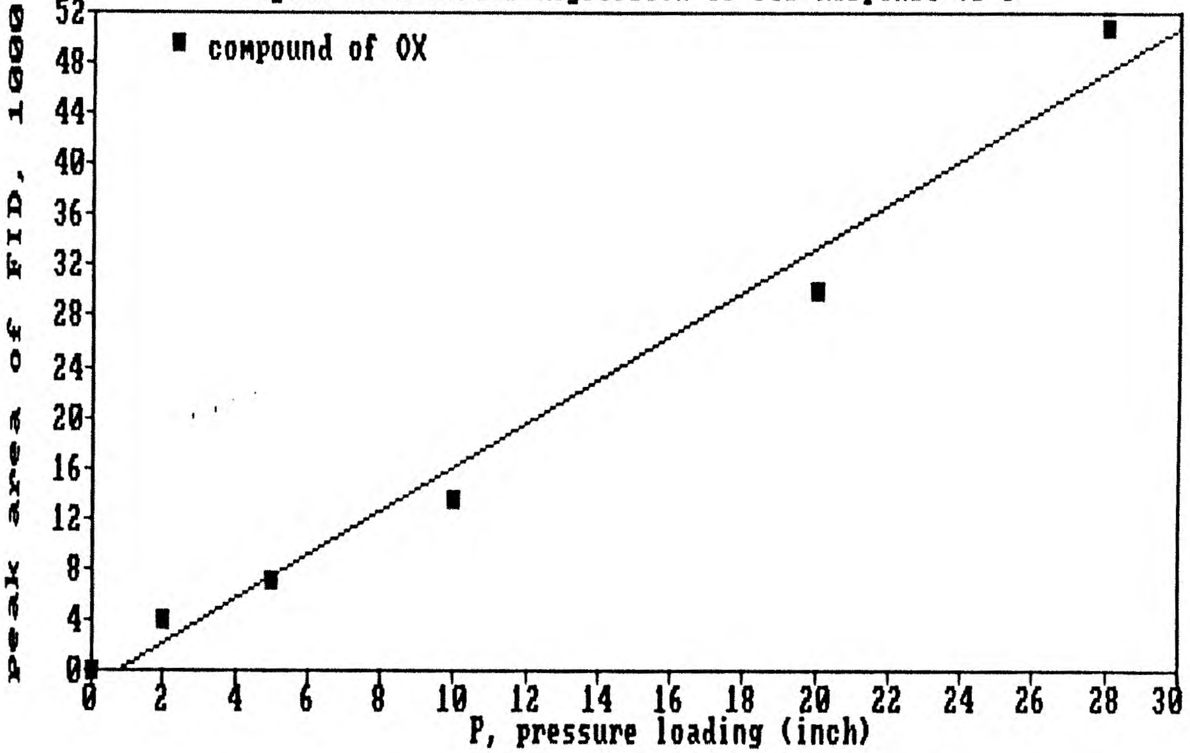
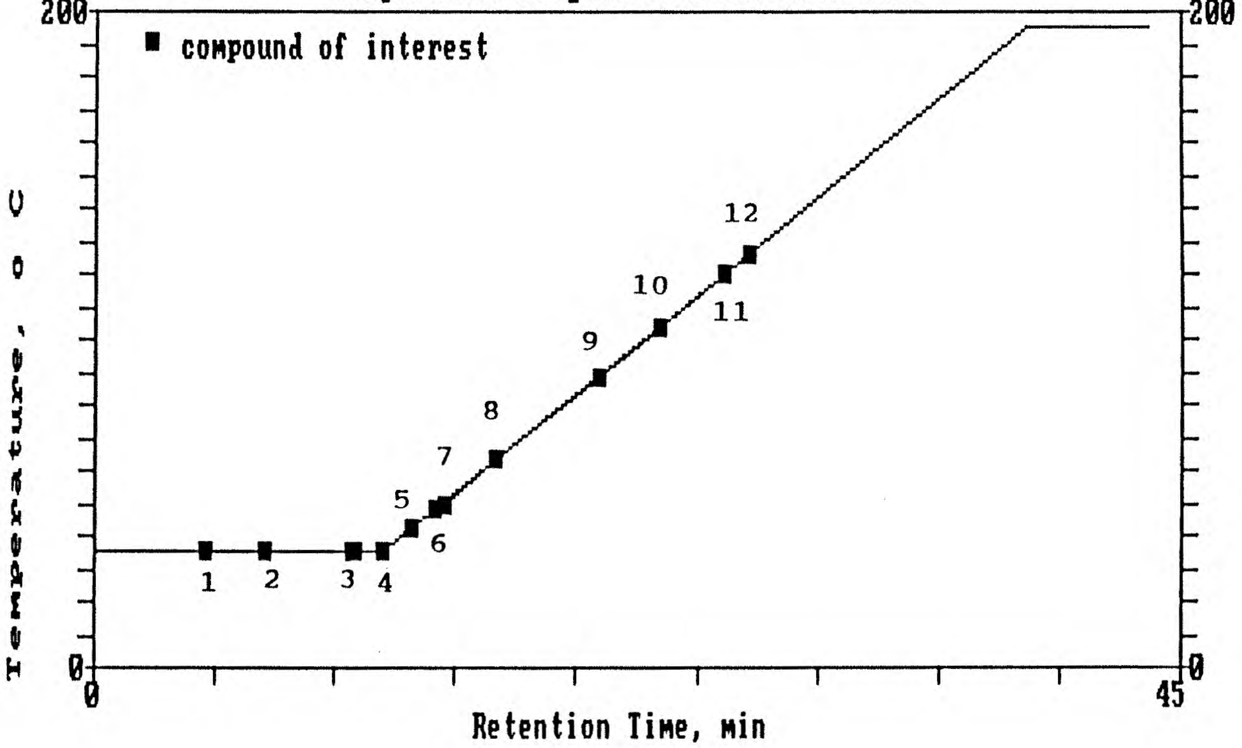
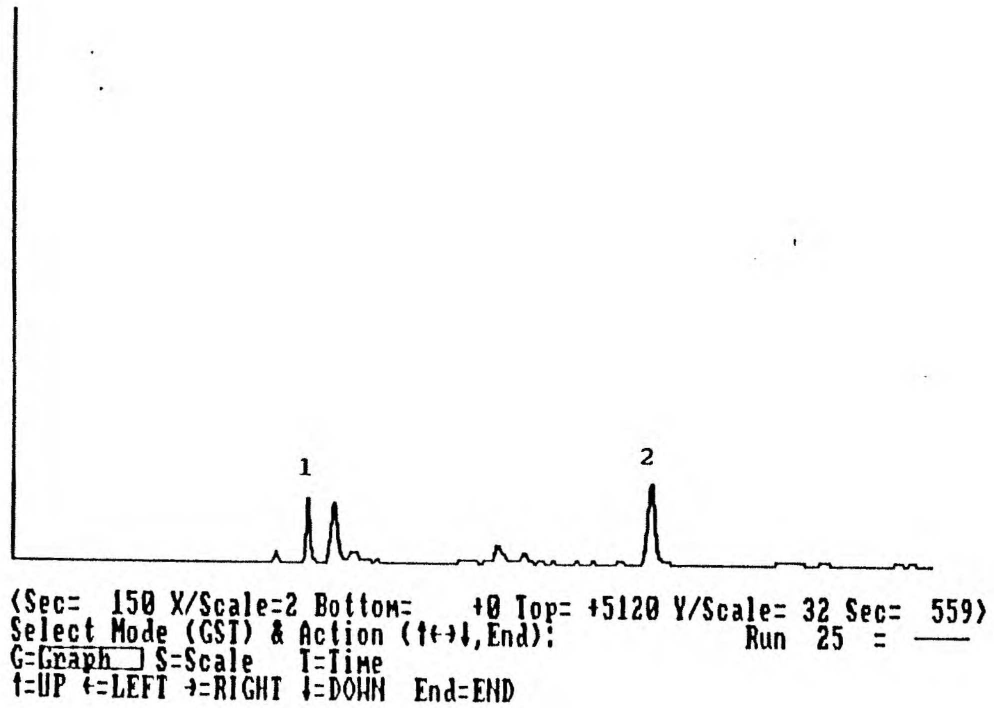


Figure 5 Temperature Program and Retention Time



Note :

1 --- MeCl 2 --- DCM 3 --- C6
4 --- CFor 5 --- 1,1,1 6 --- Bz
7 --- CCl4 8 --- Tric 9 --- Tol
10 --- Perc 11--- pmX 12--- oX



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Select Mode (GST) & Action (↑←→↓,End):              Run  25  = ___
G=Graph   S=Scale    T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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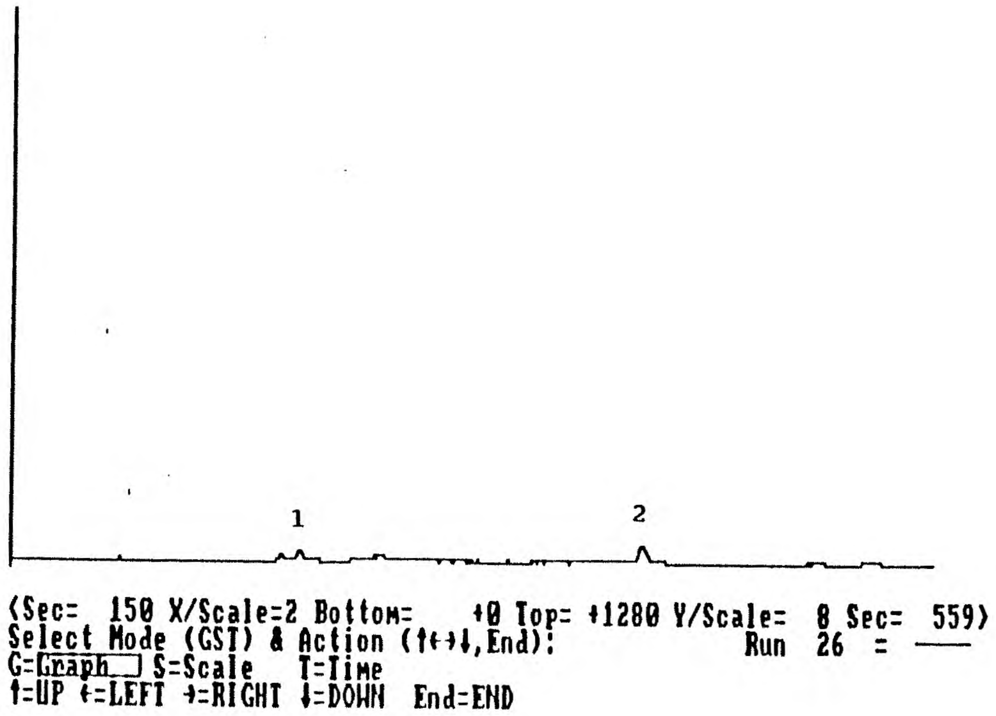
Figure 6 - 1 FID Response ^

ECD Response v (Standard Gas: 2 ml, 1 atm, 165 °C)



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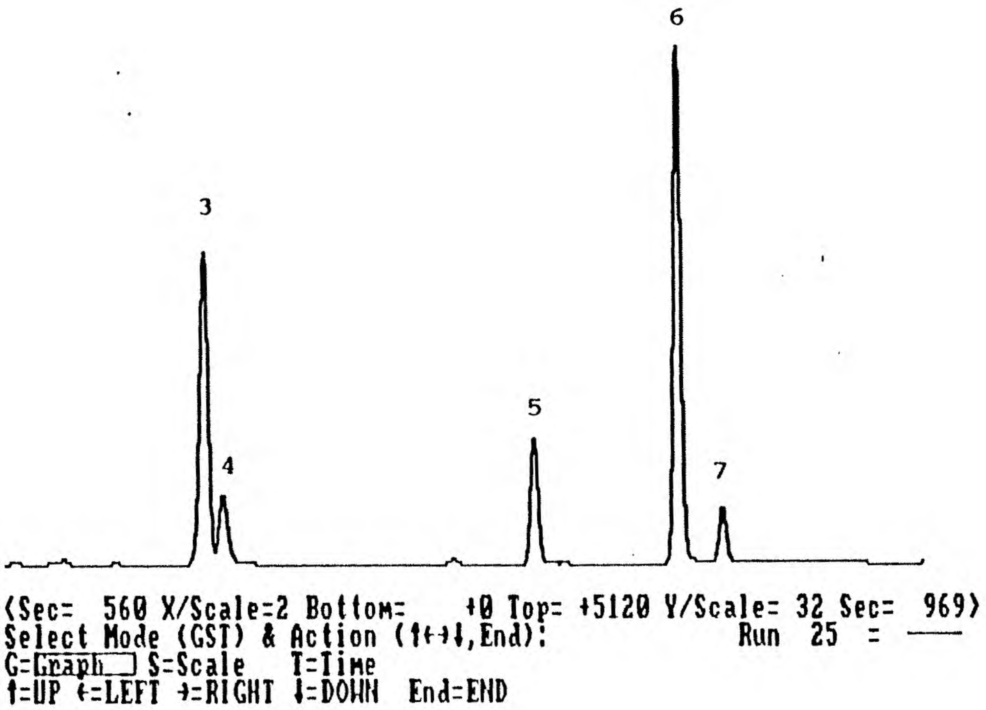

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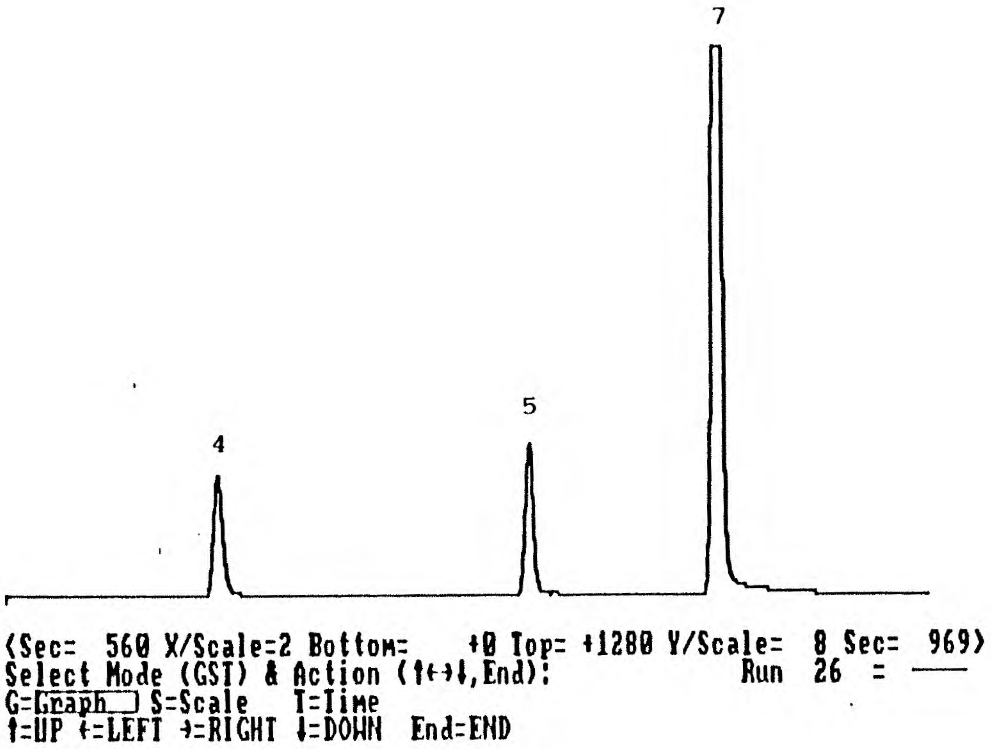
G=Graph    S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

Figure 6 - 2   FID Response ^

ECD Response v   (Standard Gas: 2 ml, 1 atm, 165 °C)



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Select Mode (GST) & Action (↑←→↓,End):              Run  26  = ——
G=Graph    S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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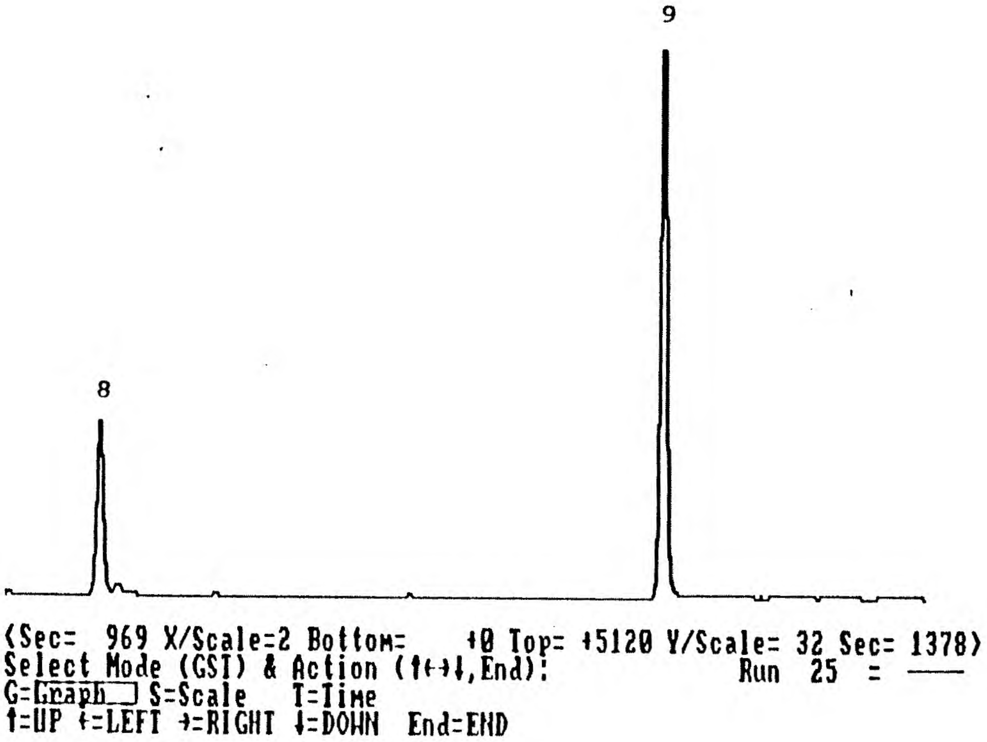

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G=Graph   S=Scale    T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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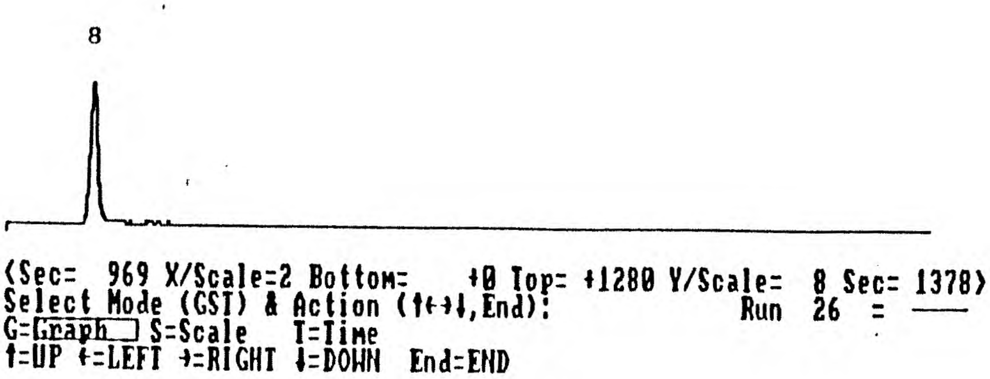
Figure 6 - 3 FID Response ^ / ------------- / ECD Response v (Standard Gas: 2 ml, 1 atm, 165 °C)



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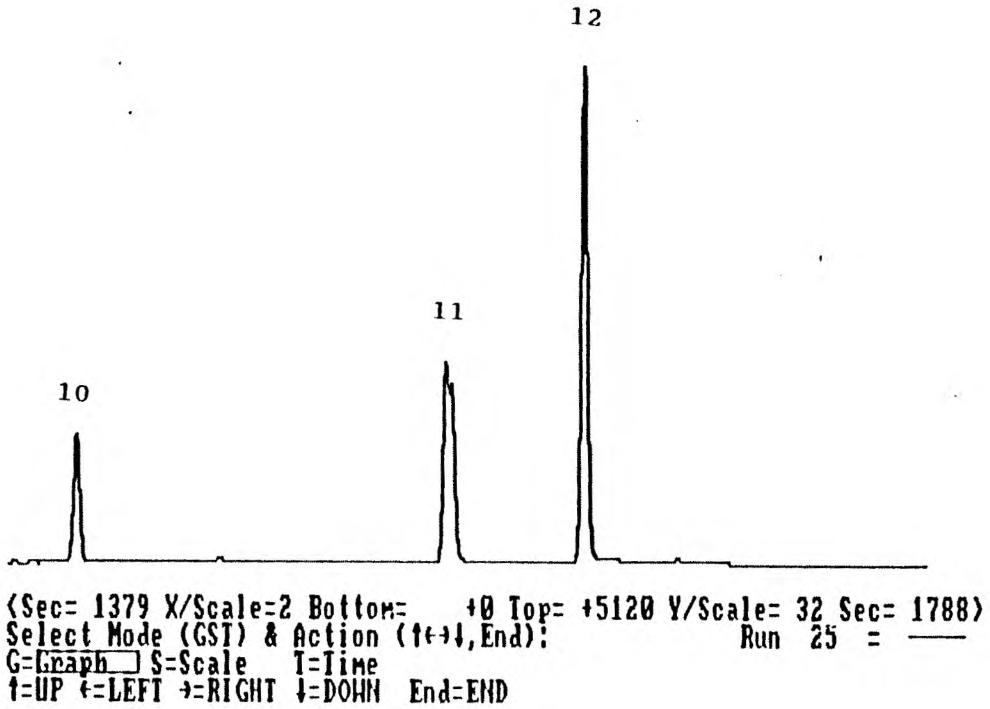
G=Graph   S=Scale    T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```



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G=Graph   S=Scale    T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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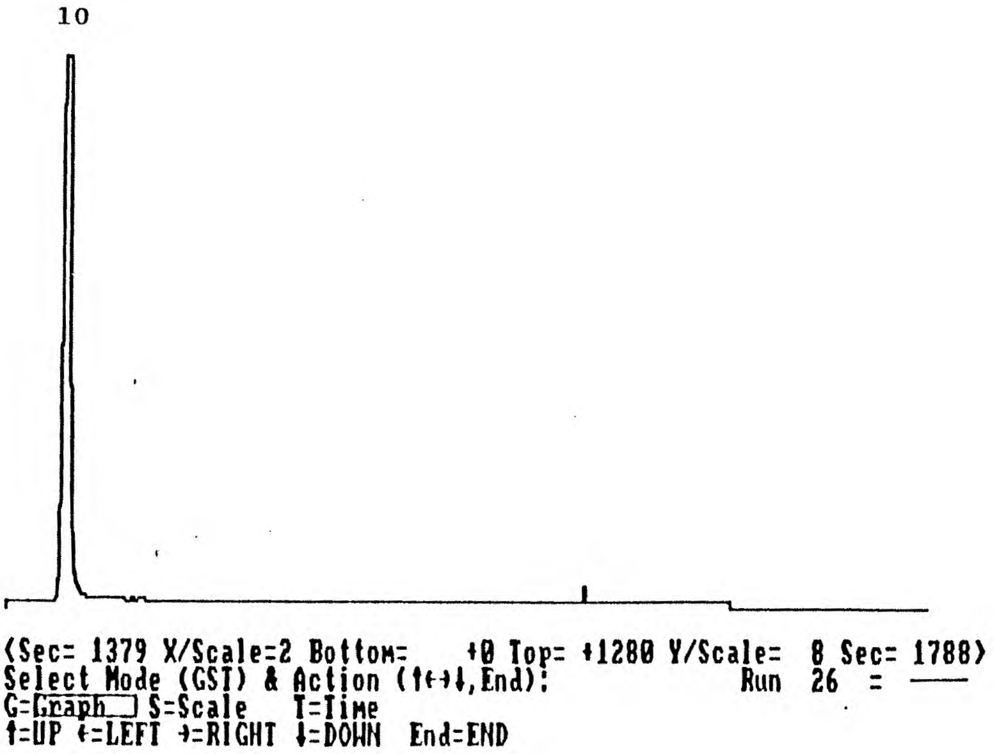
Figure 6 - 4 FID Response ^ / ------------- / ECD Response v (Standard Gas: 2 ml, 1 atm, 165 ^{O} C)



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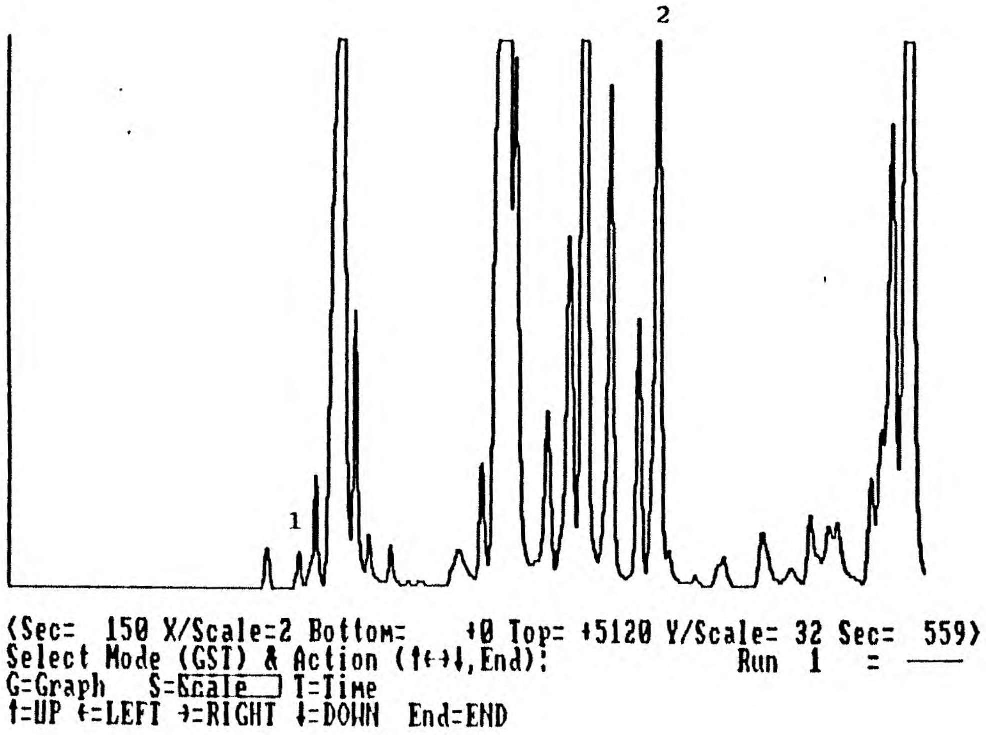

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↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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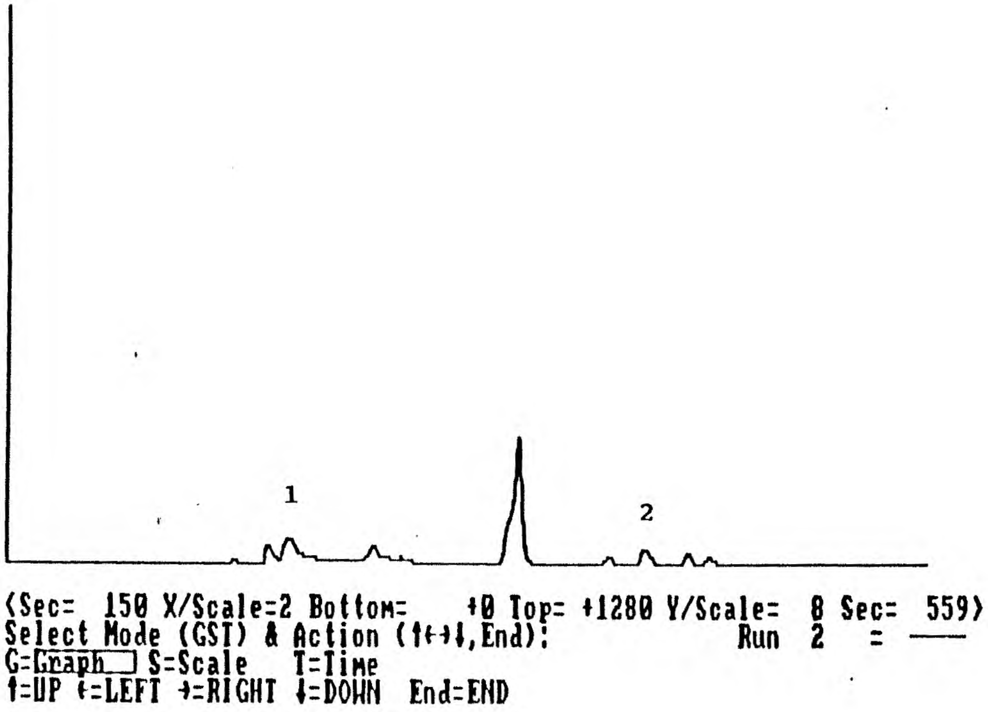
FID Response ^
Figure 7 - 1 -------------- (Spiked Air Sample)
ECD Response v



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↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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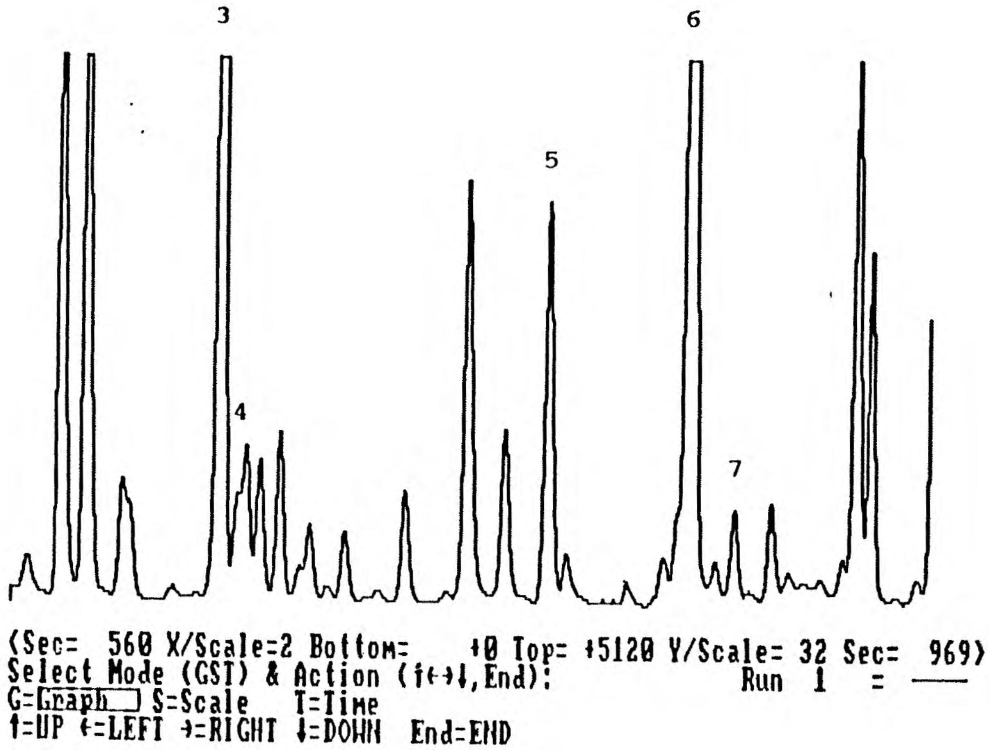

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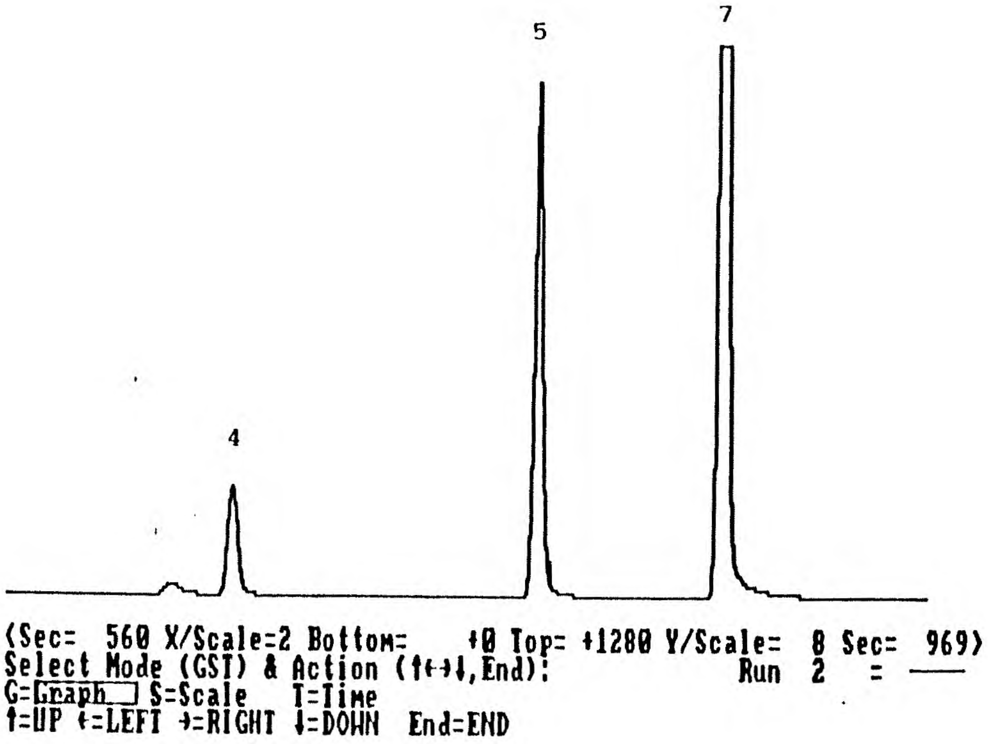
G=Graph   S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

Figure 7 - 2 FID Response ^

ECD Response v (Spiked Air Sample)



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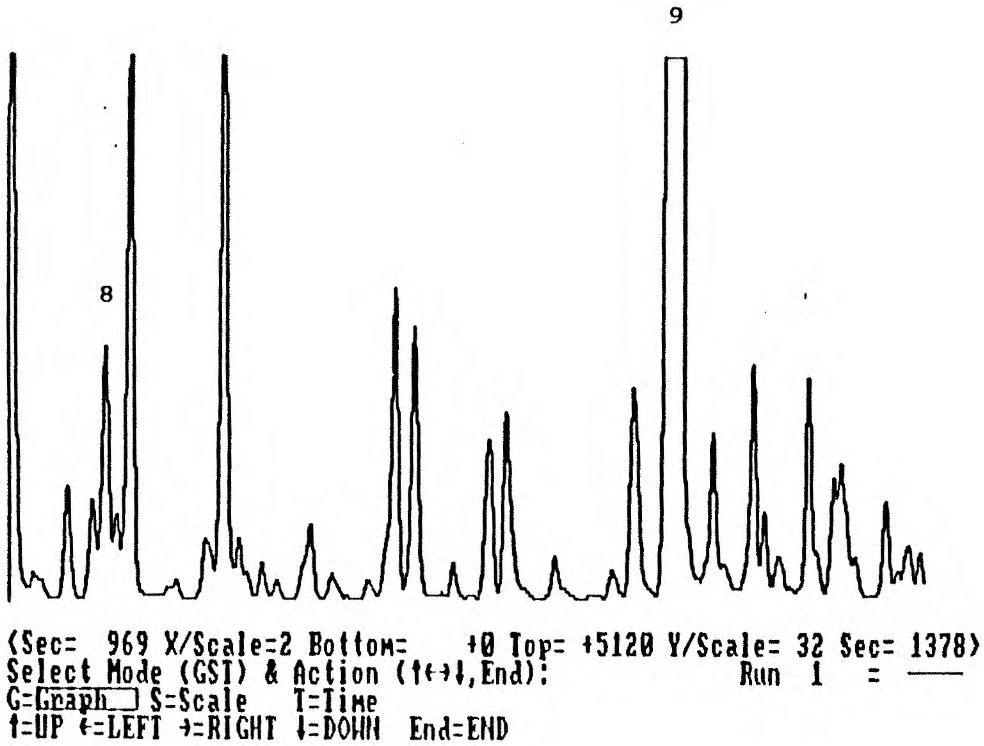
G=Graph   S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```



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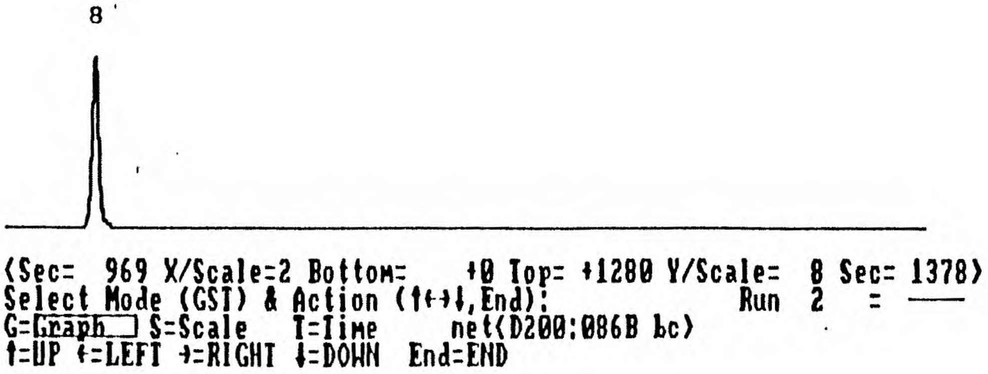
(Sec=  969 X/Scale=2 Bottom=    +0 Top= +5120 Y/Scale= 32 Sec= 1378)
Select Mode (GST) & Action (↑←→↓,End):            Run  1   = ——
G=Graph  S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

FID Response ^
Figure 7 - 3 -------------- (Spiked Air Sample)
ECD Response v



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(Sec=  969 X/Scale=2 Bottom=    +0 Top= +1280 Y/Scale=  8 Sec= 1378)
Select Mode (GST) & Action (↑←→↓,End):            Run  2   = ——
G=Graph  S=Scale   T=Time    net(D200:086B bc)
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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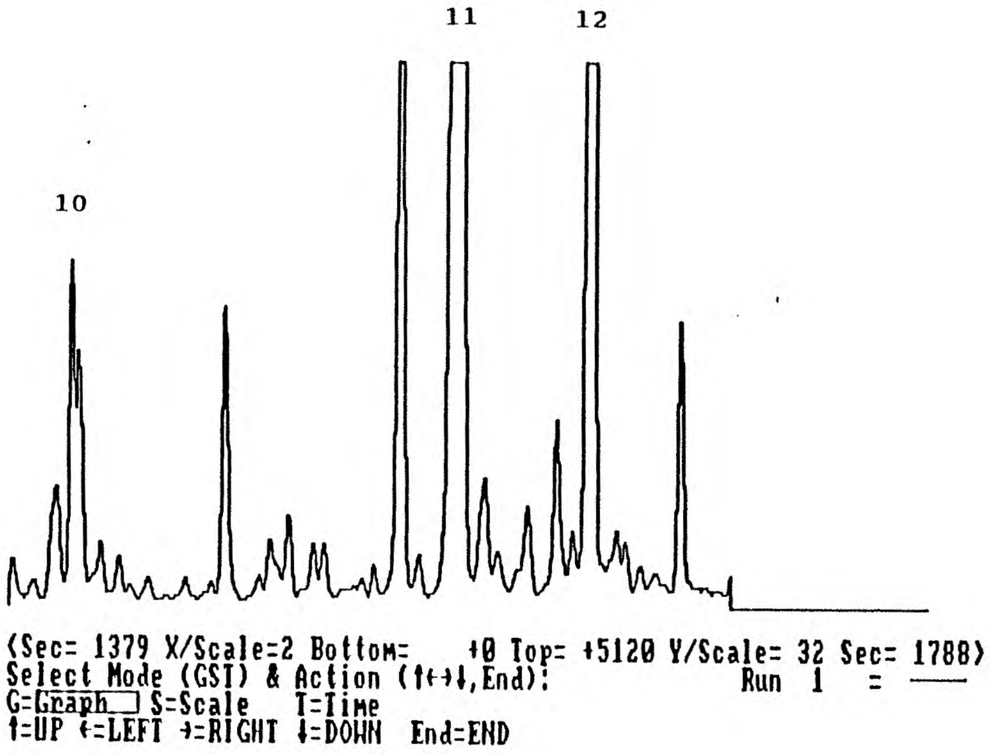

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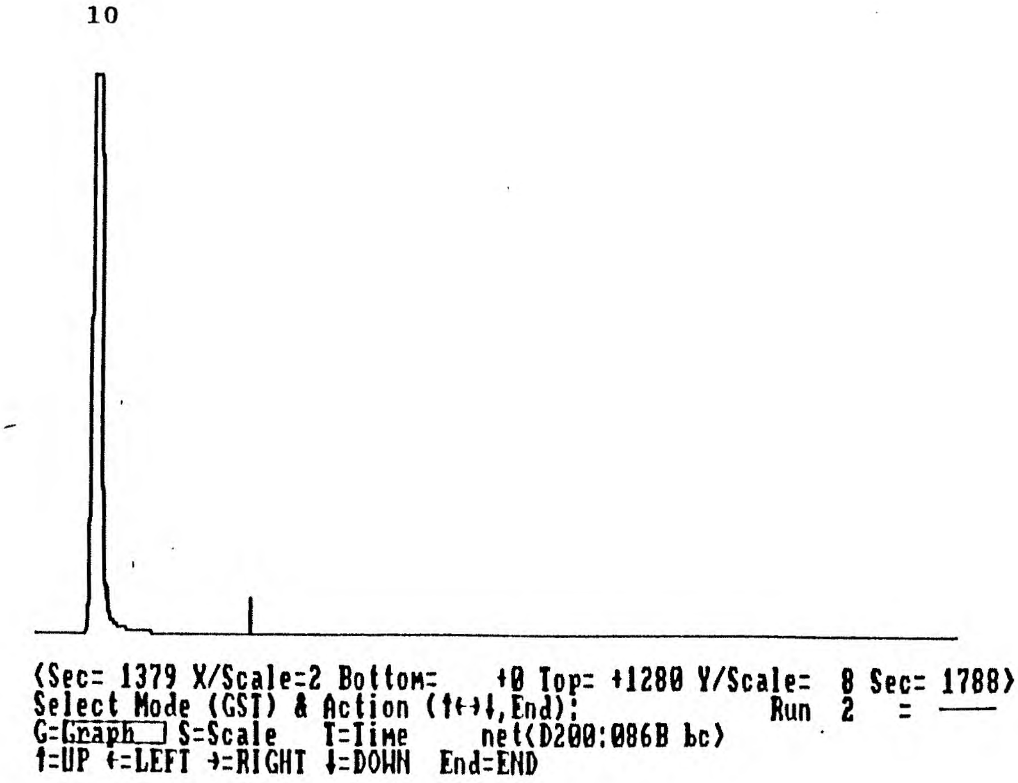
(Sec= 1379 X/Scale=2 Bottom=    +0 Top= +5120 Y/Scale= 32 Sec= 1788)
Select Mode (GST) & Action (↑←→↓,End):              Run  1   =  ——
G=Graph   S=Scale    T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

FID Response ^
Figure 7 - 4 ------------- (Spiked Air Sample)
ECD Response v



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(Sec= 1379 X/Scale=2 Bottom=    +0 Top= +1280 Y/Scale=  8 Sec= 1788)
Select Mode (GST) & Action (↑←→↓,End):              Run  2   =  ——
G=Graph   S=Scale    T=Time      net(D200:086B bc)
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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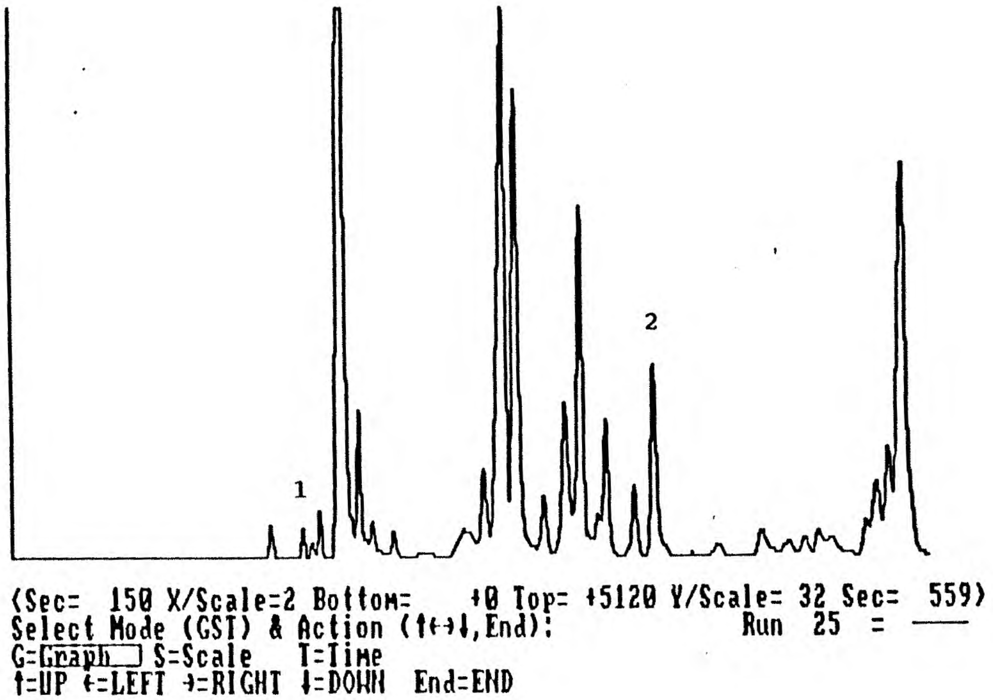

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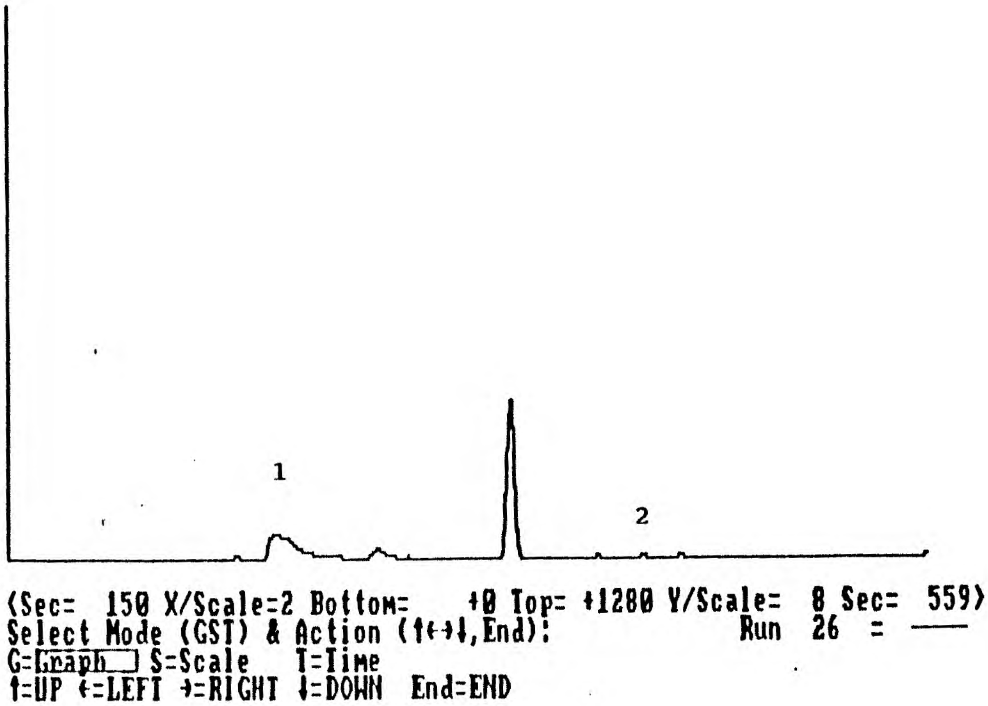
(Sec=  150 X/Scale=2 Bottom=    +0 Top= +5120 Y/Scale= 32 Sec=  559)
Select Mode (GST) & Action (↑←→↓,End):             Run  25  = ——
G=Graph  S=Scale    T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

FID Response ^
Figure 8 - 1 ------------- (Air Sample)
ECD Response v



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Select Mode (GST) & Action (↑←→↓,End):             Run  26  = ——
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↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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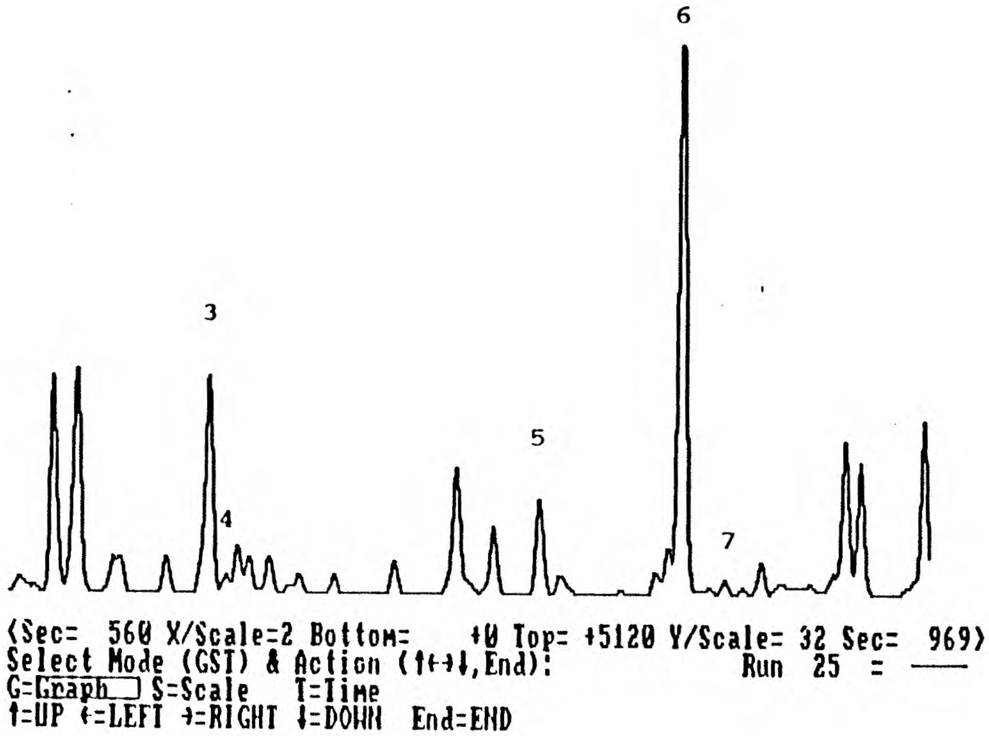

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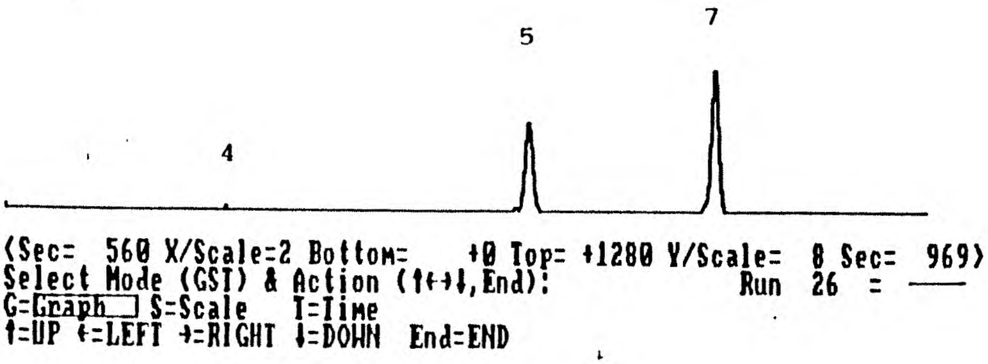
Select Mode (GST) & Action (↑←→↓,End):                    Run  25  =  ——
G=Graph    S=Scale    T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

```
                      FID Response ^
Figure 8  -  2        -------------     ( Air Sample )
                      ECD Response v
```



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Select Mode (GST) & Action (↑←→↓,End):                    Run  26  =  ——
G=Graph    S=Scale    T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
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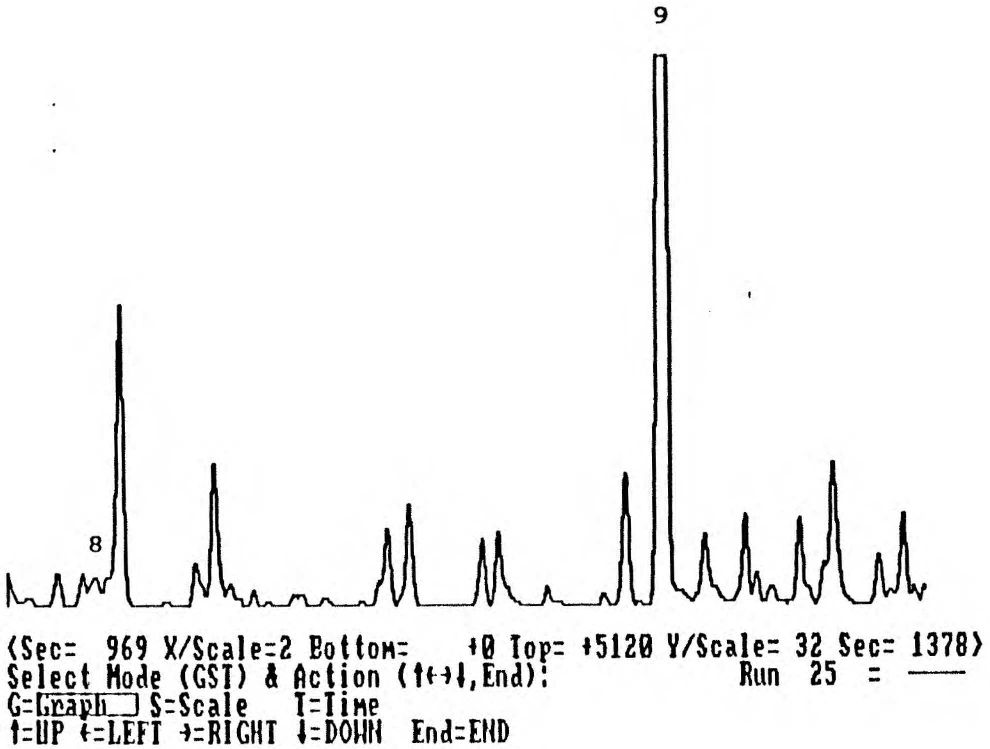

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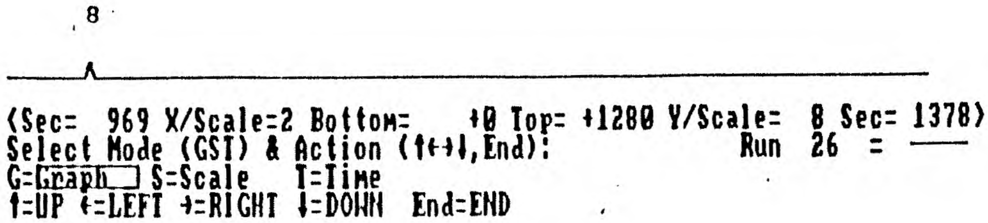
<Sec=  969 X/Scale=2 Bottom=    +0 Top= +5120 Y/Scale= 32 Sec= 1378>
Select Mode (GST) & Action (↑←→↓,End):               Run  25  = ——
G=Graph  S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

Figure 8 - 3   FID Response ^
                -------------   (Air Sample)
                ECD Response v



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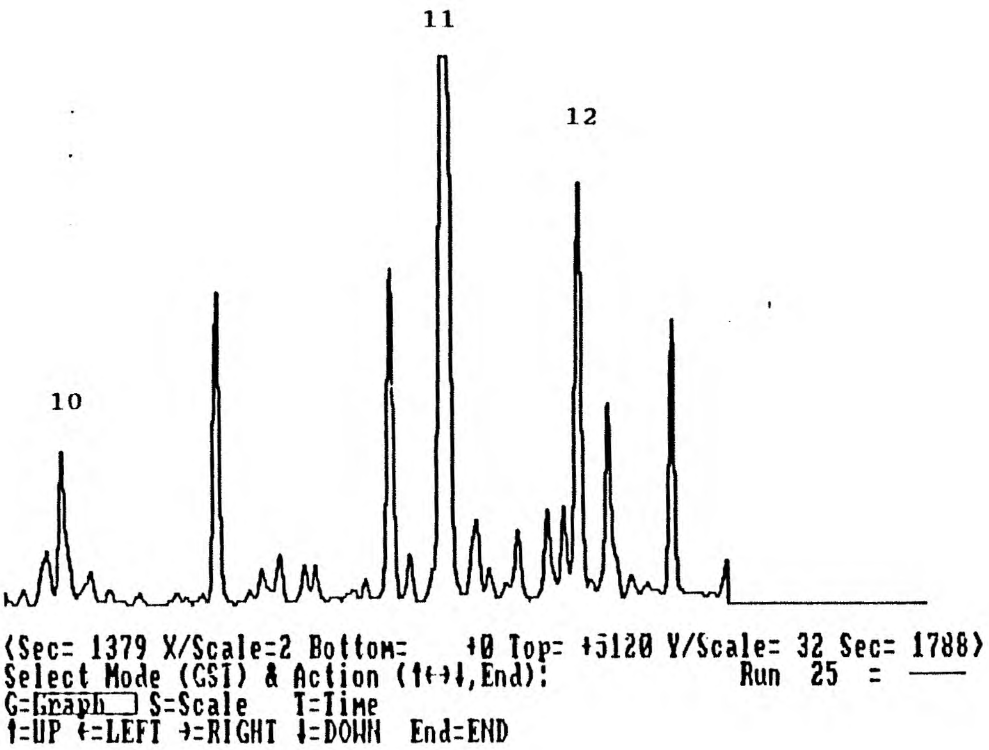
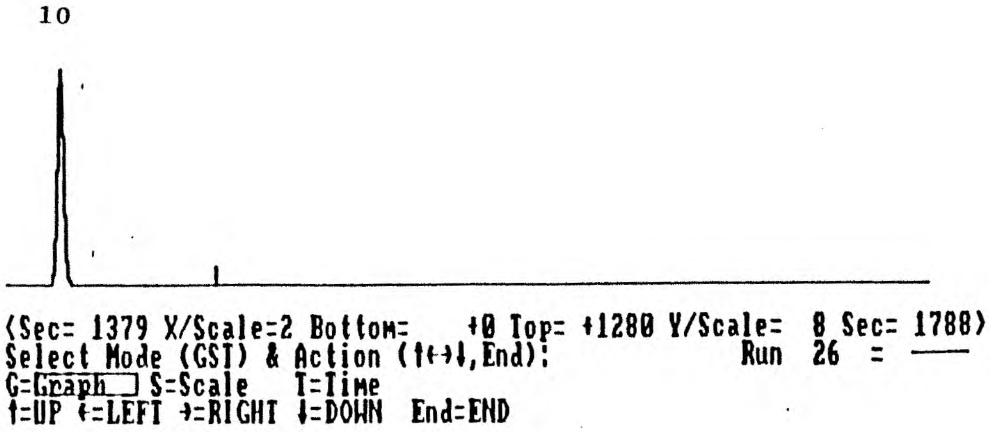
Select Mode (GST) & Action (↑←→↓,End):               Run  26  = ——
G=Graph  S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```



```
(Sec= 1379 X/Scale=2 Bottom=    +0 Top= +5120 Y/Scale= 32 Sec= 1788)
Select Mode (GST) & Action (↑←→↓,End):              Run  25  = ——
G=Graph   S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

Figure 8 - 4 $\frac{\text{FID Response } \hat{}}{\text{ECD Response v}}$ (Air Sample)



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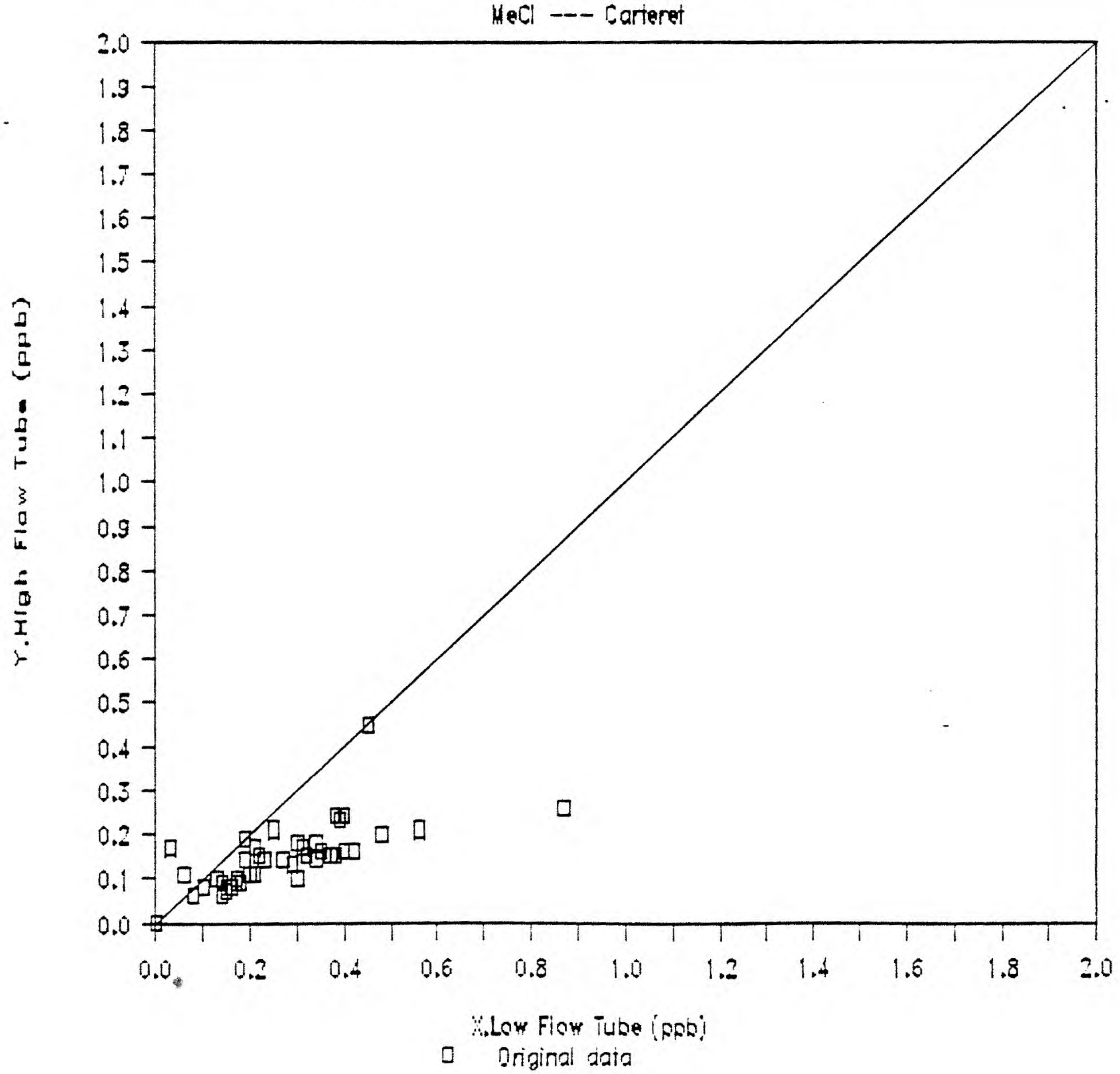
(Sec= 1379 X/Scale=2 Bottom=    +0 Top= +1280 Y/Scale=  8 Sec= 1788)
Select Mode (GST) & Action (↑←→↓,End):              Run  26  = ——
G=Graph   S=Scale   T=Time
↑=UP ←=LEFT →=RIGHT ↓=DOWN  End=END
```

Figure 9 - 1 Correlation Between Hi & Lo Flow Tubes



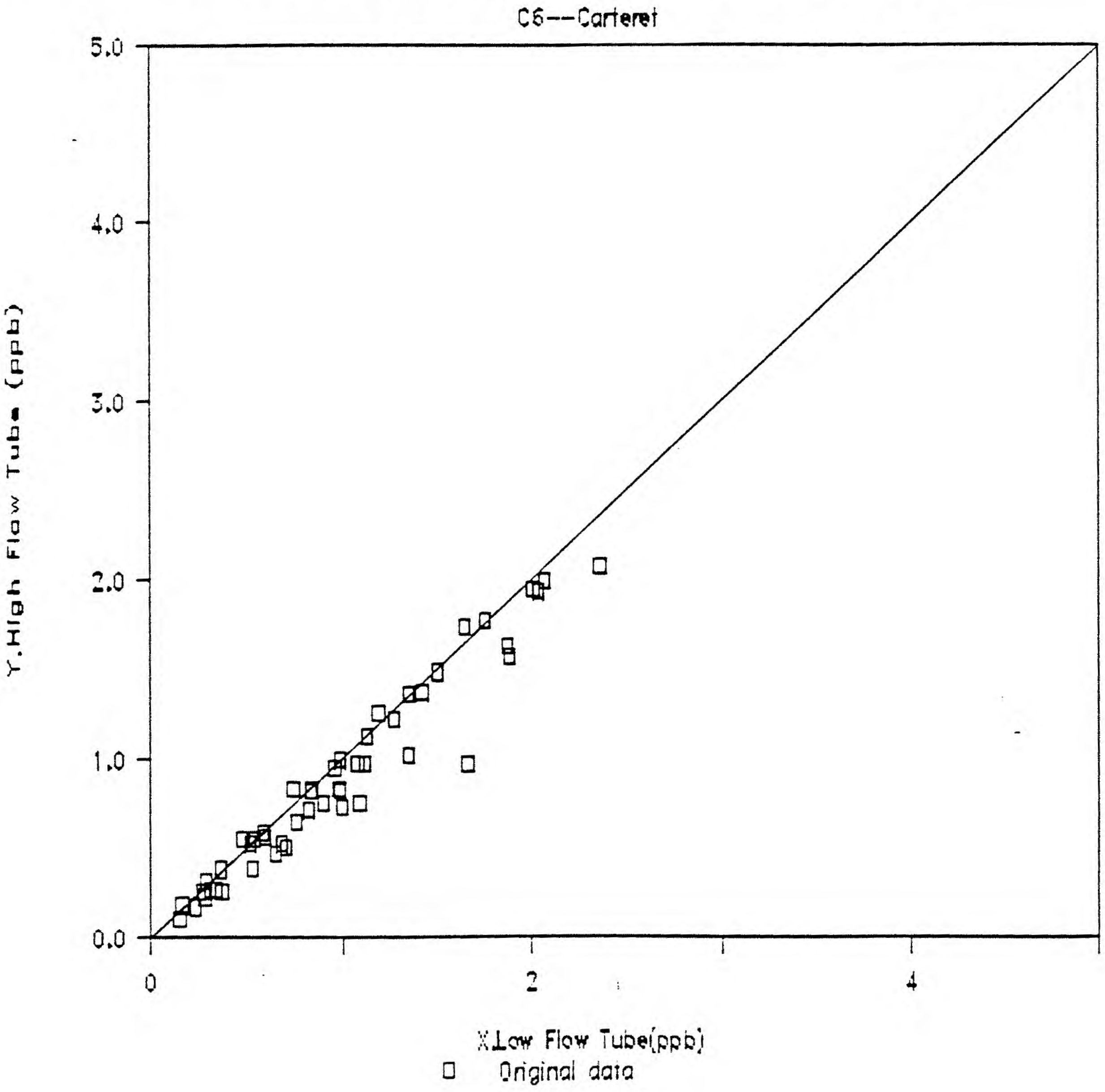


Figure 9 - 2 Correlation Between Hi & Lo Flow Tubes

Figure 9 - 3 **Correlation Between Hi & Lo Flow Tubes**

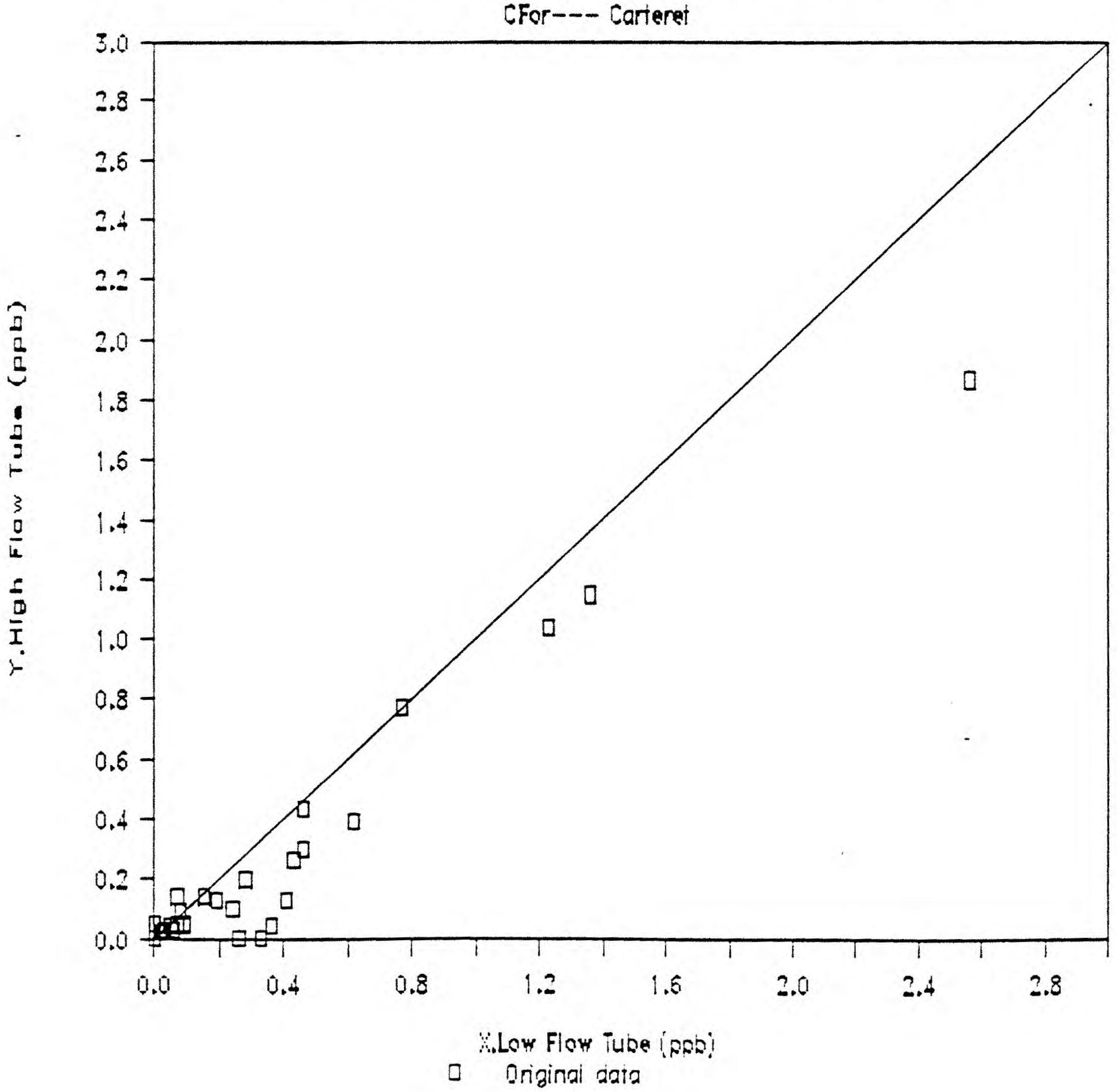
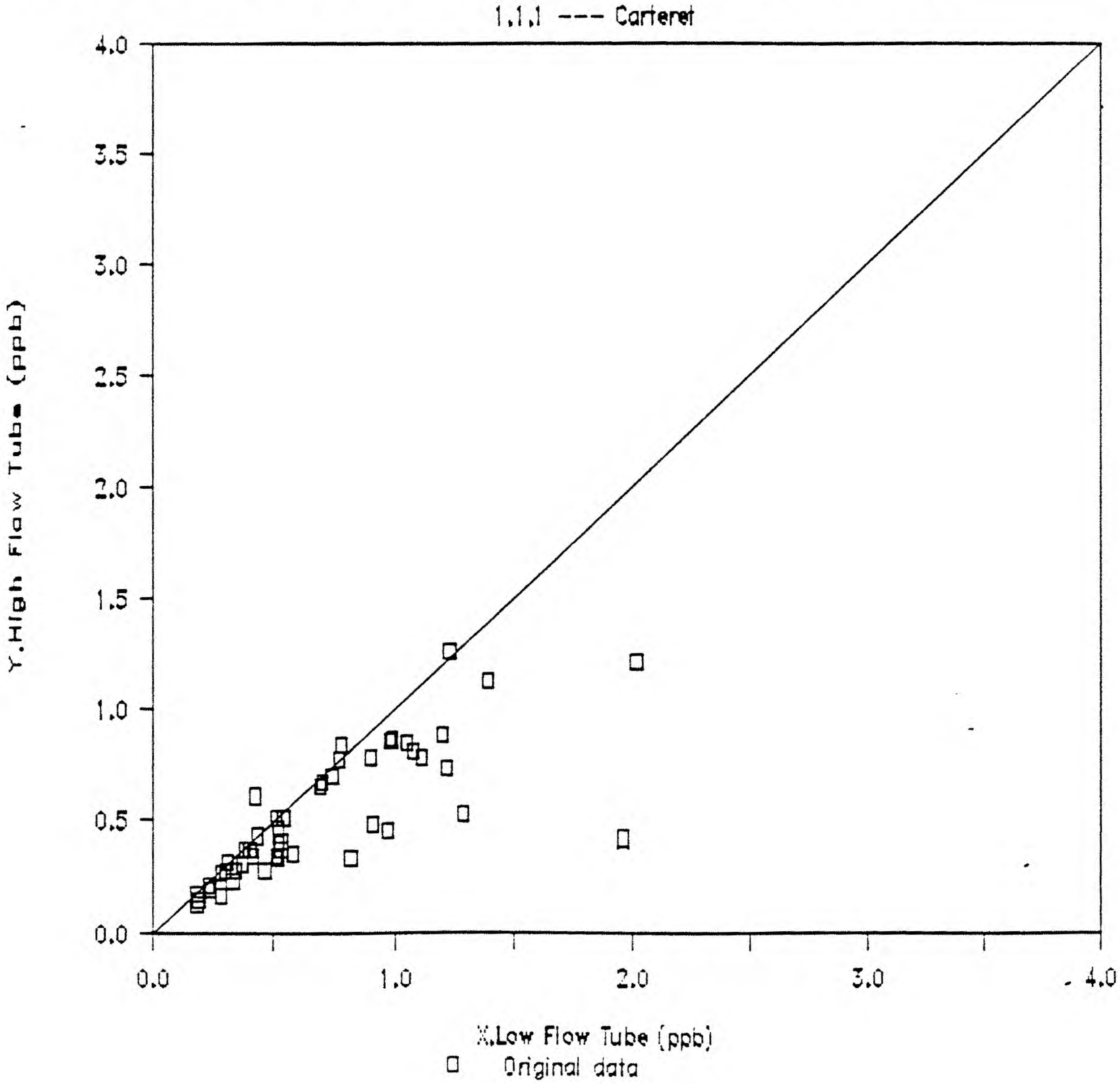


Figure 9 - 4 **Correlation Between Hi & Lo Flow Tubes**



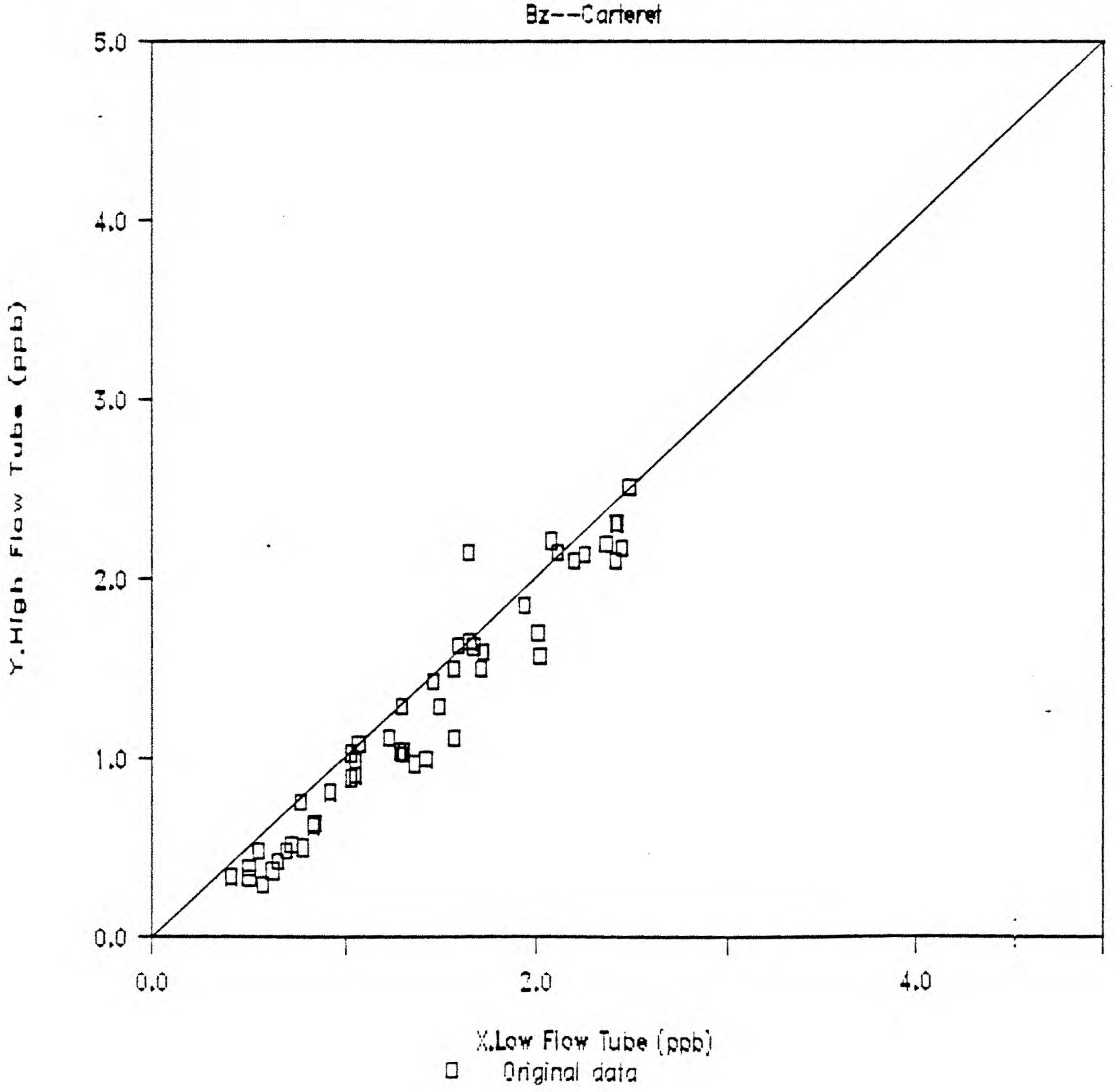


Figure 9 - 5 Correlation Between Hi & Lo Flow Tubes

Figure 9 - 6 **Correlation Between Hi & Lo Flow Tubes**

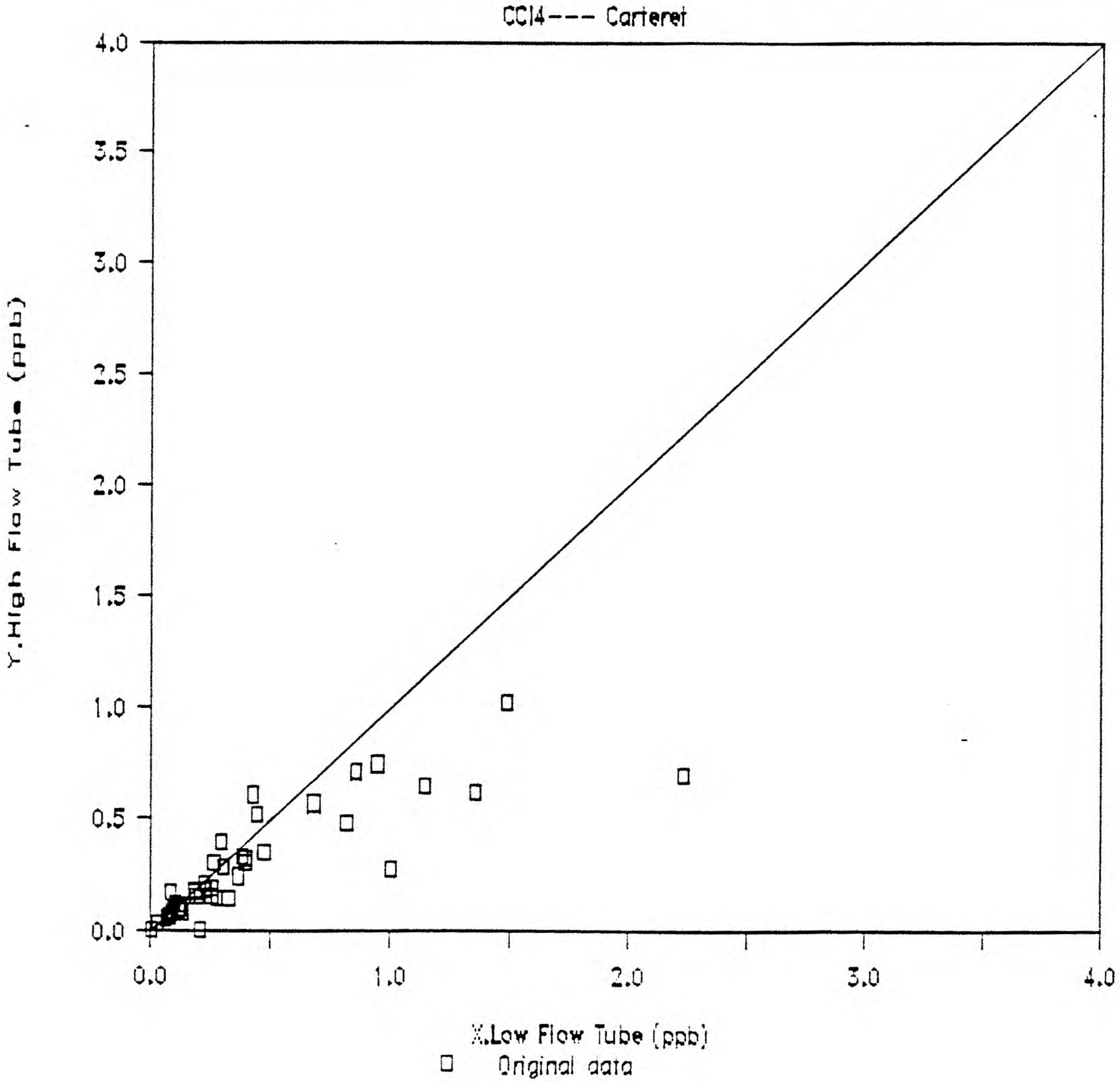


Figure 9 - 7 **Correlation Between Hi & Lo Flow Tubes**

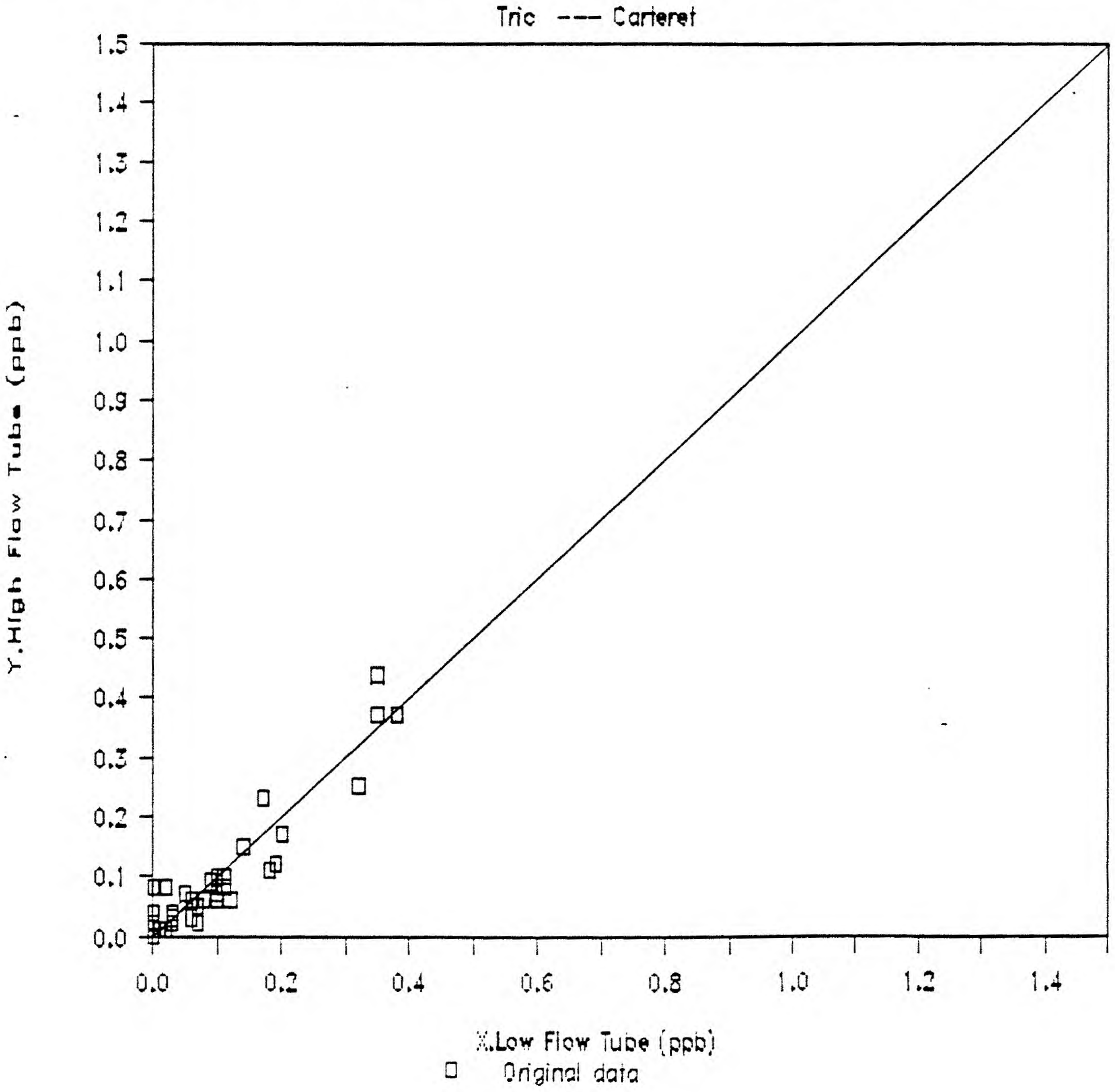


Figure 9 - 8 **Correlation Between Hi & Lo Flow Tubes**

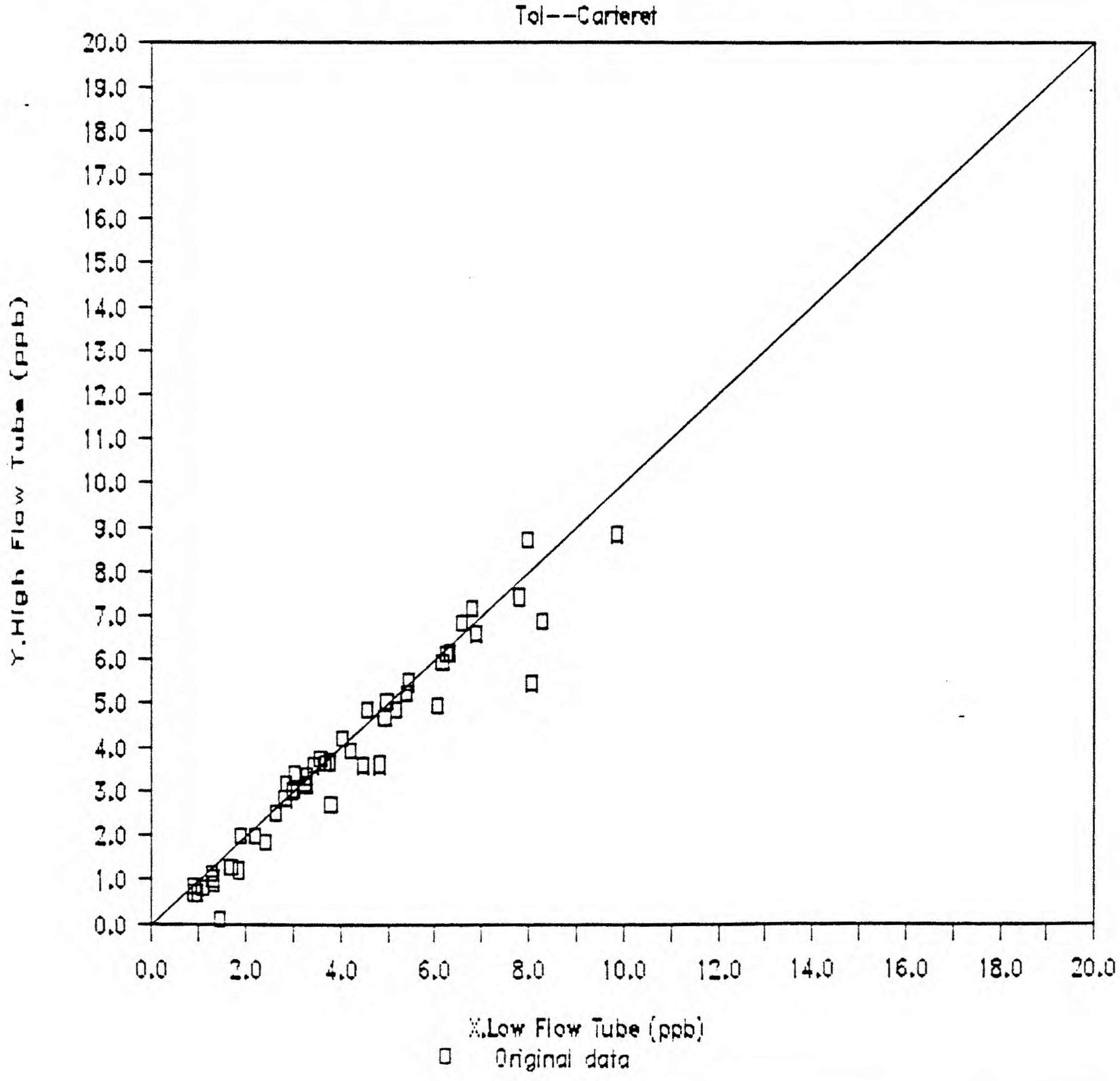


Figure 9 - 9 Correlation Between Hi & Lo Flow Tubes

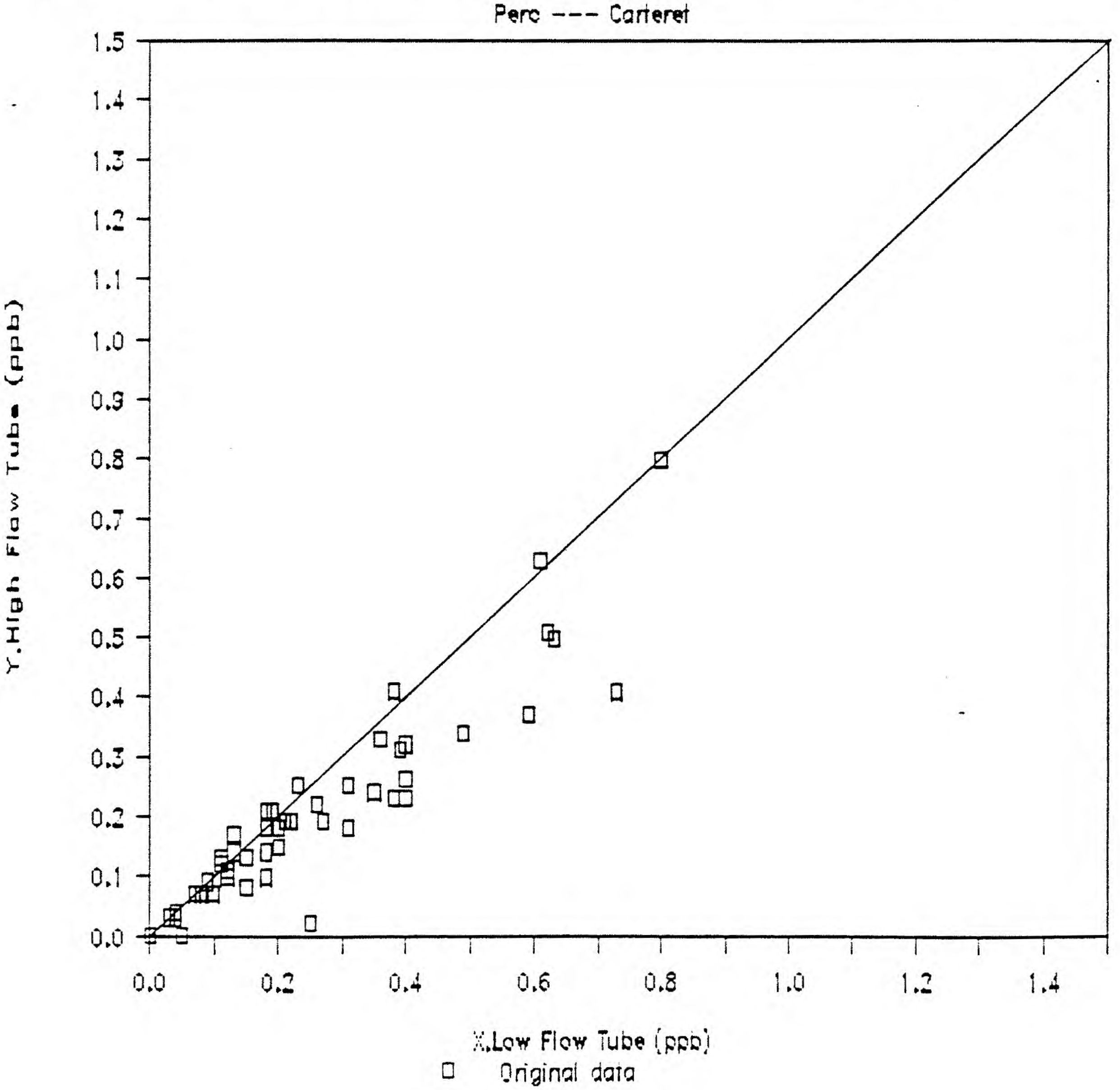


Figure 9 - 10 Correlation Between Hi & Lo Flow Tubes

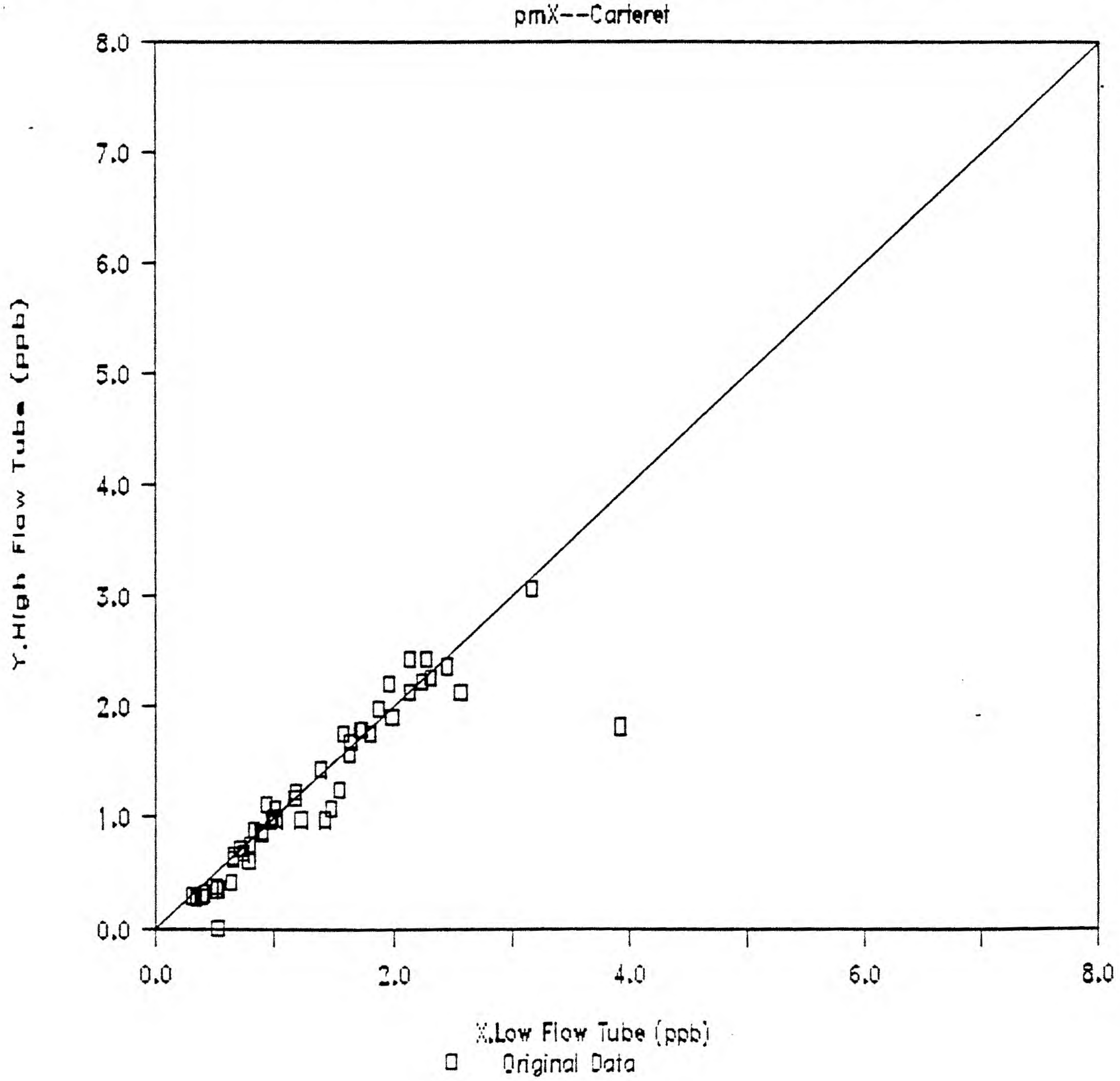


Figure 9 - 11 Correlation Between Hi & Lo Flow Tubes

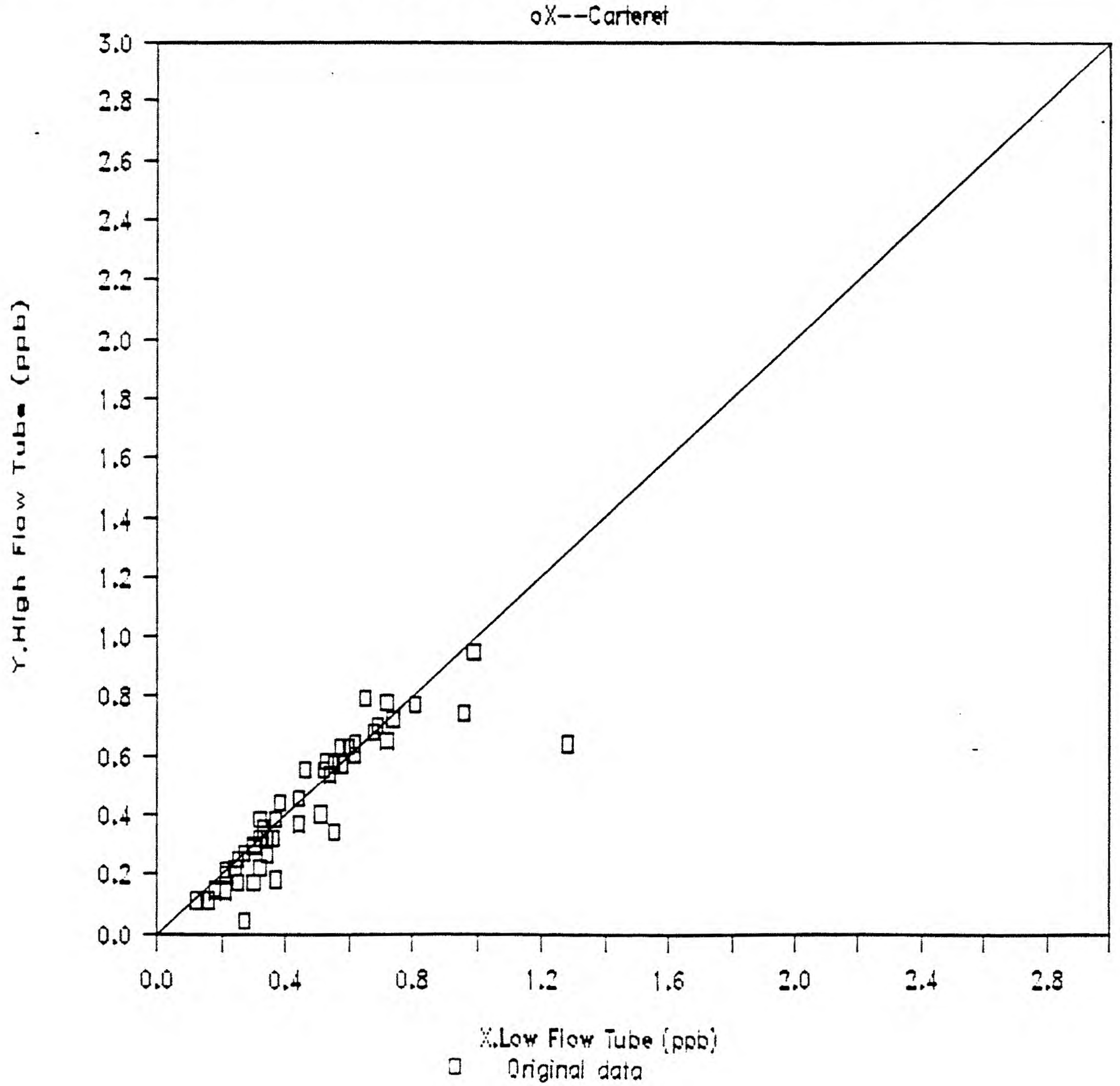


Figure 9 - 12 **Correlation Between Hi & Lo Flow Tubes**

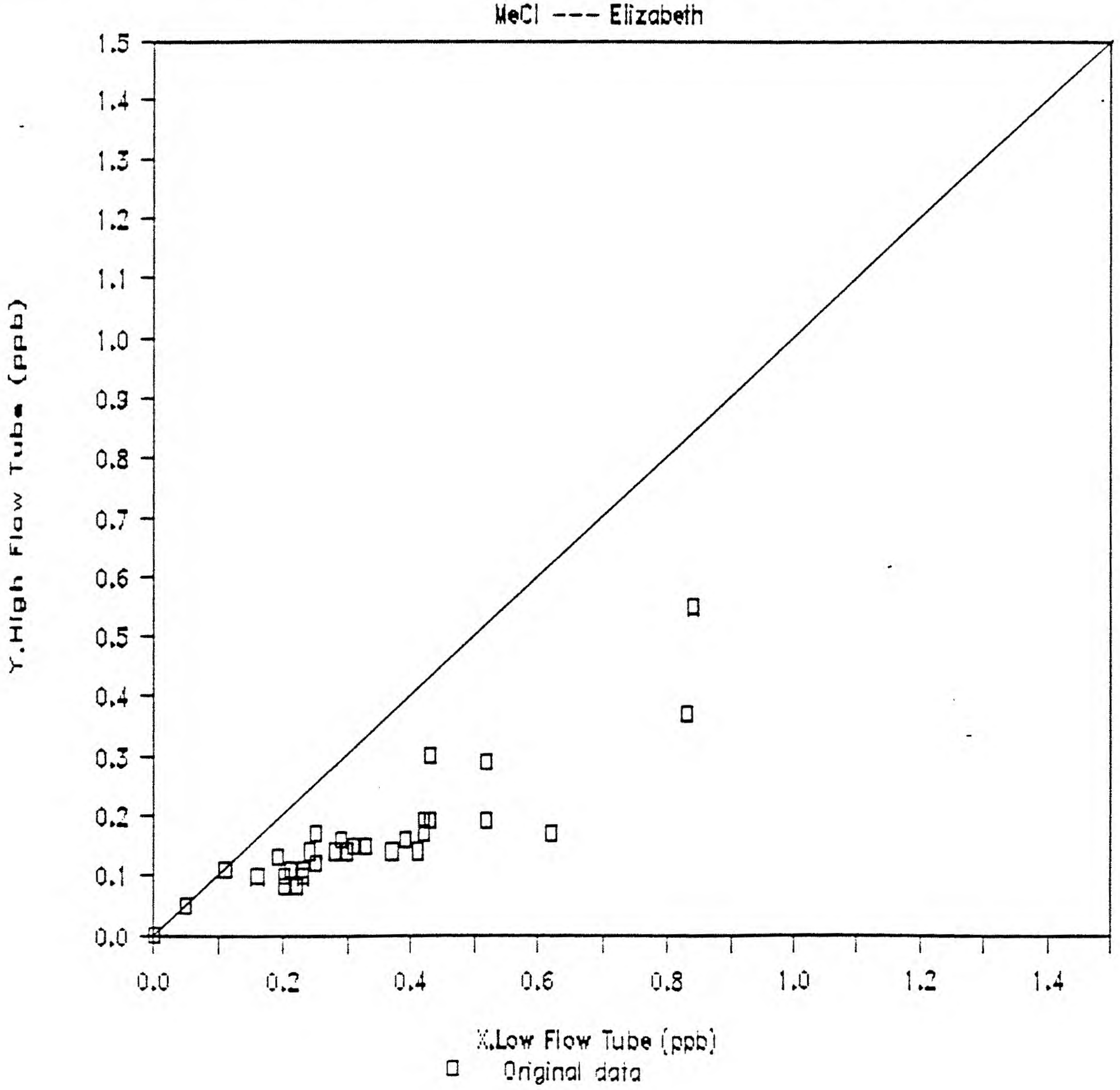


Figure 9 - 13 Correlation Between Hi & Lo Flow Tubes

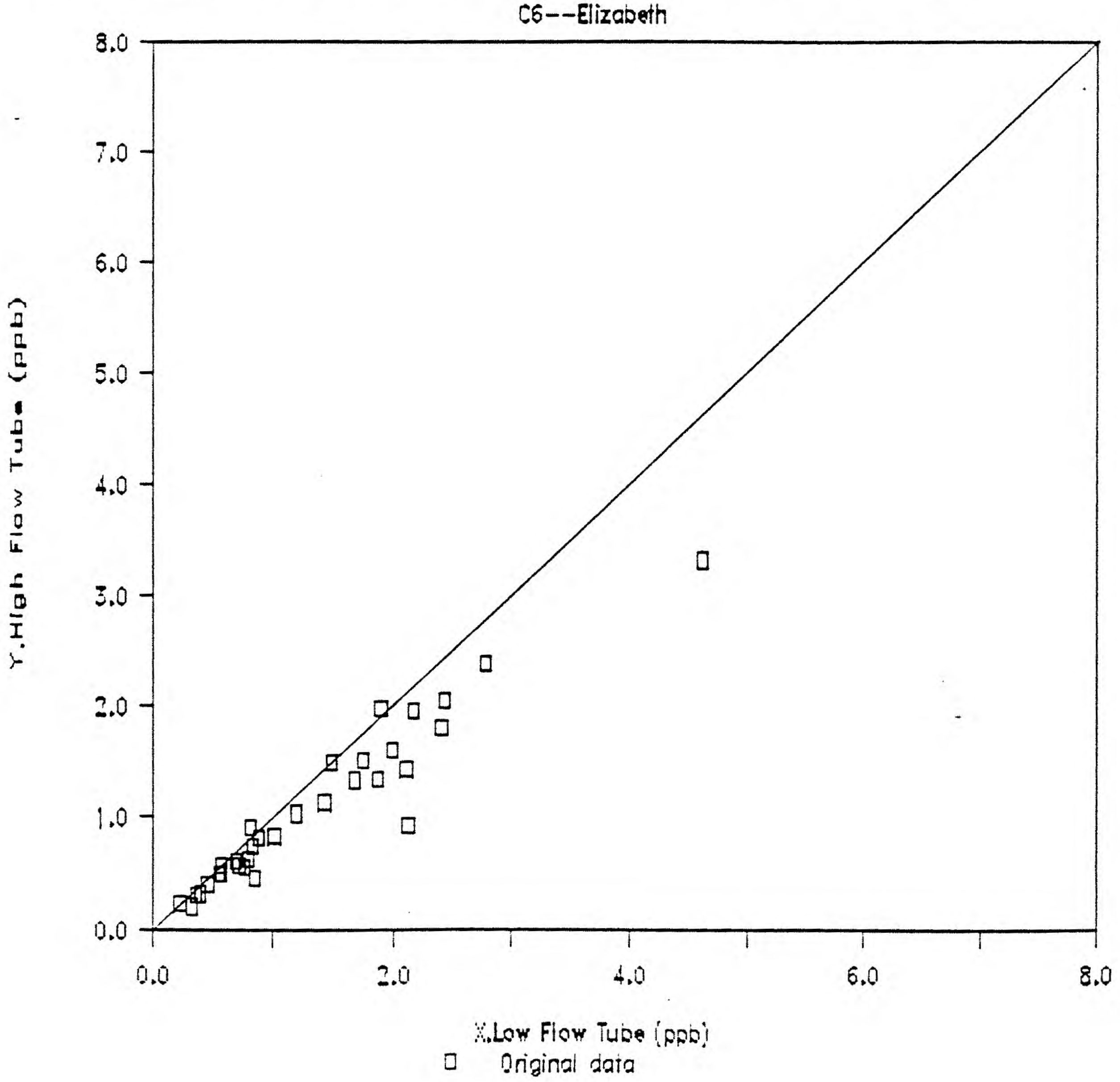


Figure 9 - 14 Correlation Between Hi & Lo Flow Tubes

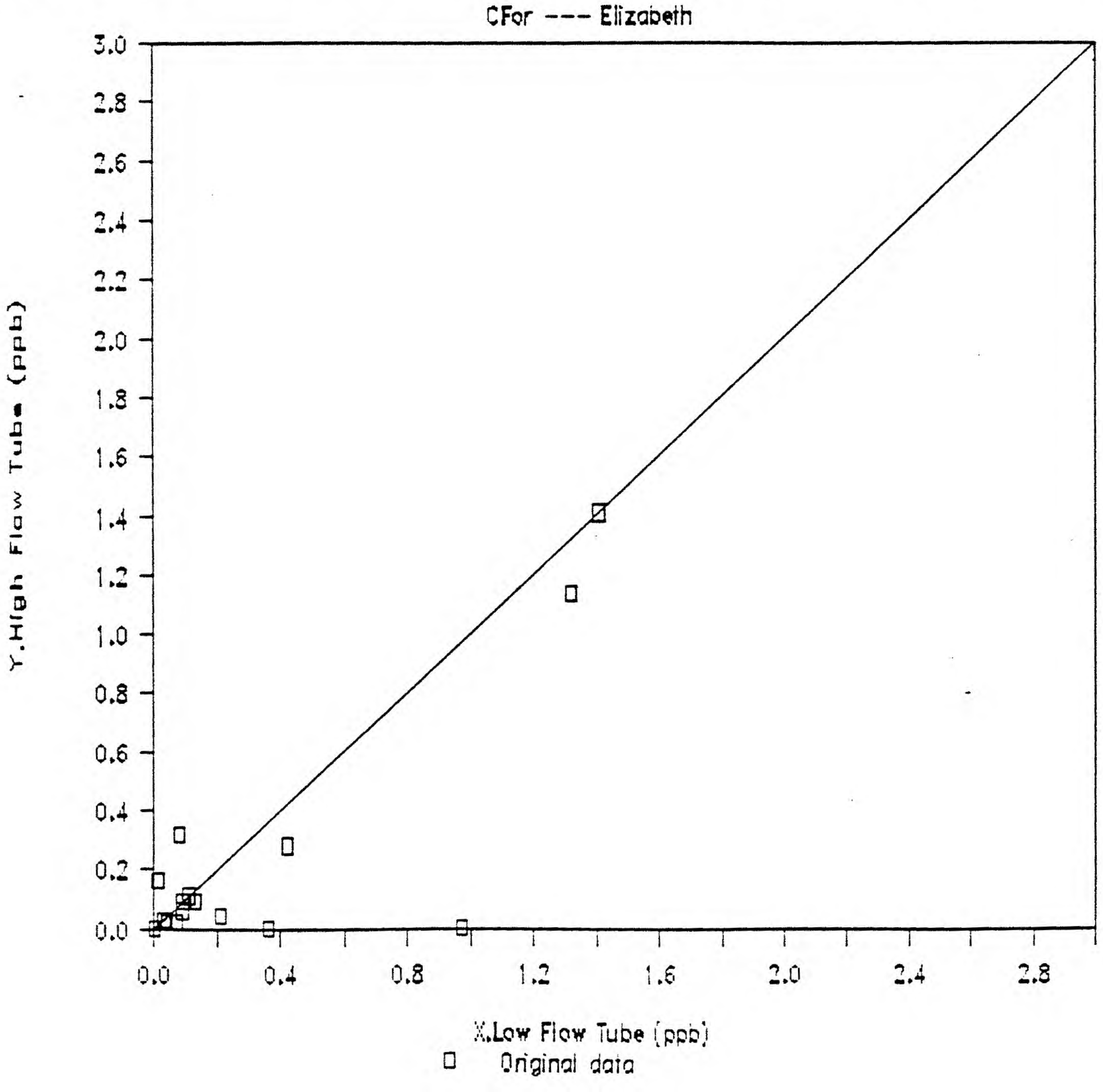


Figure 9 - 15 Correlation Between Hi & Lo Flow Tubes

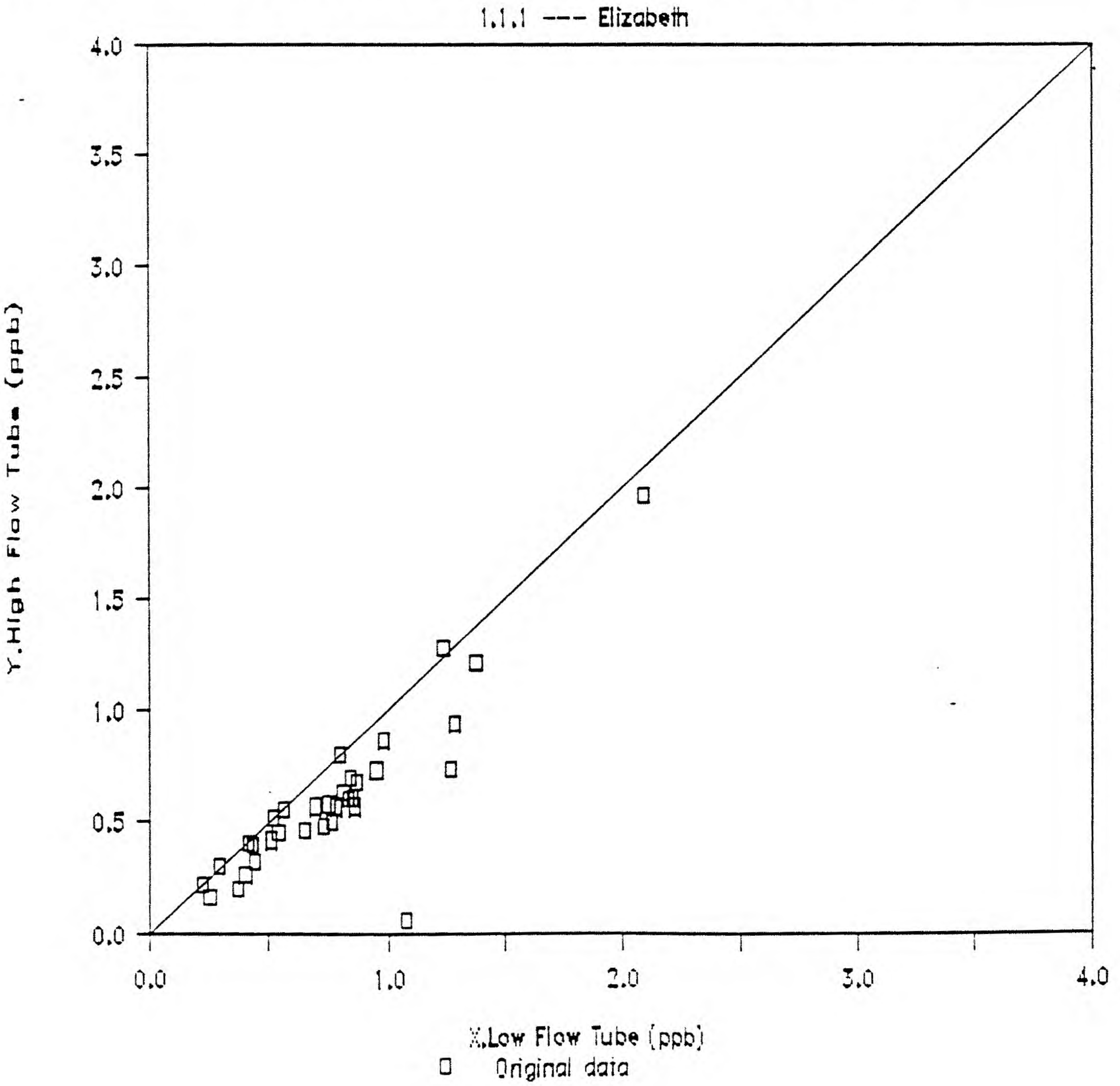


Figure 9 - 16 Correlation Between Hi & Lo Flow Tubes

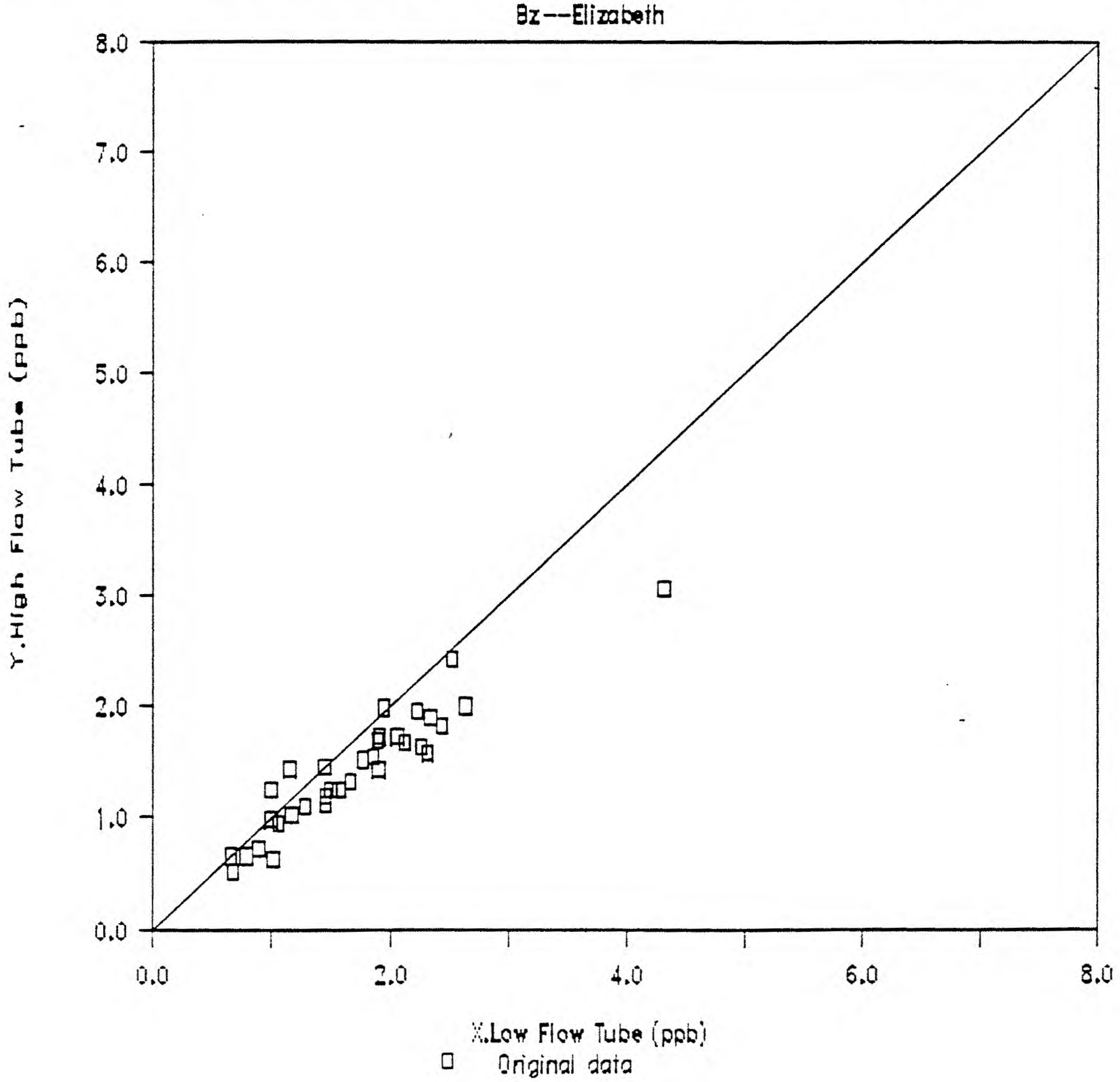


Figure 9 - 17 Correlation Between Hi & Lo Flow Tubes

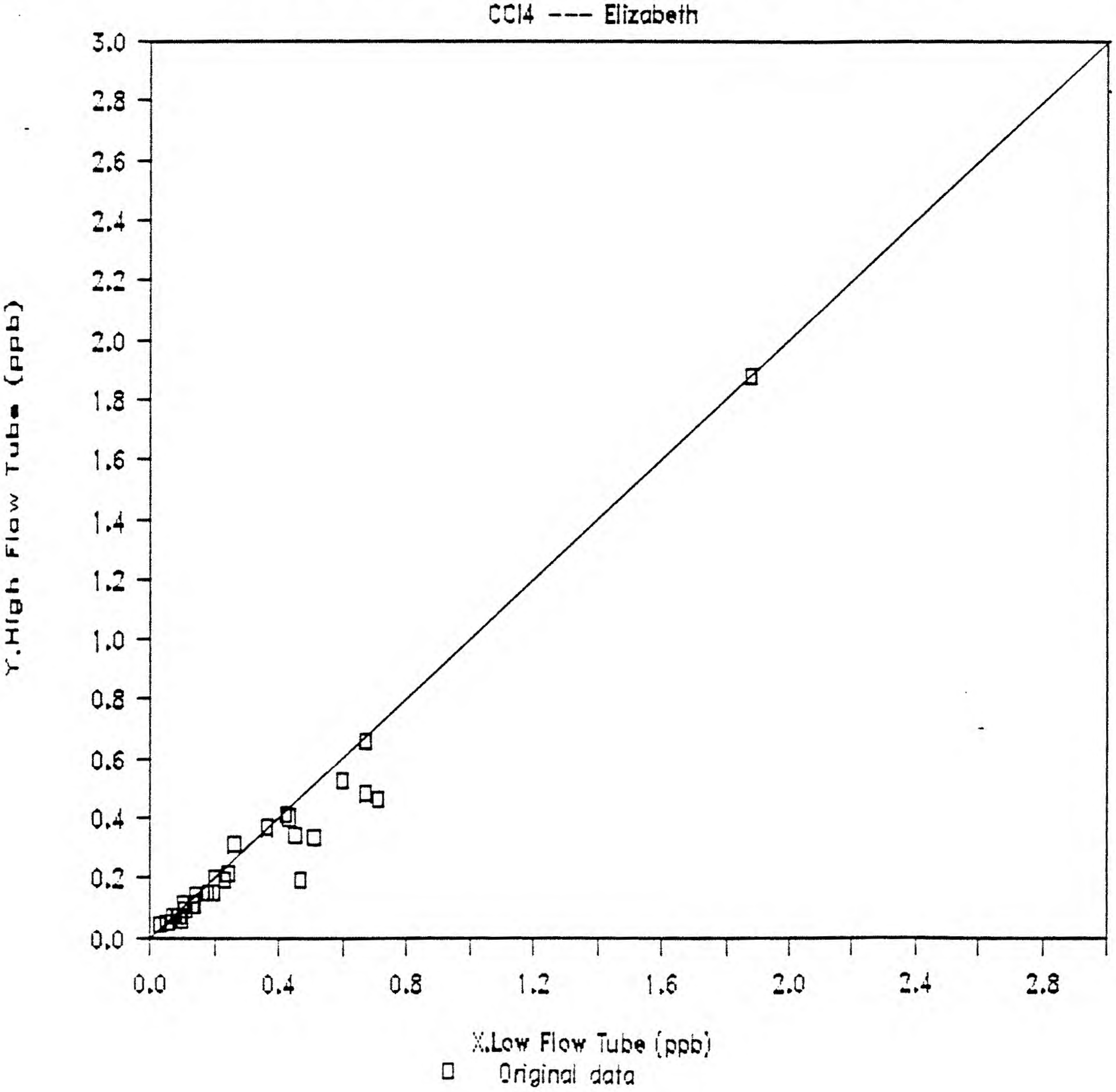


Figure 9 - 18 Correlation Between Hi & Lo Flow Tubes

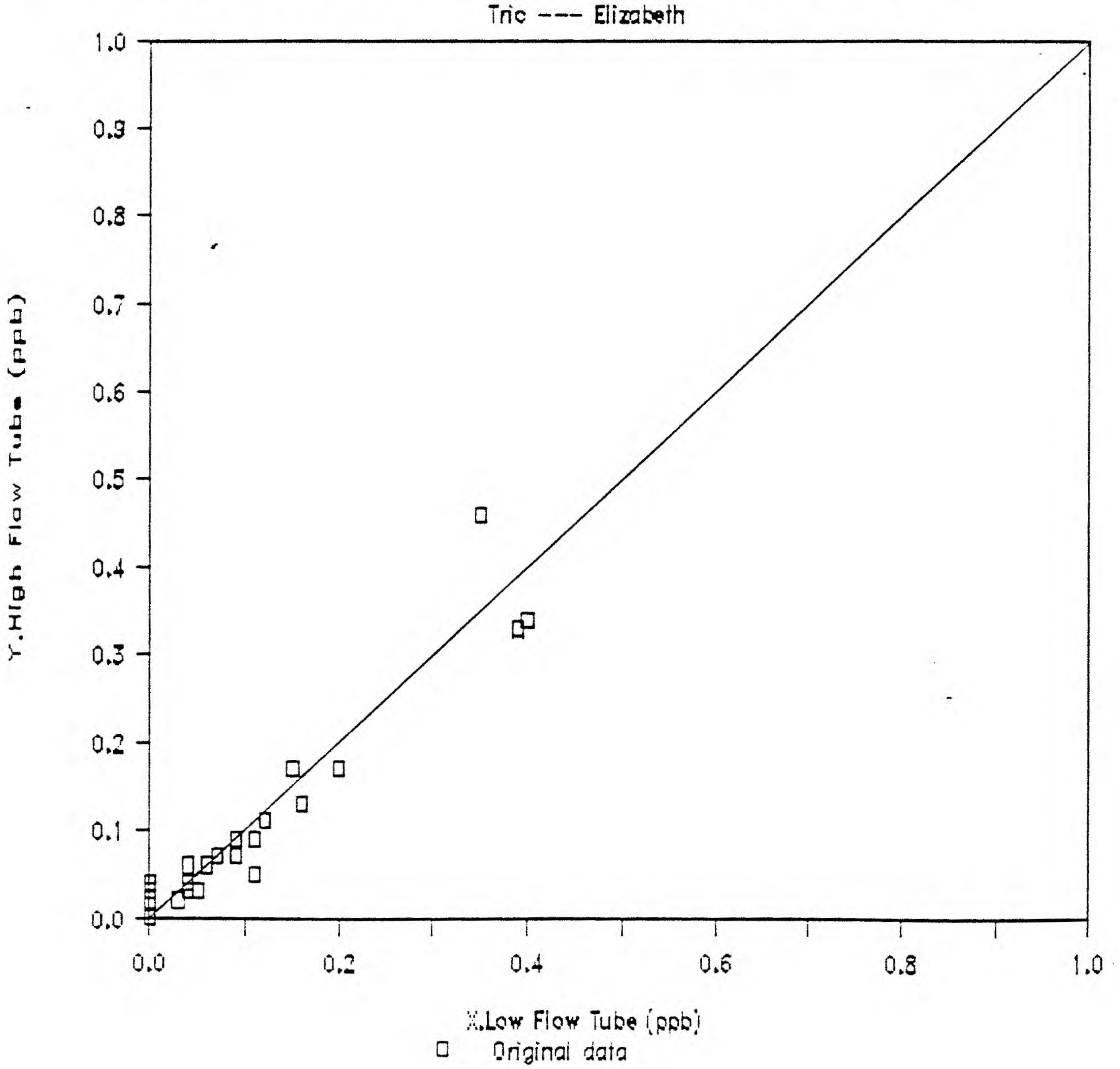


Figure 9 - 19 **Correlation Between Hi & Lo Flow Tubes**

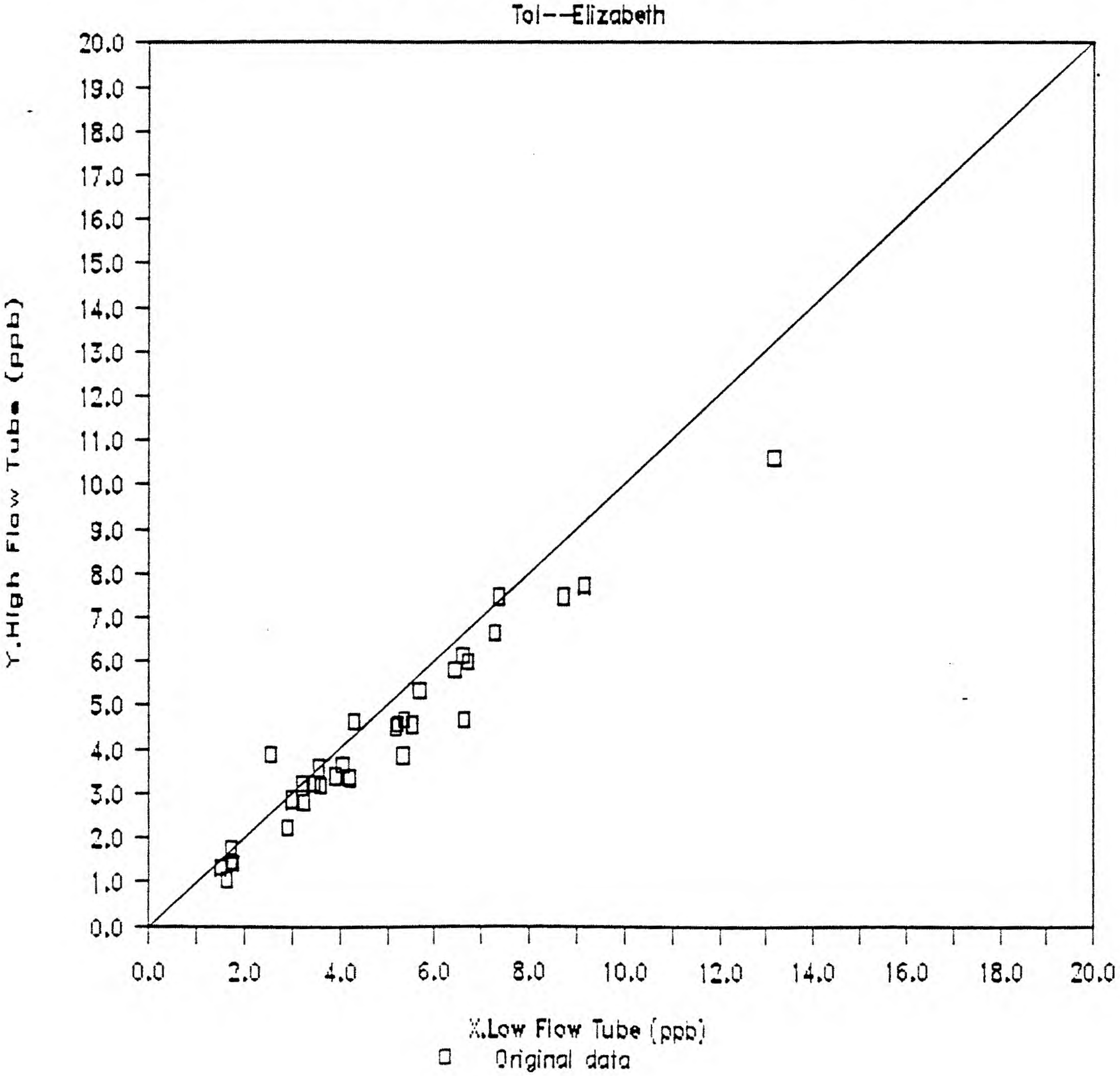


Figure 9 - 20 **Correlation Between Hi & Lo Flow Tubes**

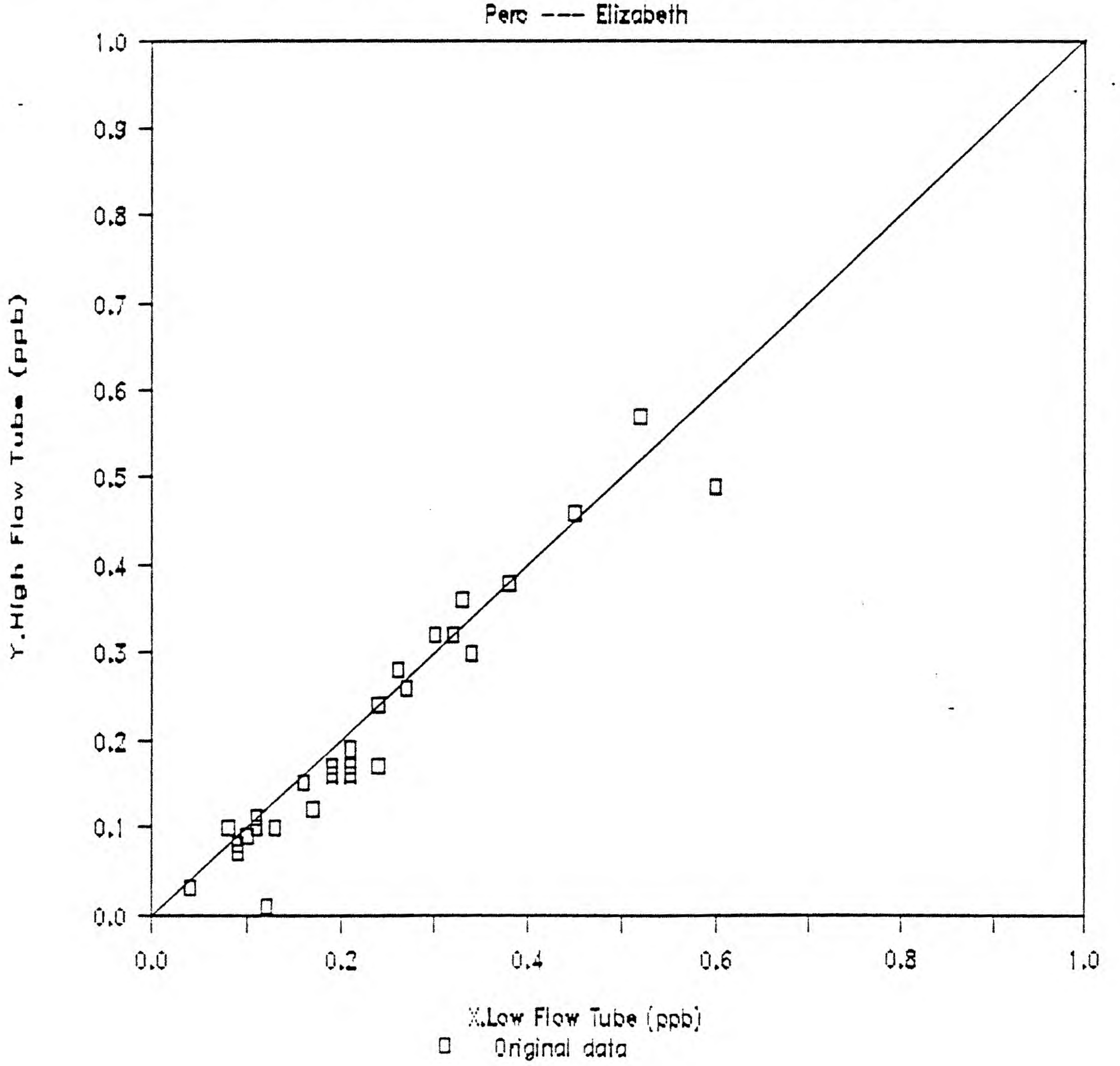


Figure 9 - 21 Correlation Between Hi & Lo Flow Tubes

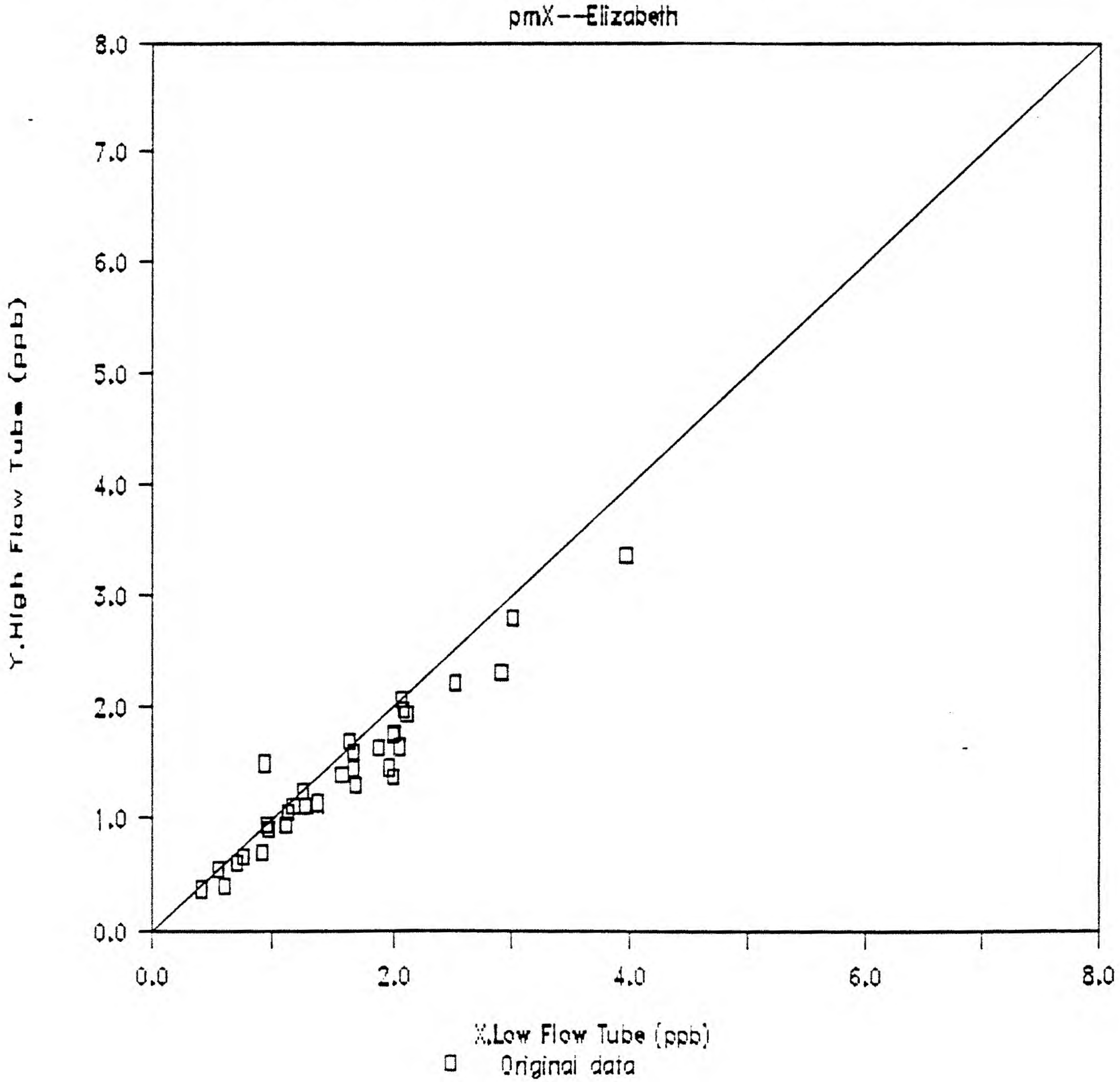


Figure 9 - 22 **Correlation Between Hi & Lo Flow Tubes**

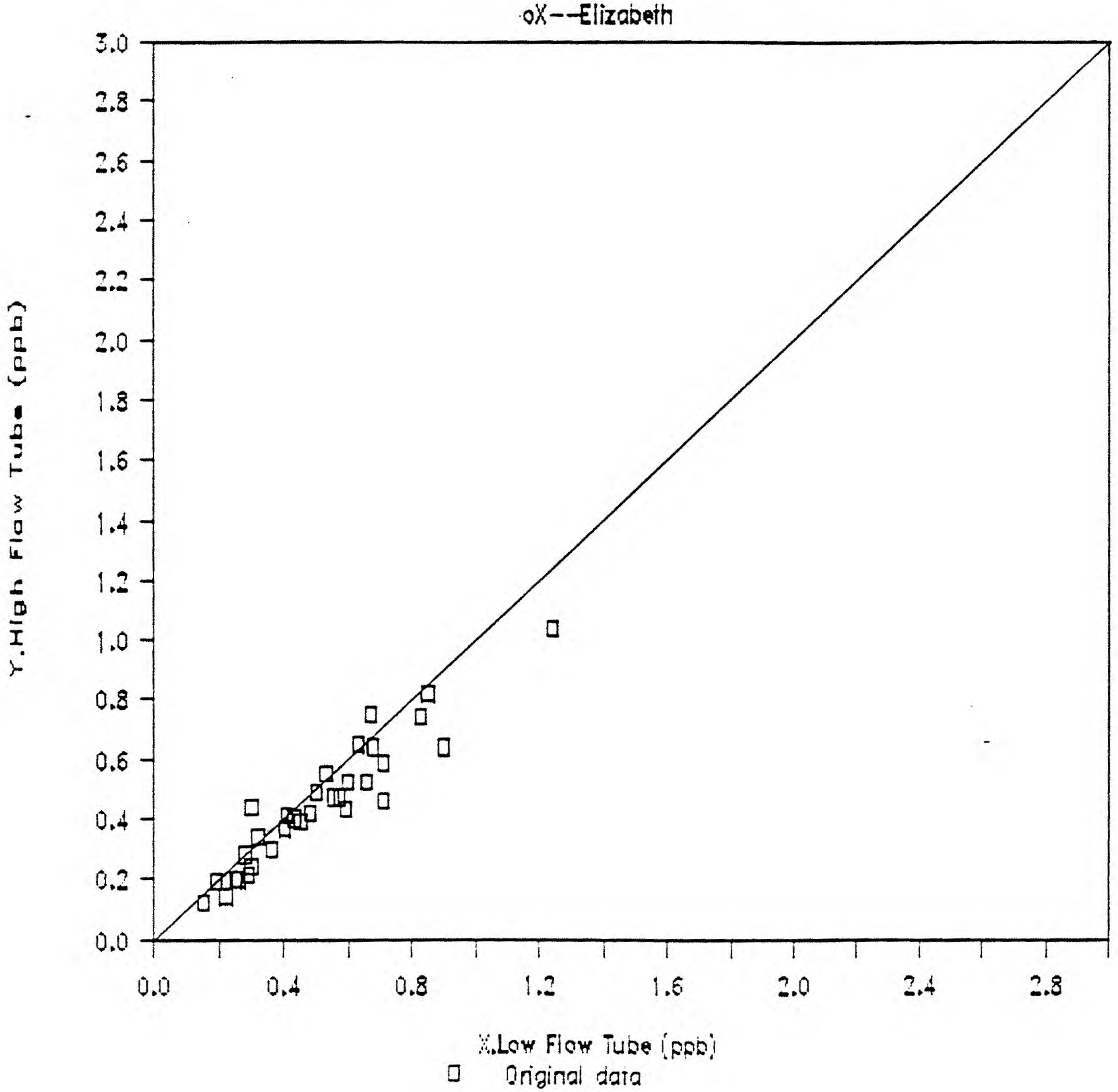


Figure 10 - 1 Concentration Trend of C6

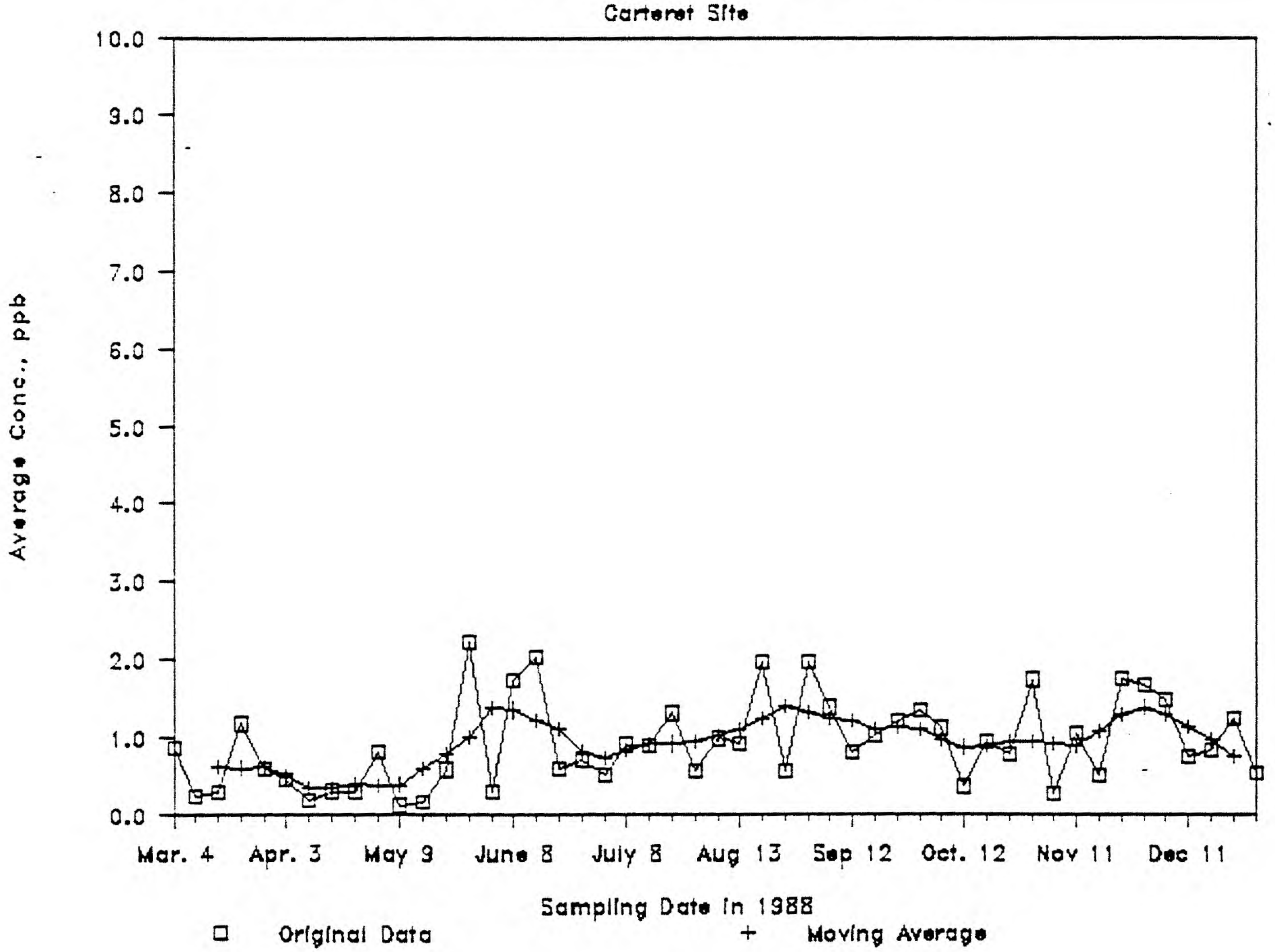


Figure 10 - 2 **Concentration Trend of 1,1,1**

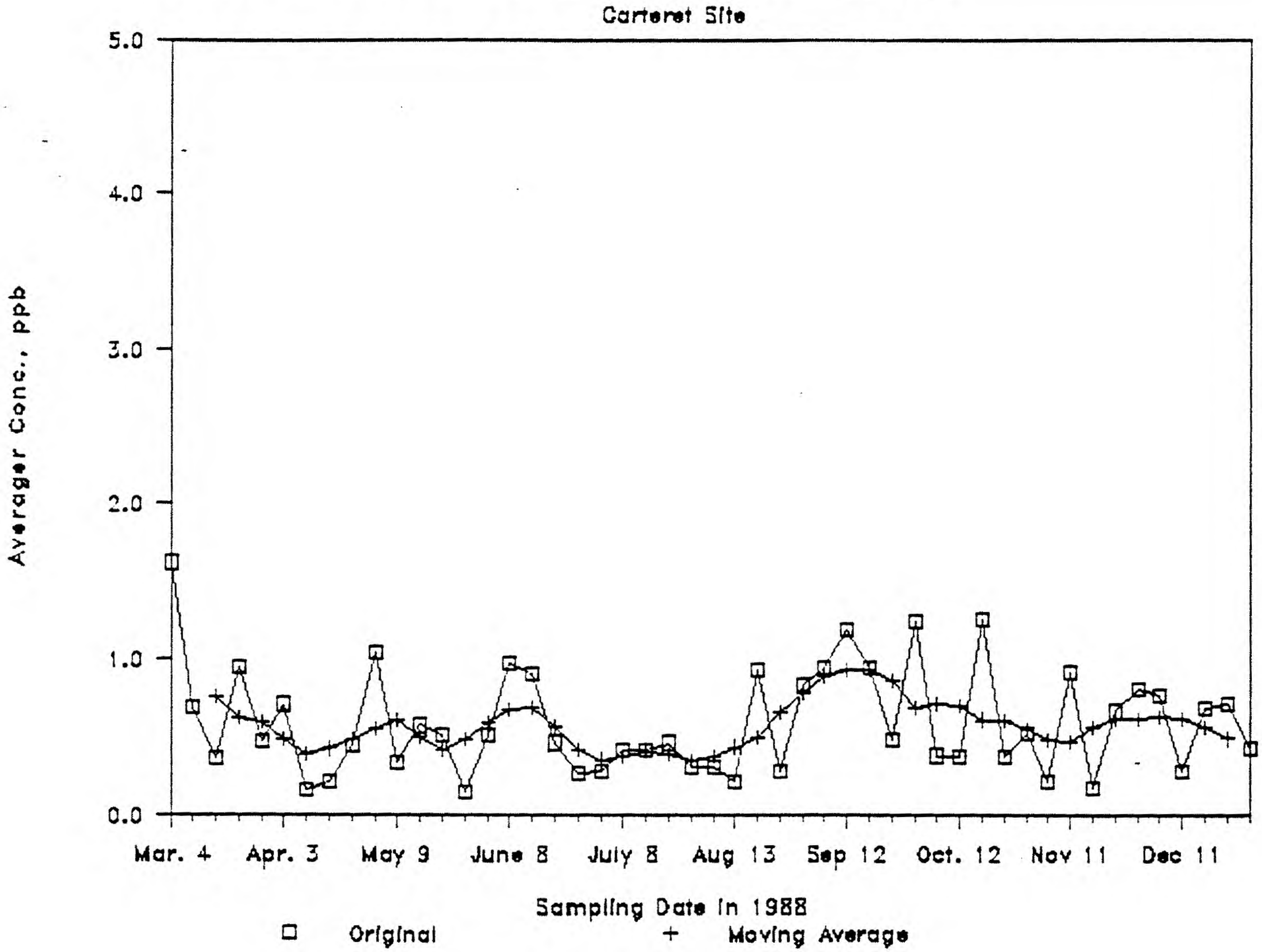


Figure 10 - 3 Concentration Trend of Bz

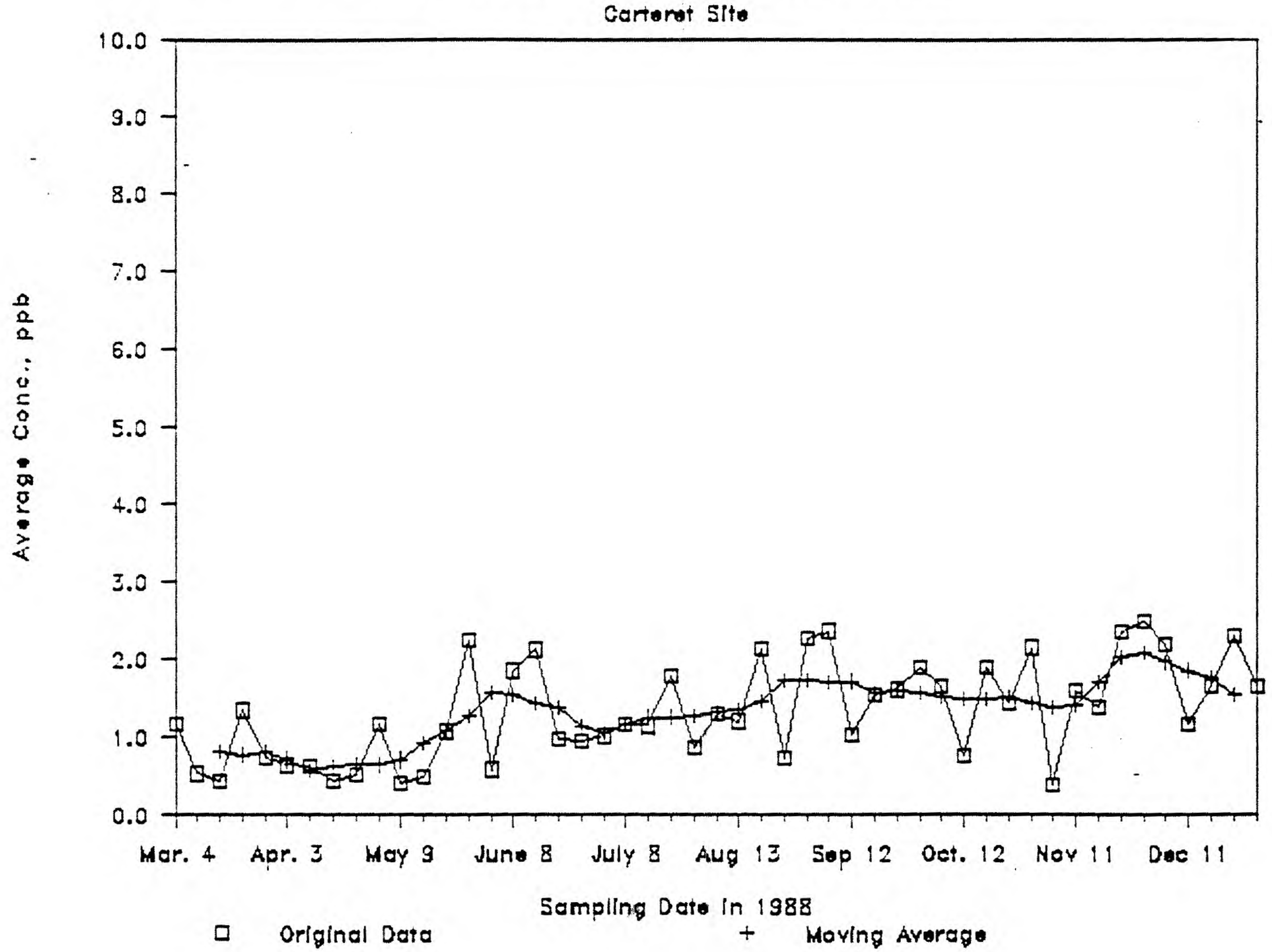


Figure 10 - 4 Concentration Trend of CCl4

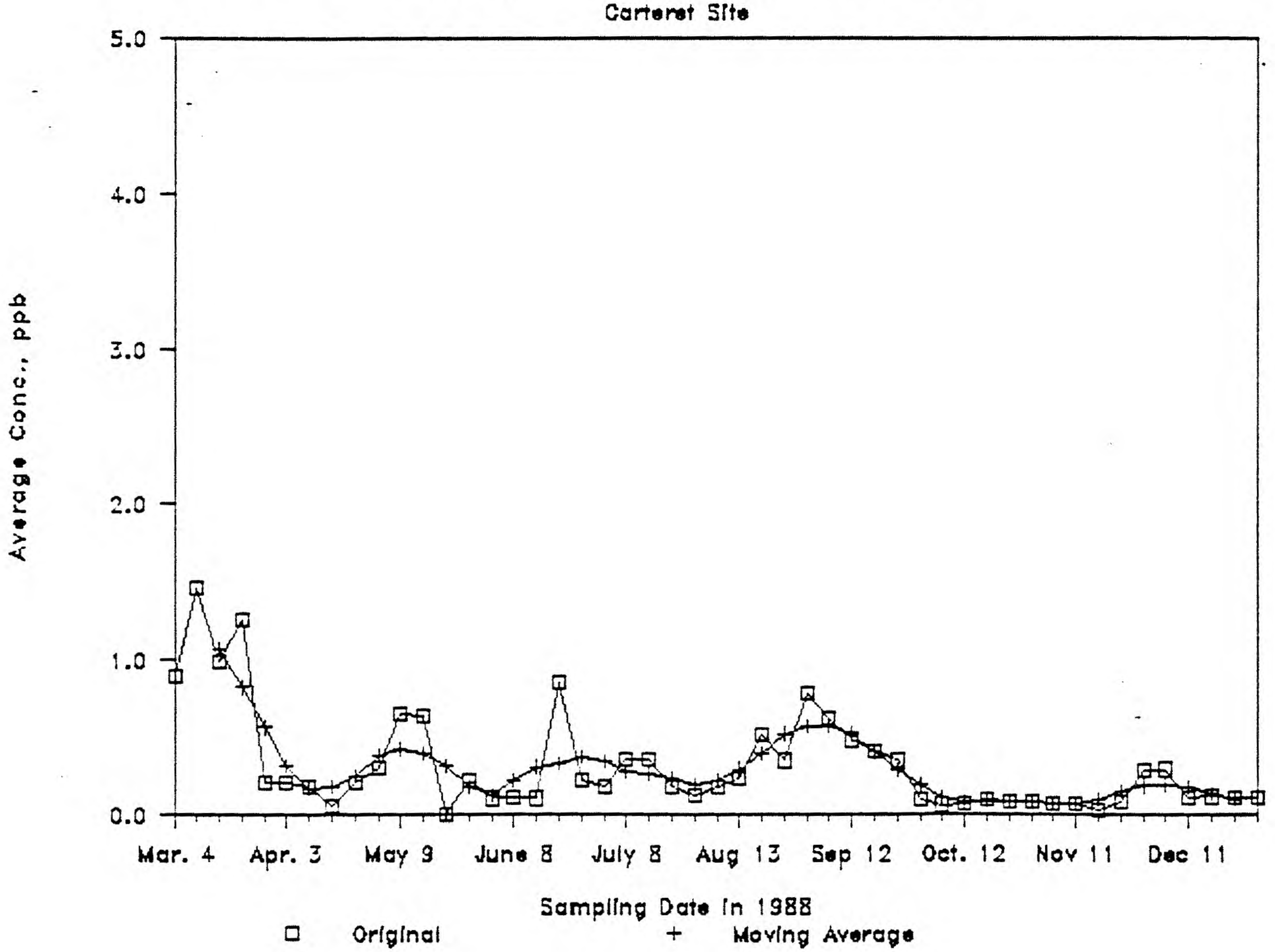


Figure 10 - 5 Concentration Trend of Tol.

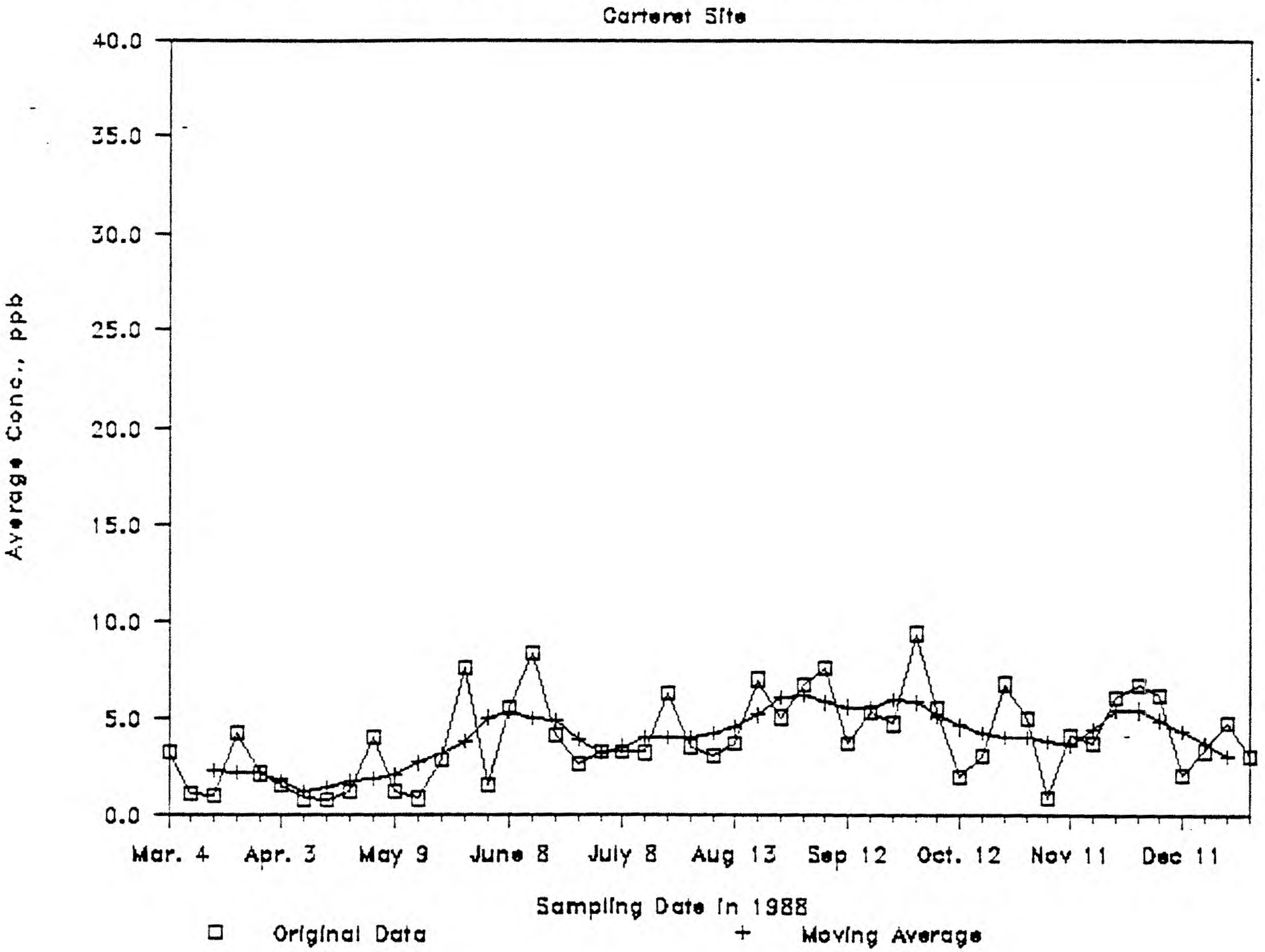


Figure 10 - 6 **Concentration Trend of Perc**

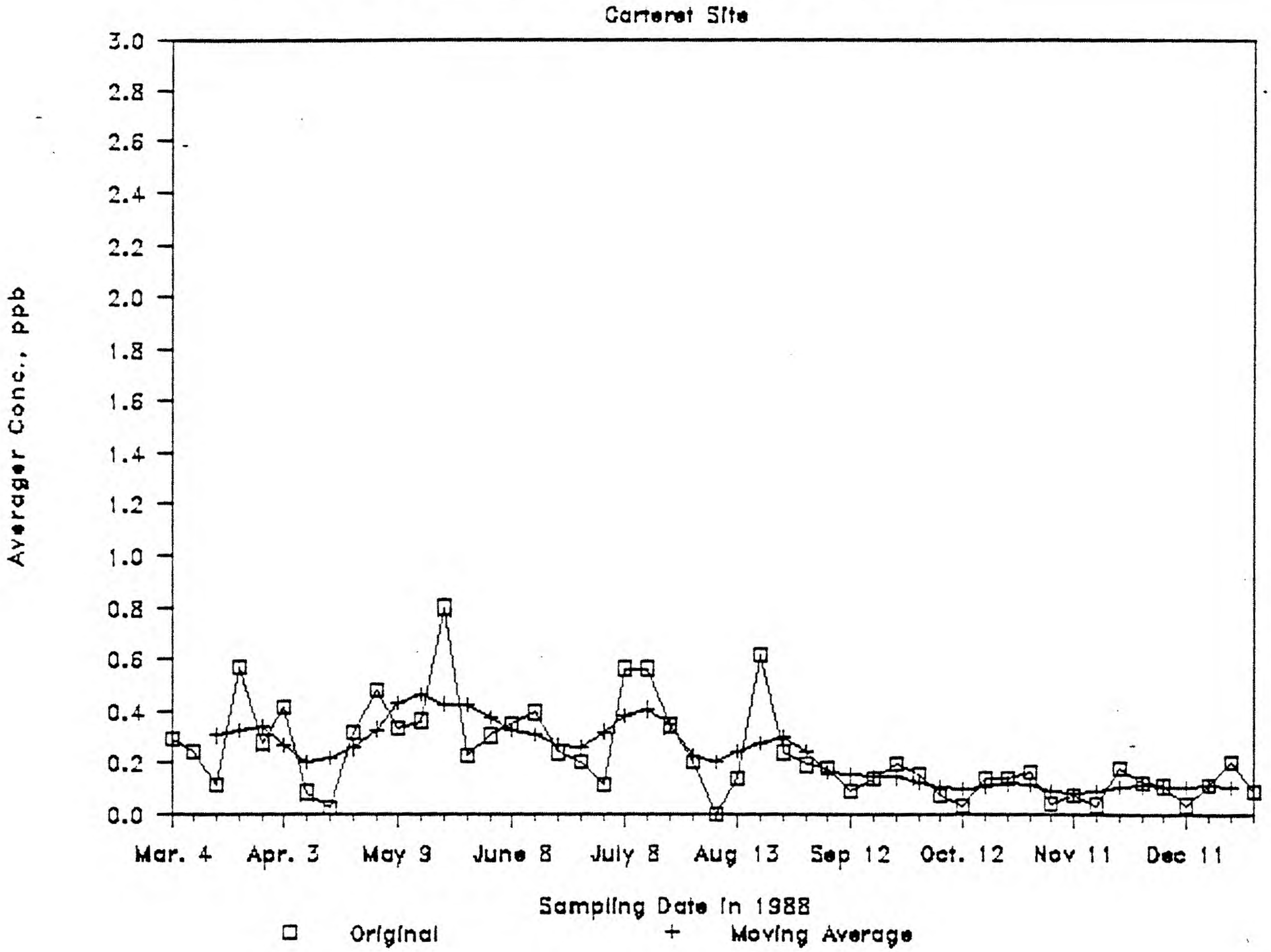


Figure 10 - 7 **Concentration Trend of pmX**

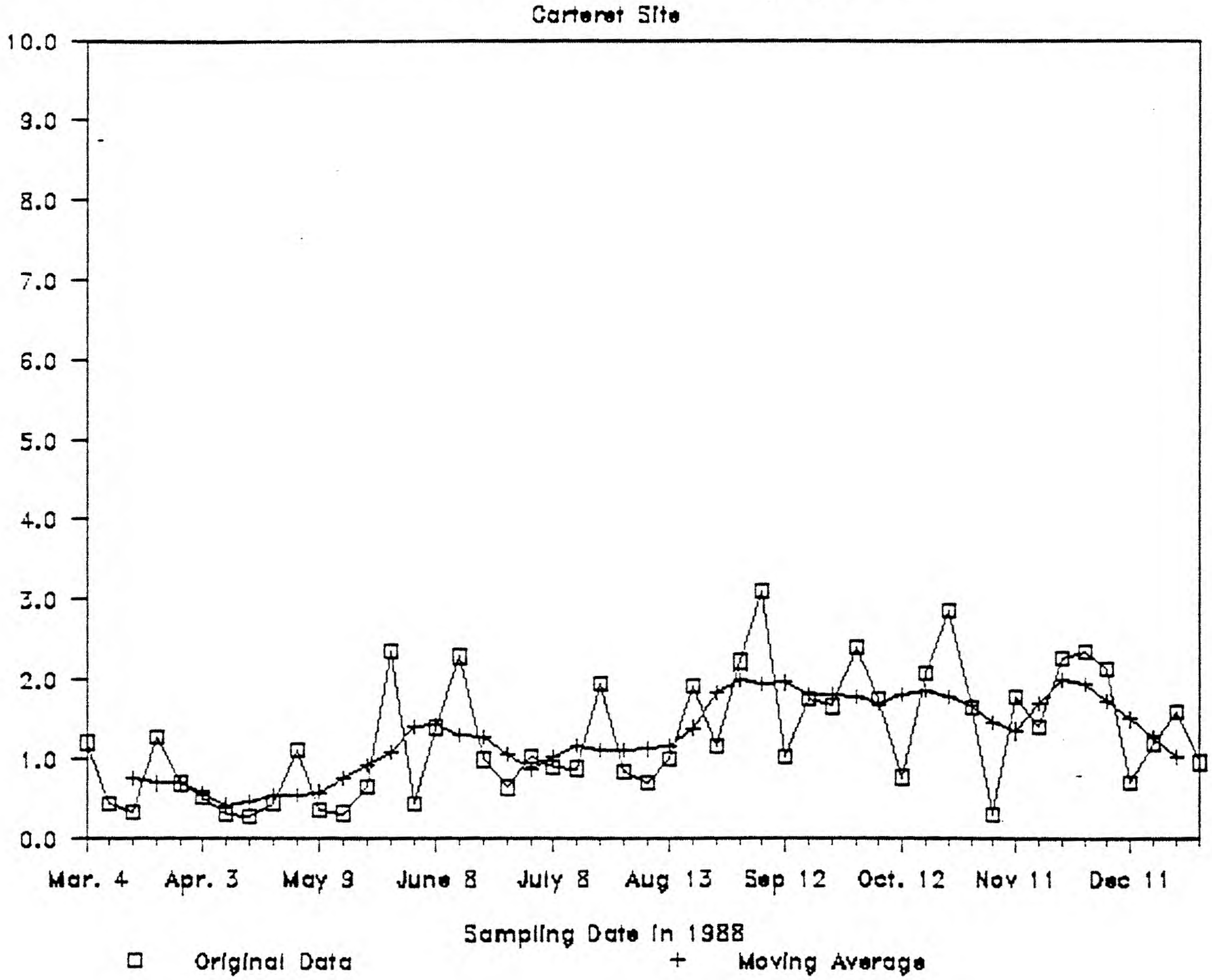


Figure 10 - 8 Concentration Trend of oX

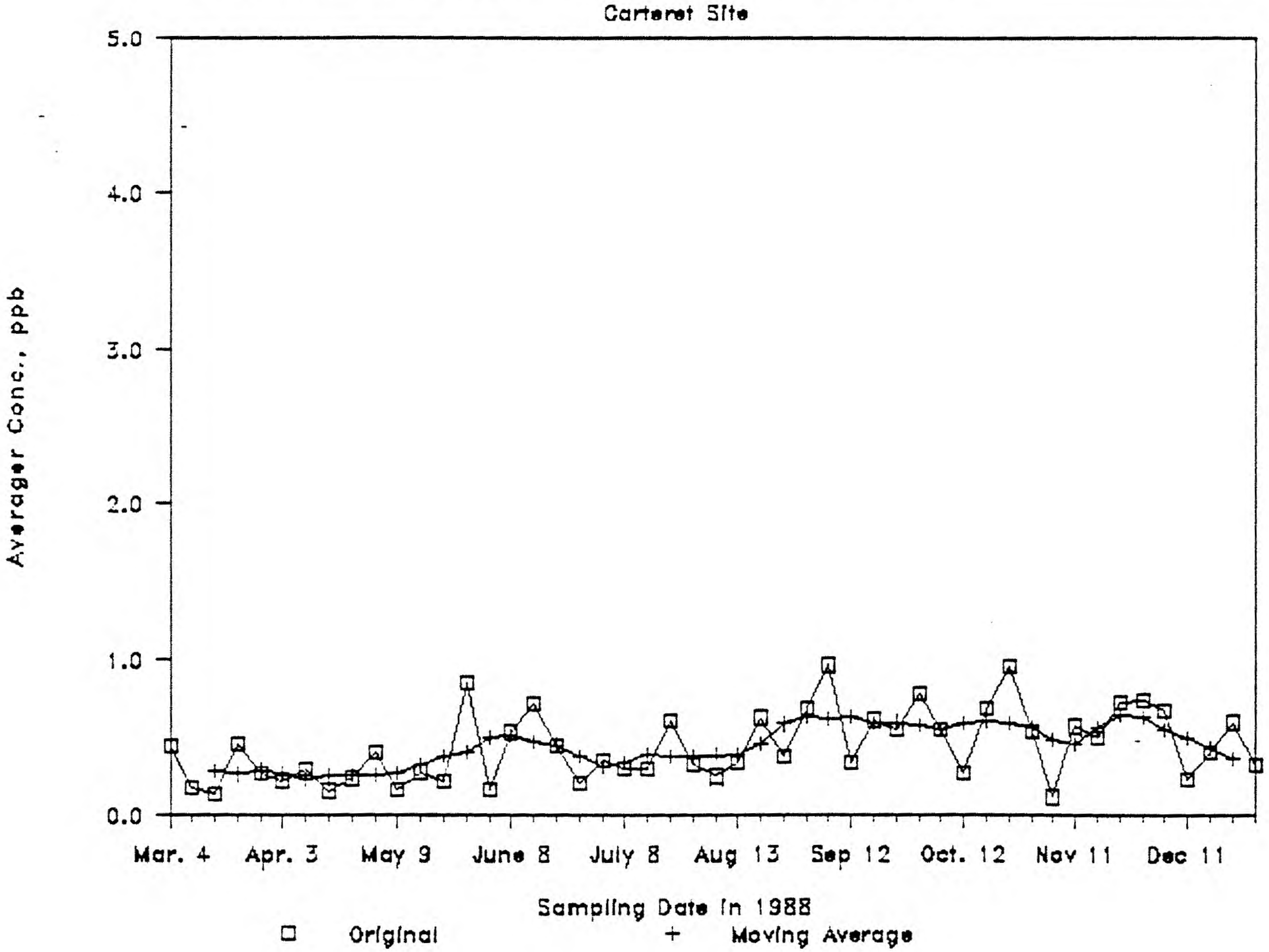


Figure 10 - 9 **Concentration Trend of C6**

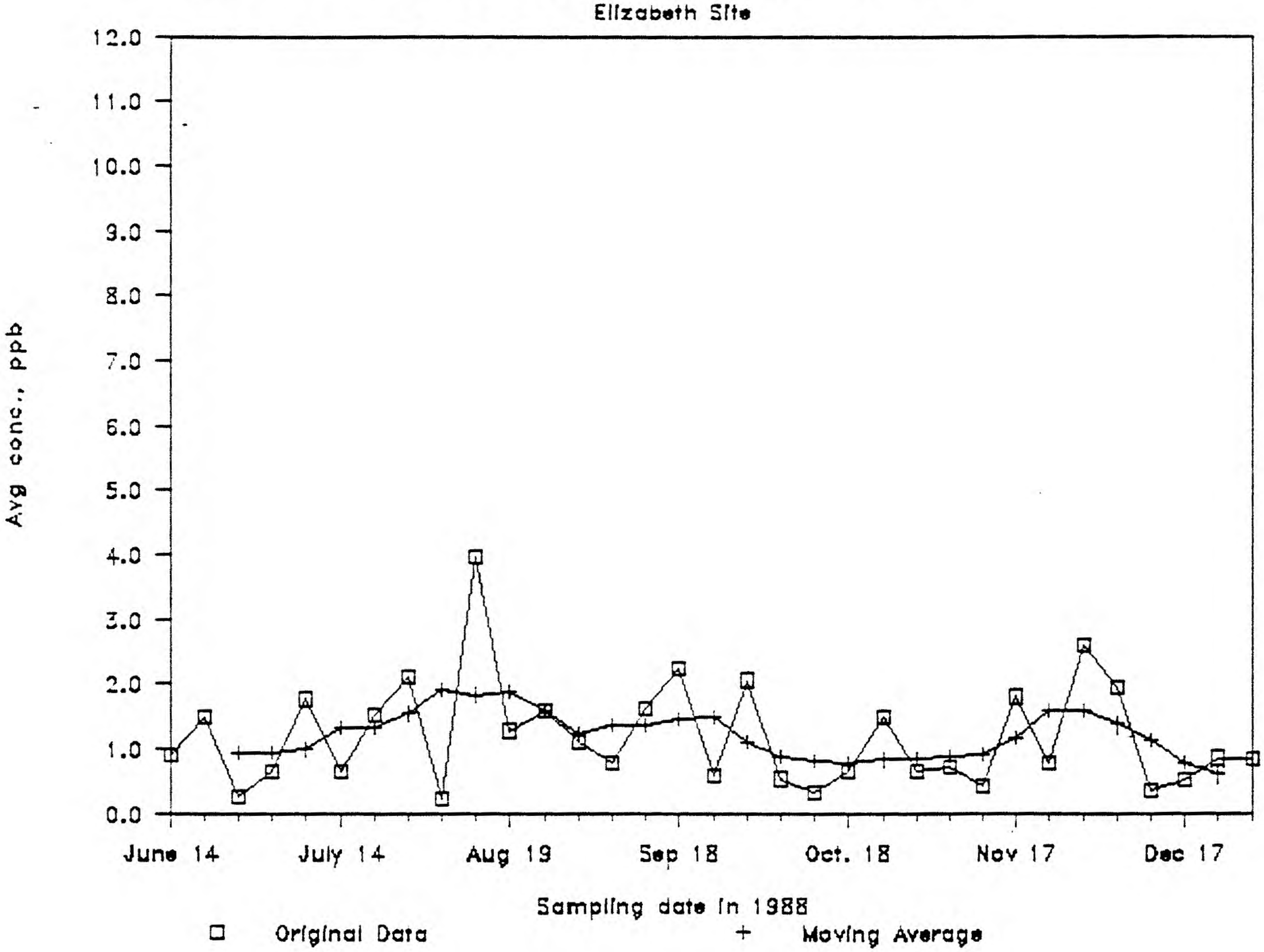


Figure 10 - 10 **Concentration Trend of 1,1,1**

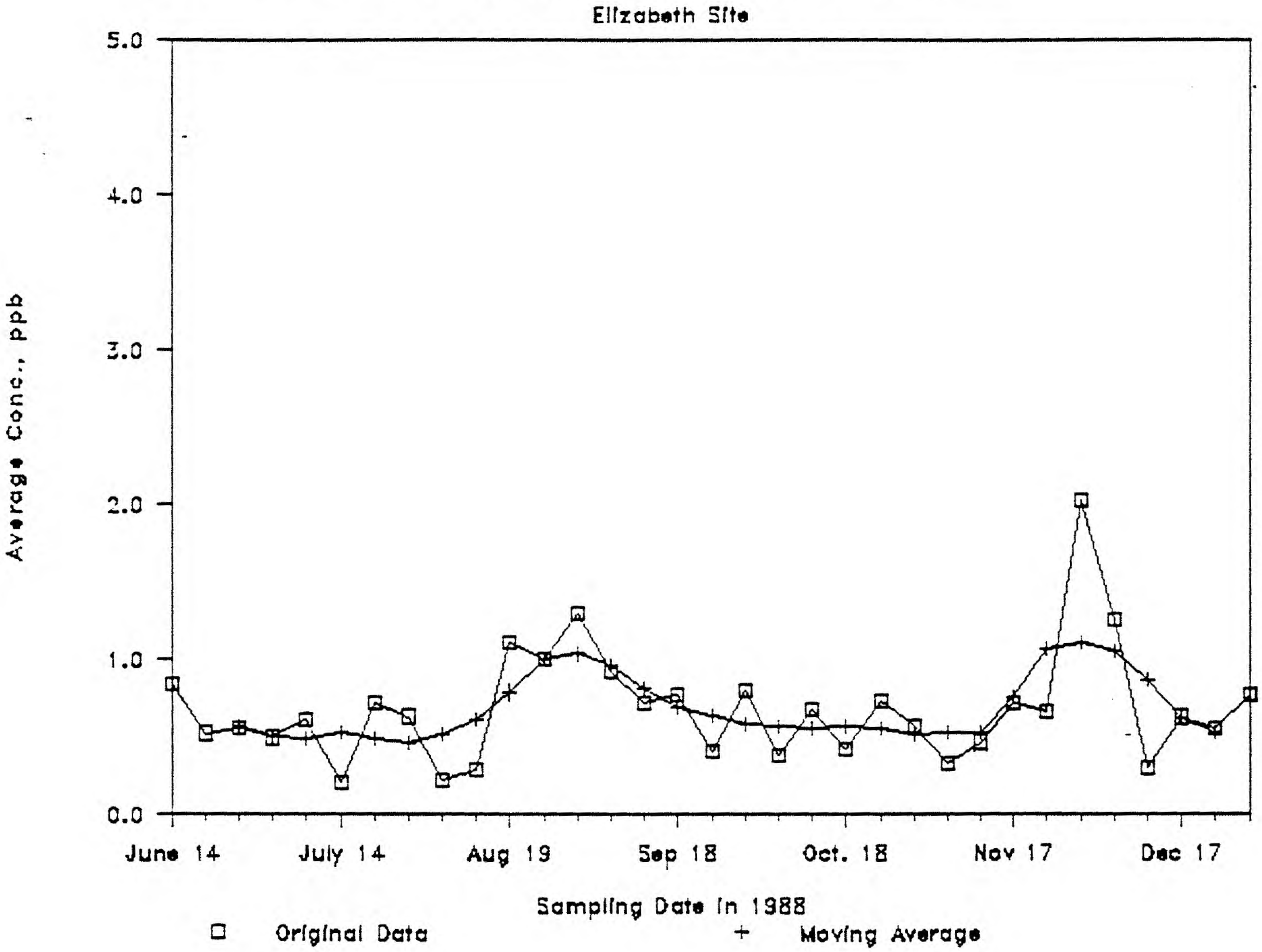


Figure 10 - 11 Concentration Trend of Bz

Elizabeth Site

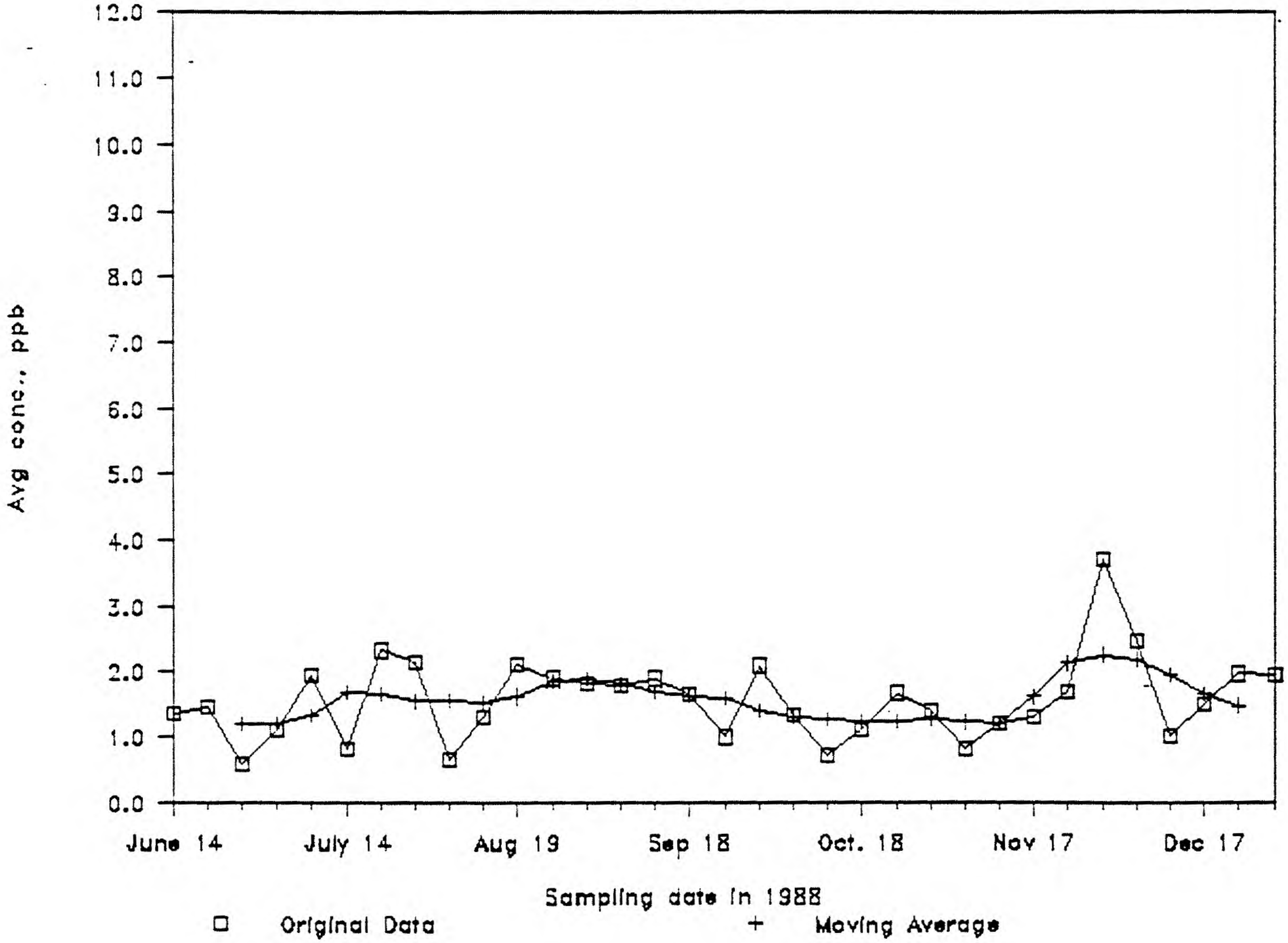


Figure 10 - 12 **Concentration Trend of CCl4**

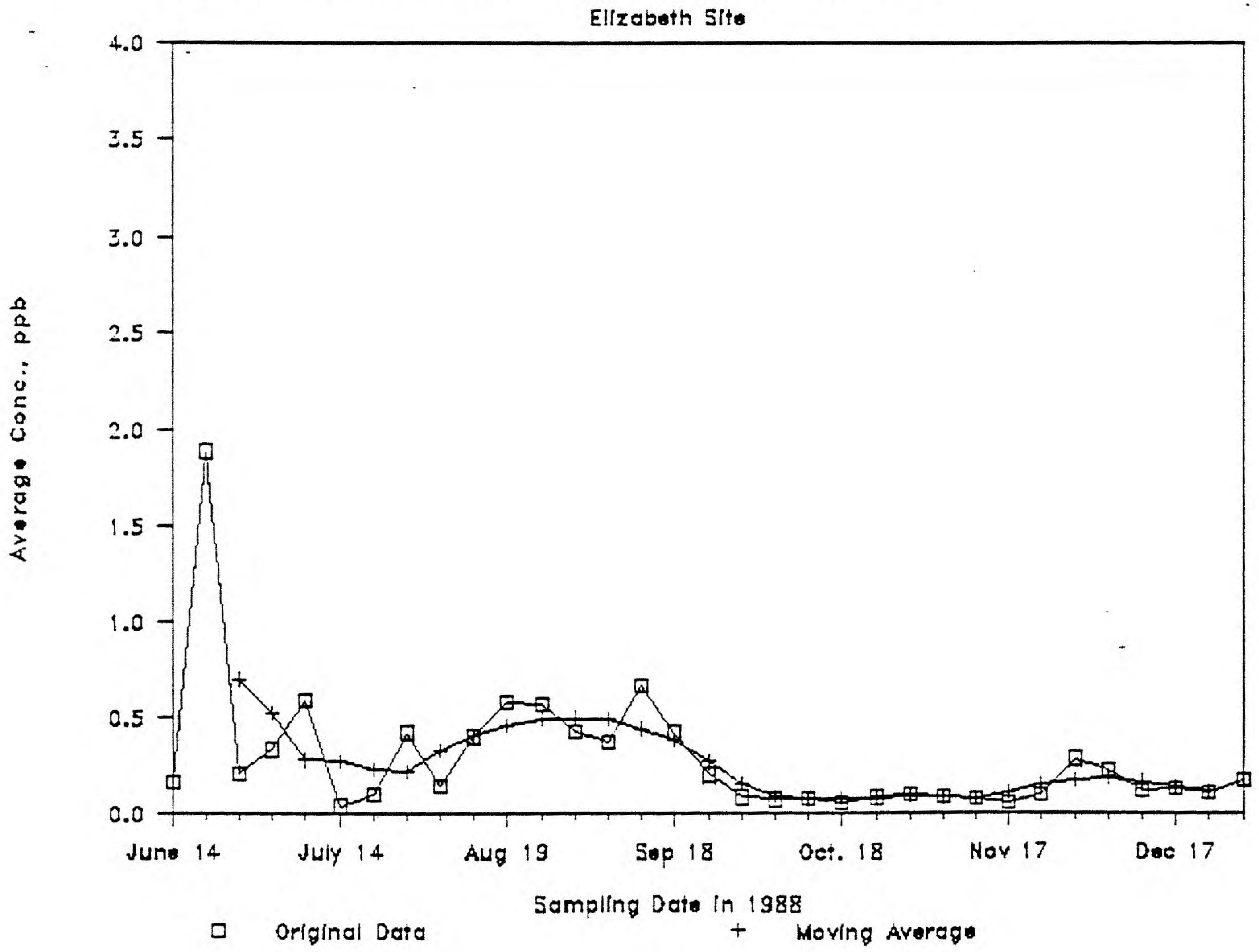


Figure 10 - 13 Concentration Trend of Tol

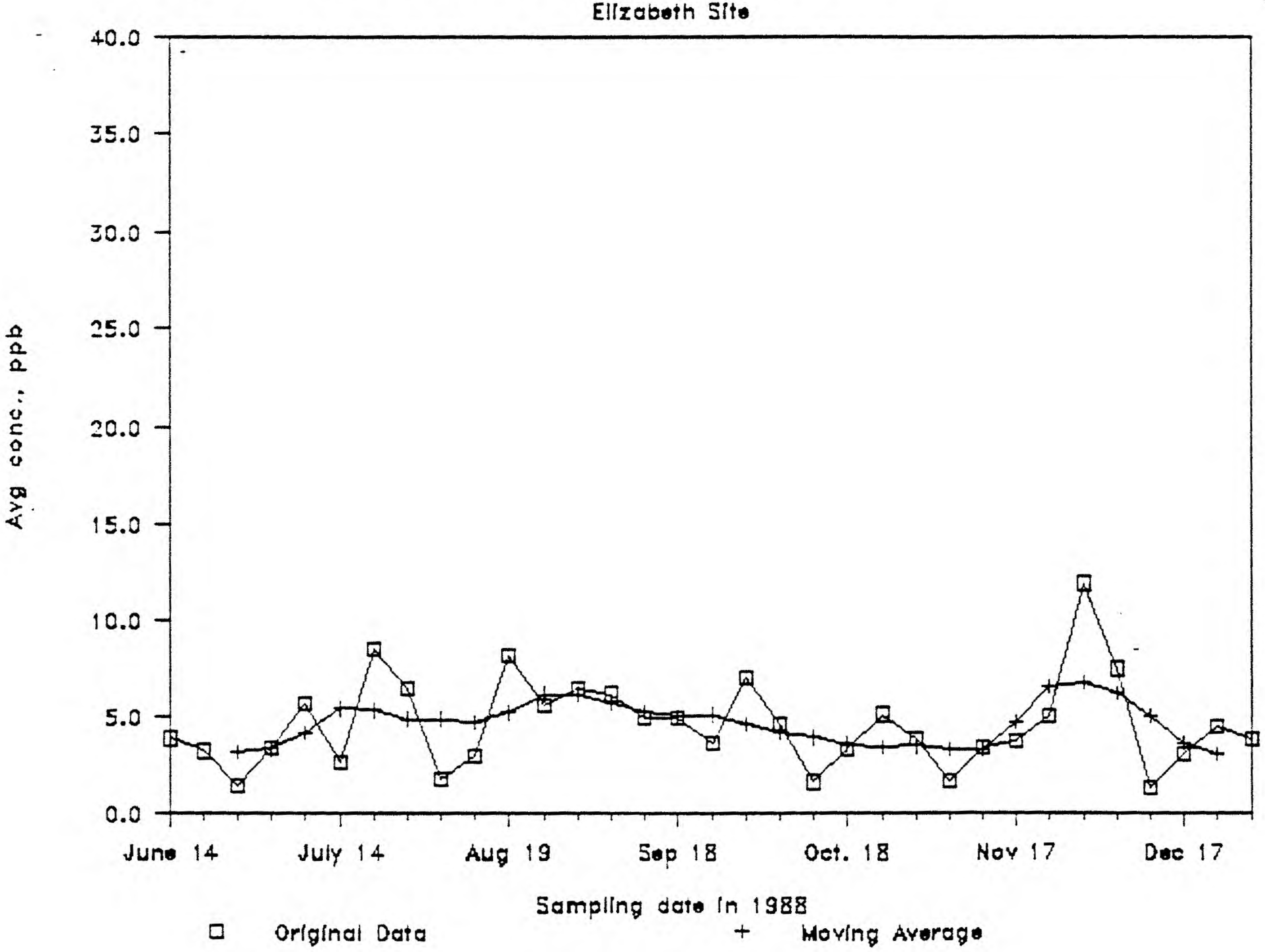


Figure 10 - 14 Concentration Trend of Perc

Elizabeth Site

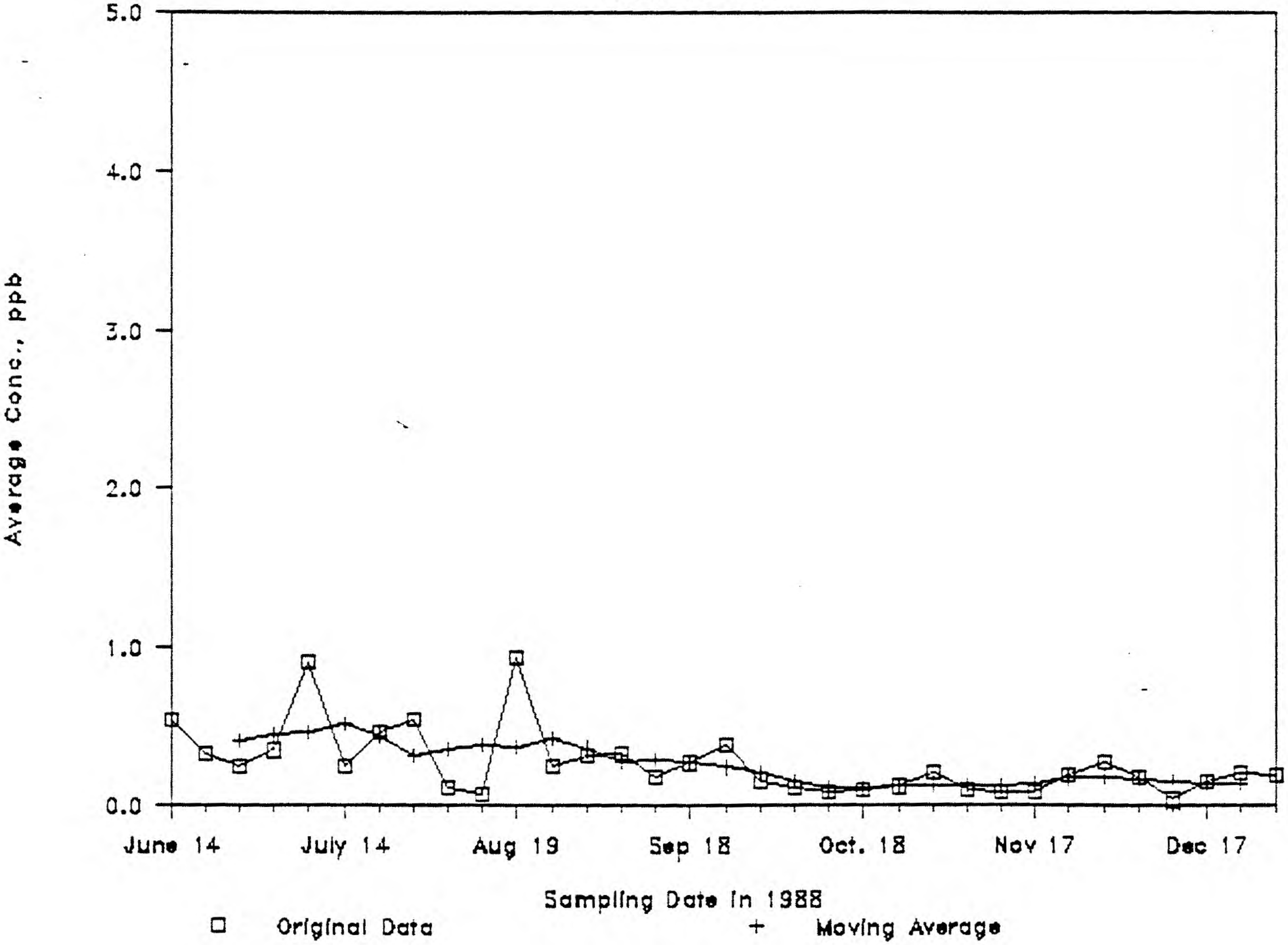


Figure 10 - 15 Concentration Trend of pmX

Elizabeth Site

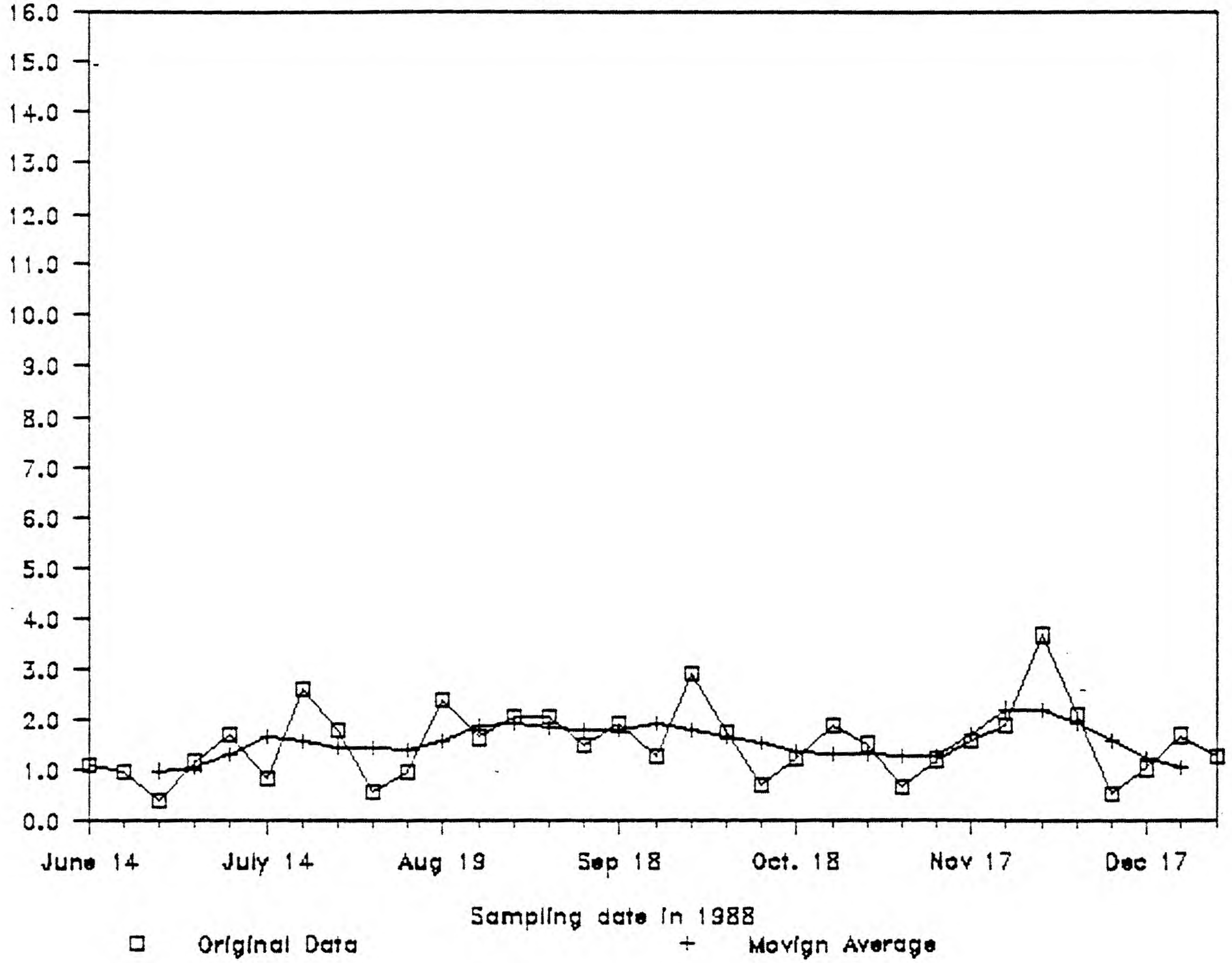


Figure 10 - 16 Concentration Trend of oX

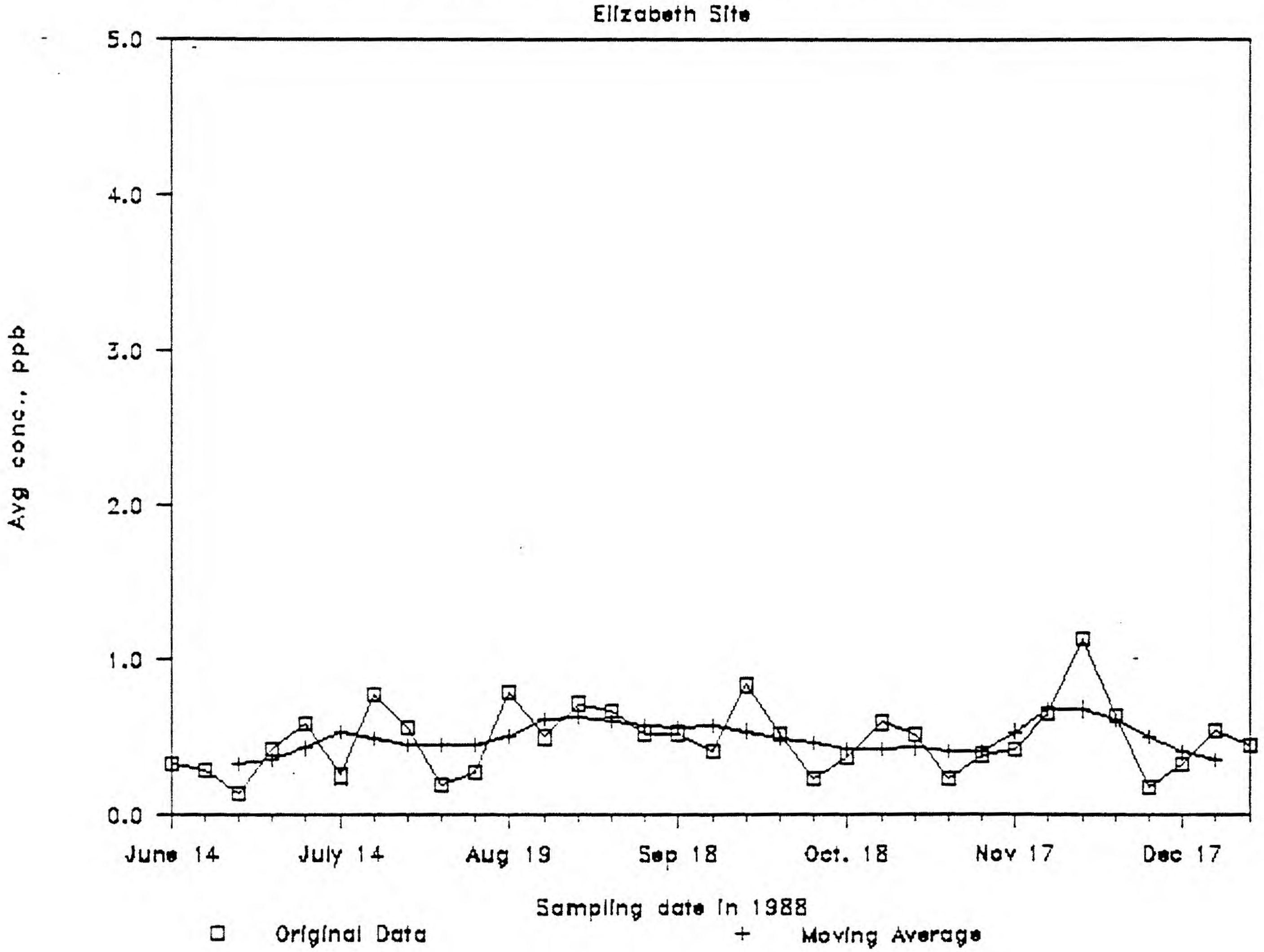


Figure 11 - 1 Comparison of Moving Avg at Carteret

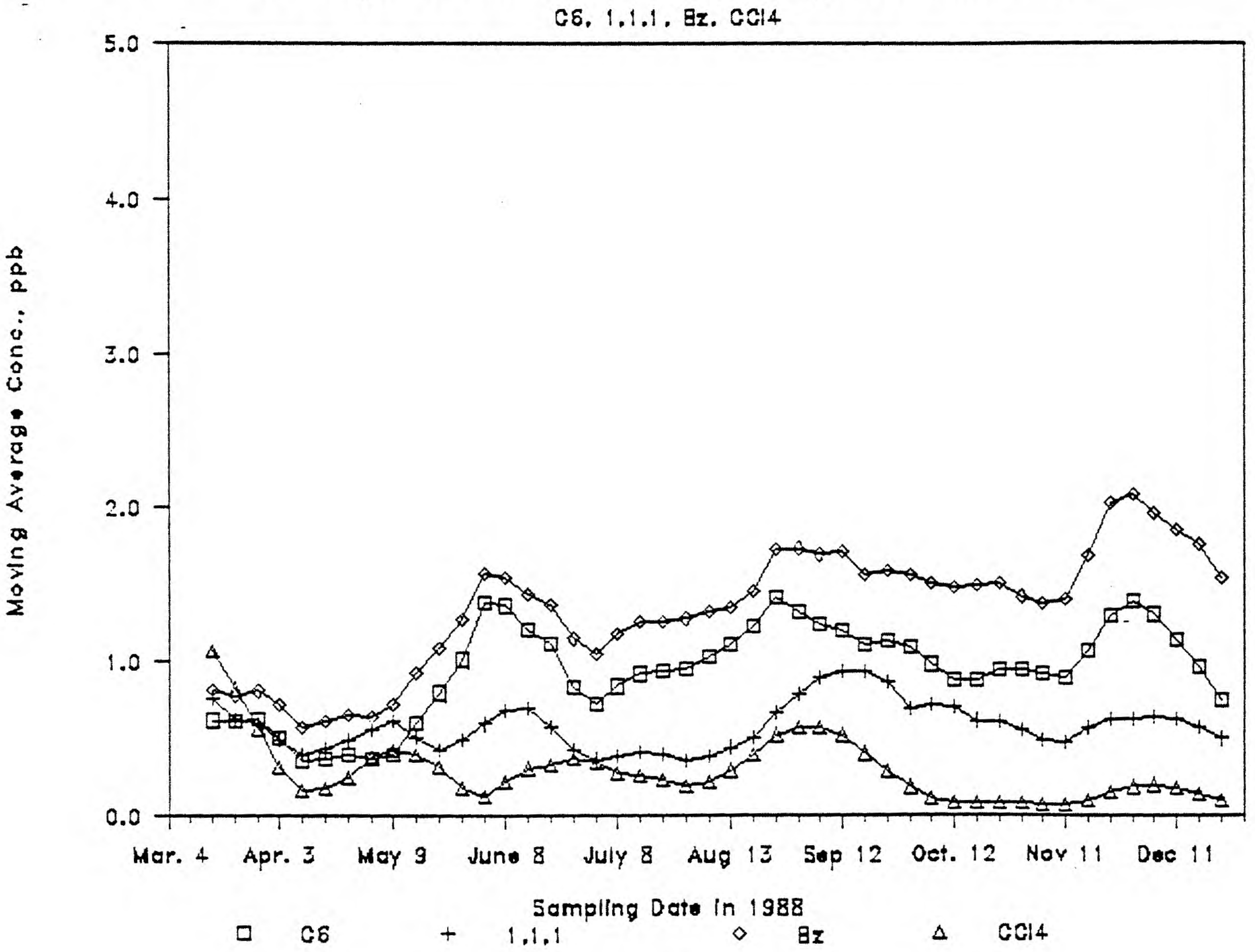


Figure 11 - 2 Comparison of Moving Avg at Carteret

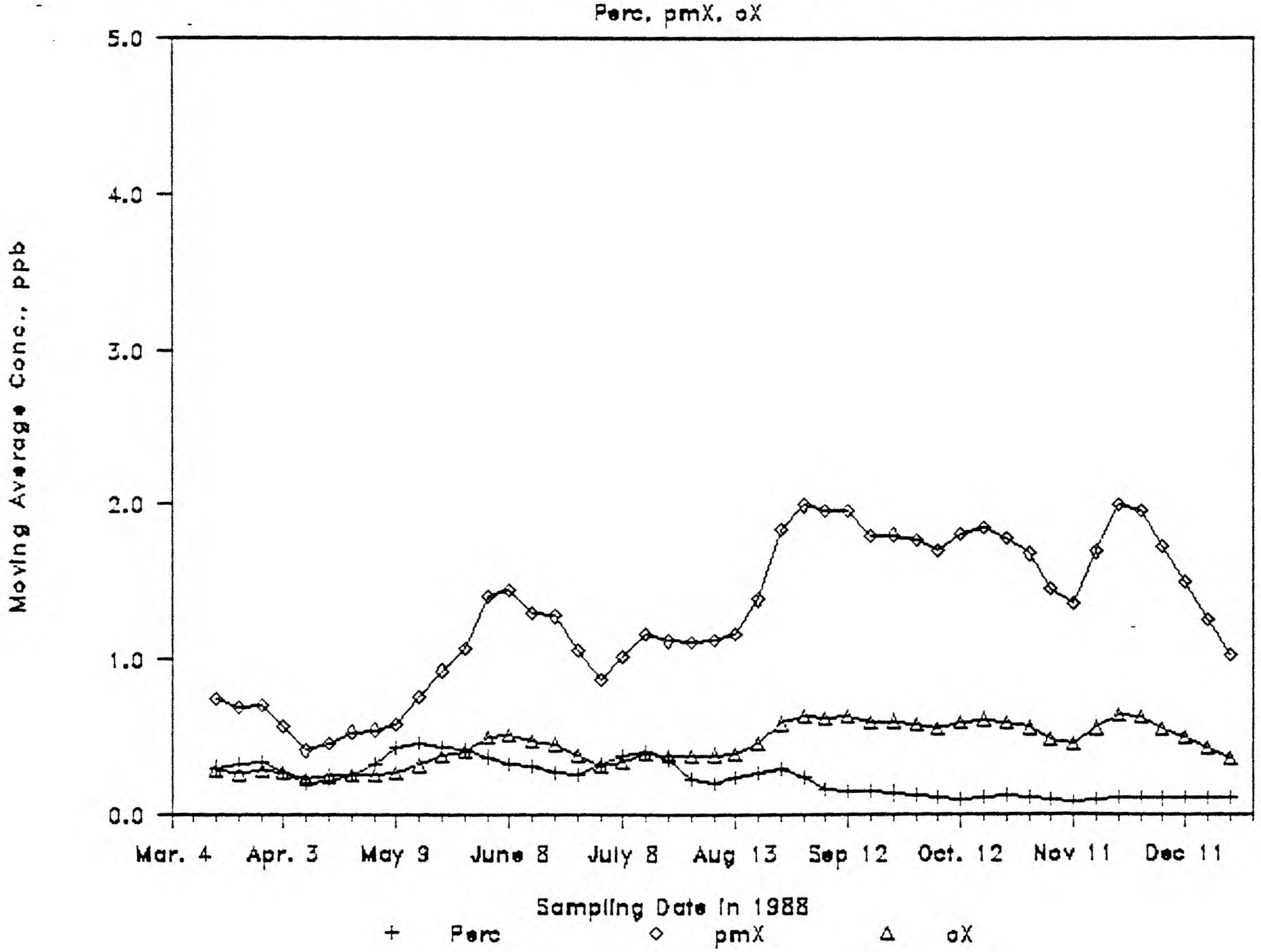


Figure 11 - 3 Comparison of Moving Avg at Elizabeth

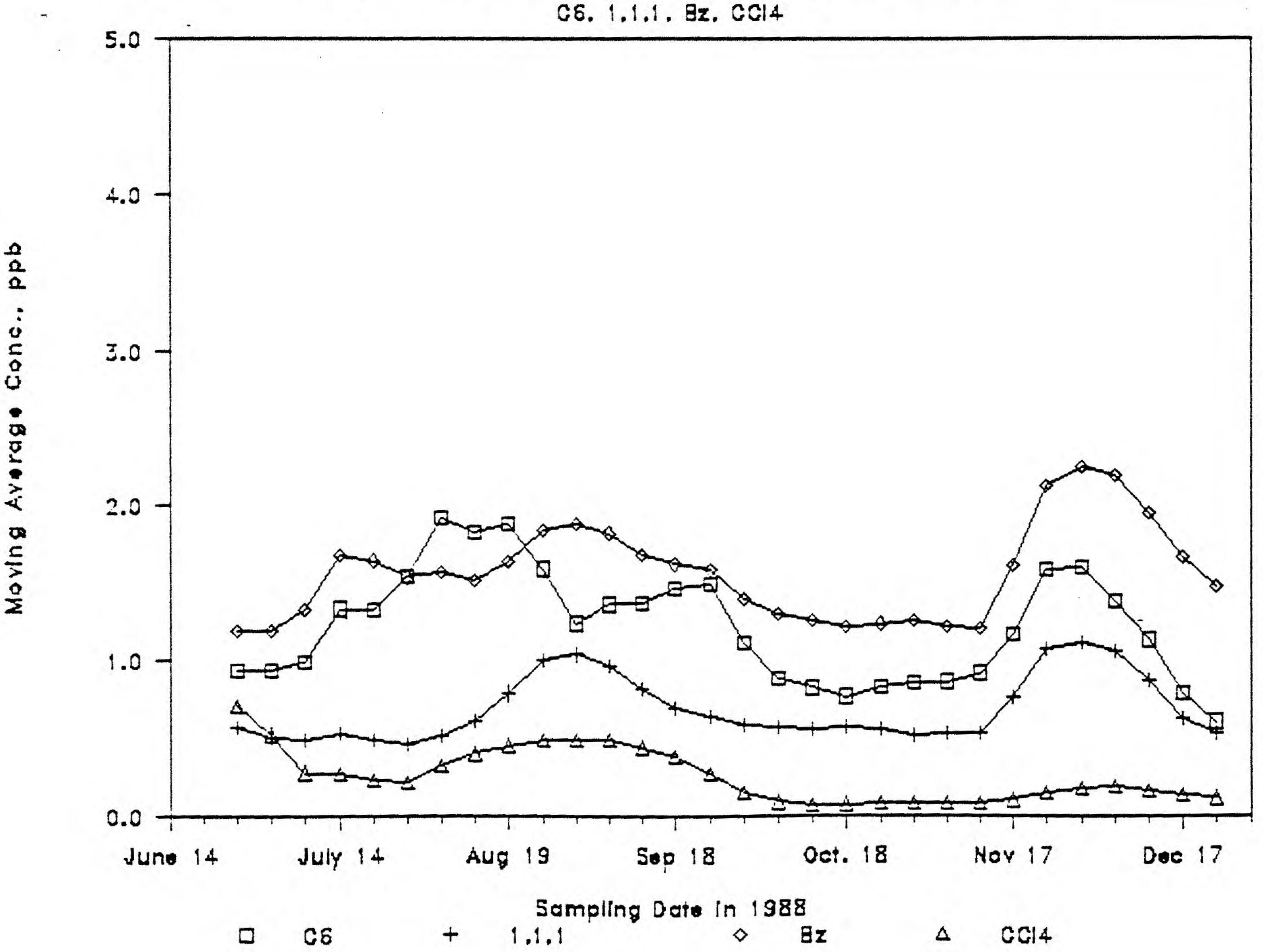


Figure 11 - 4 Comparison of Moving Avg at Elizabeth

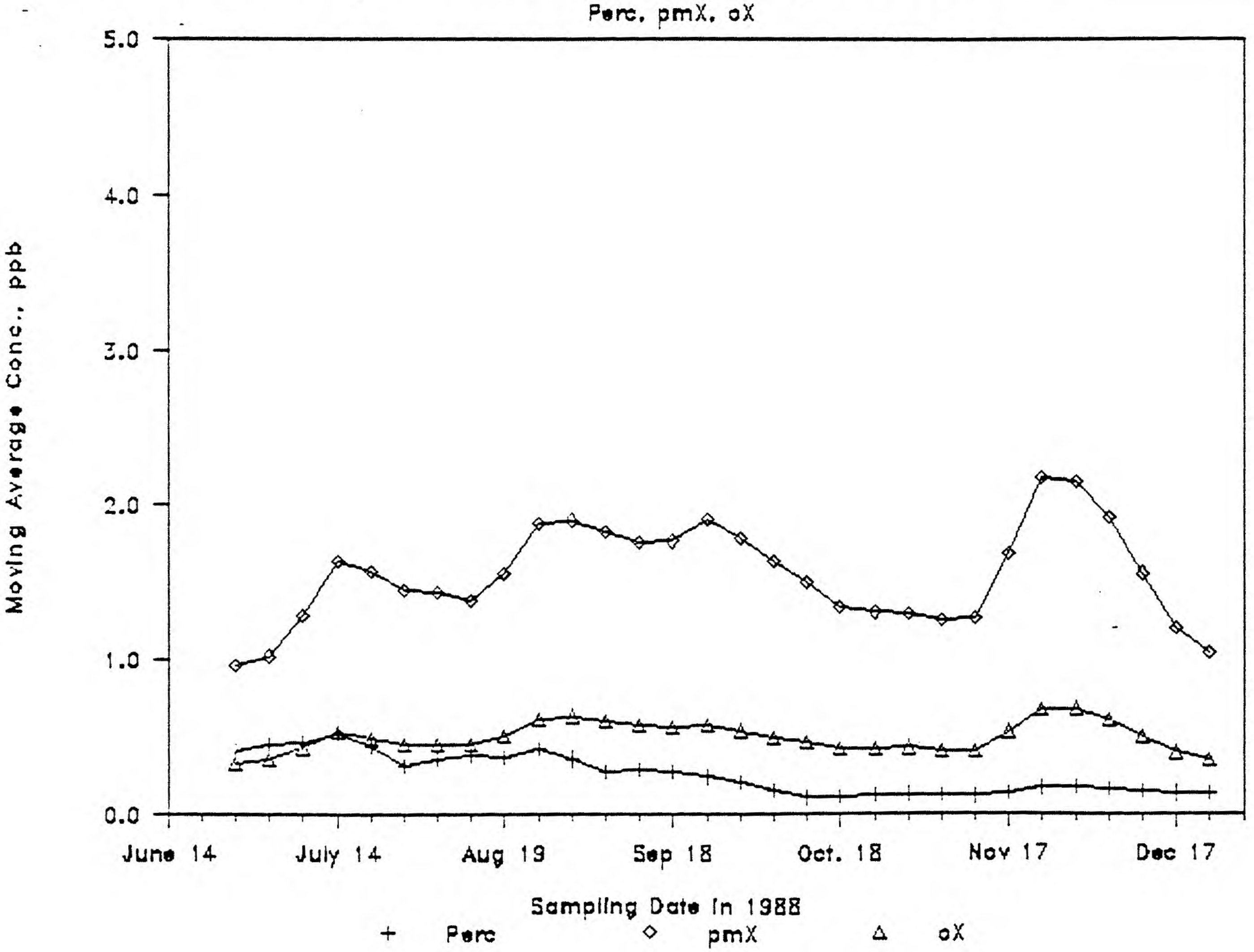


Figure 12 - 1 Comparison of Moving Avg Between Sites

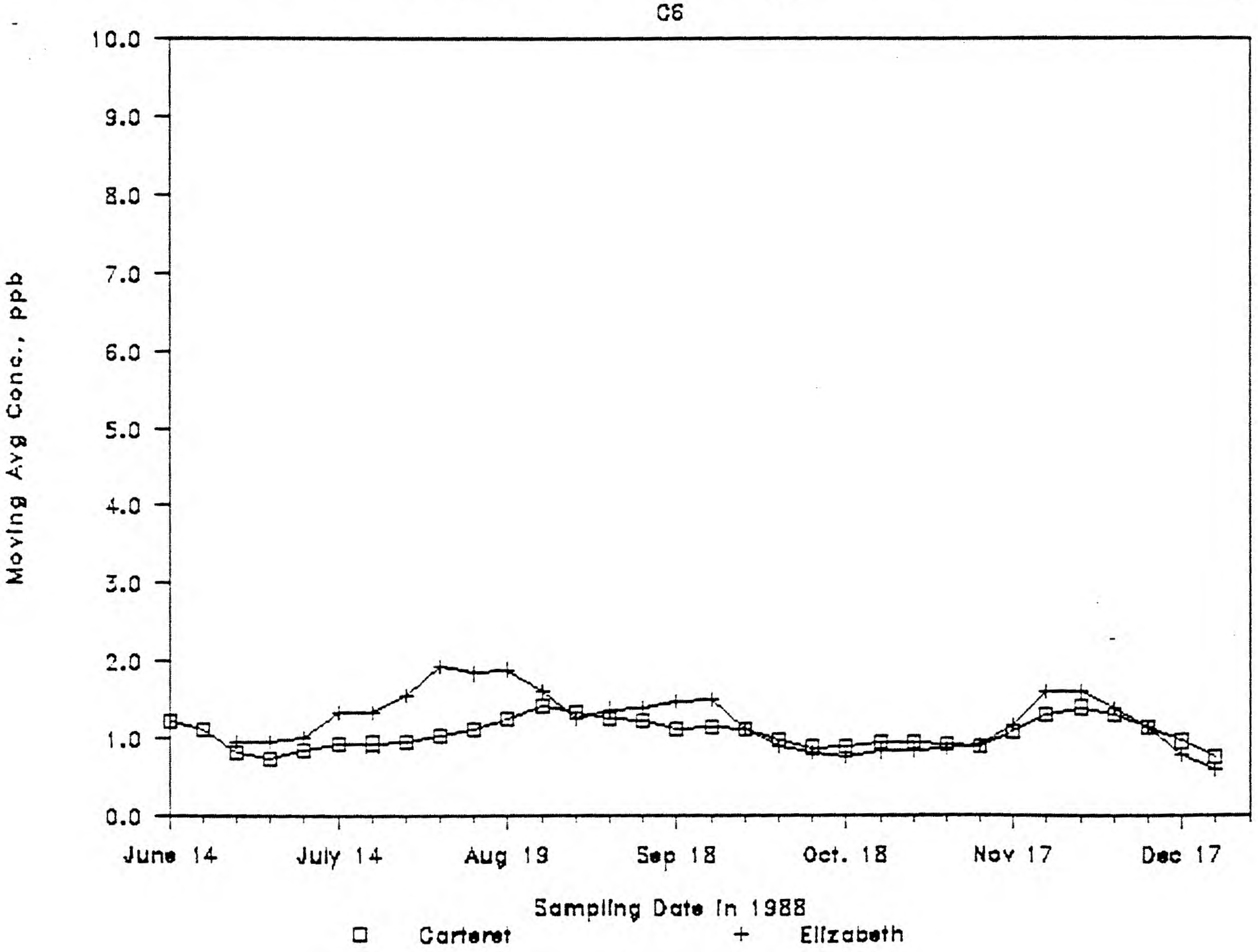


Figure 12 - 2 **Comparison of Moving Avg Between Site**

1,1,1

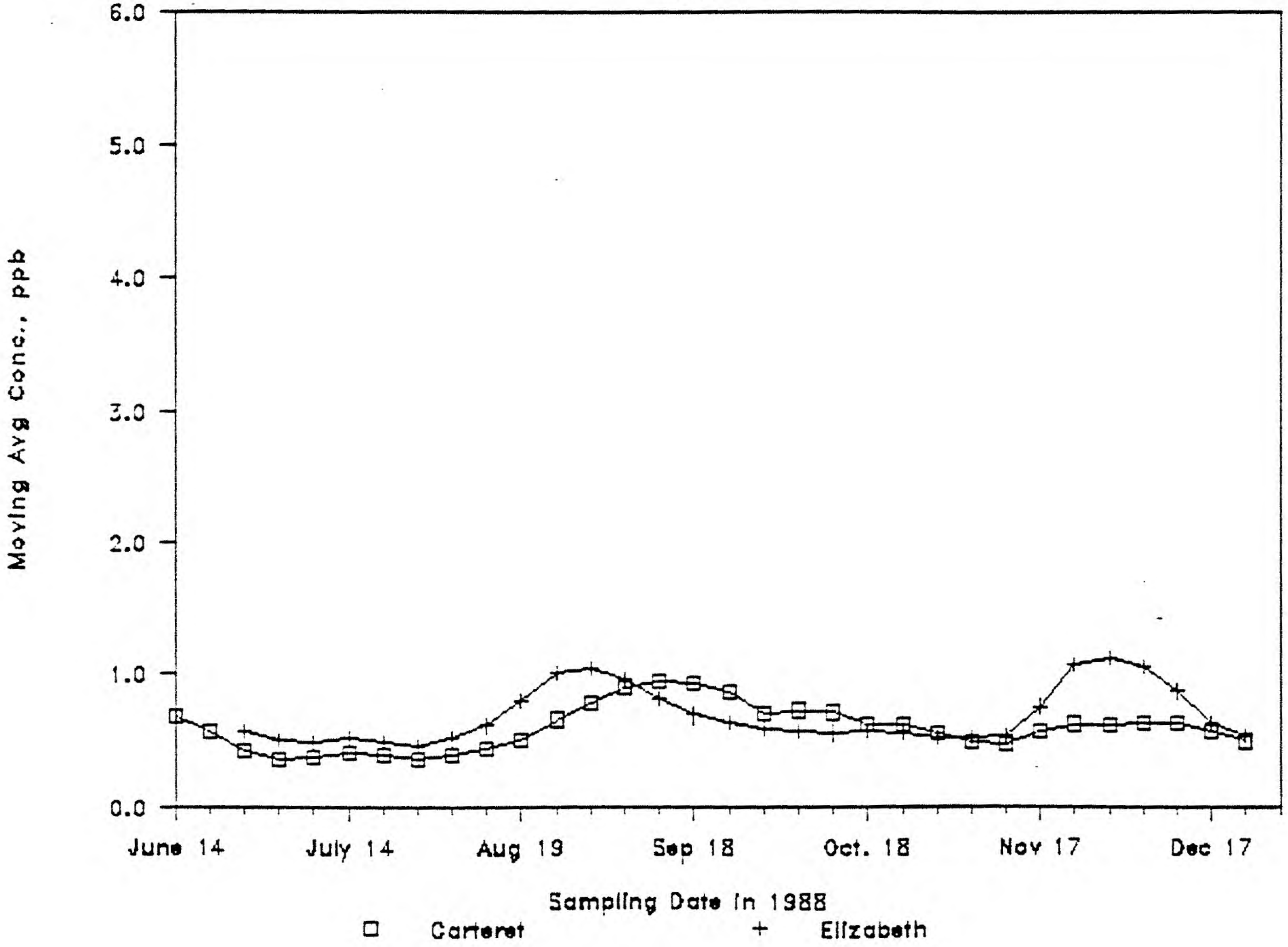


Figure 12 - 3 Comparison of Moving Avg Between Sites

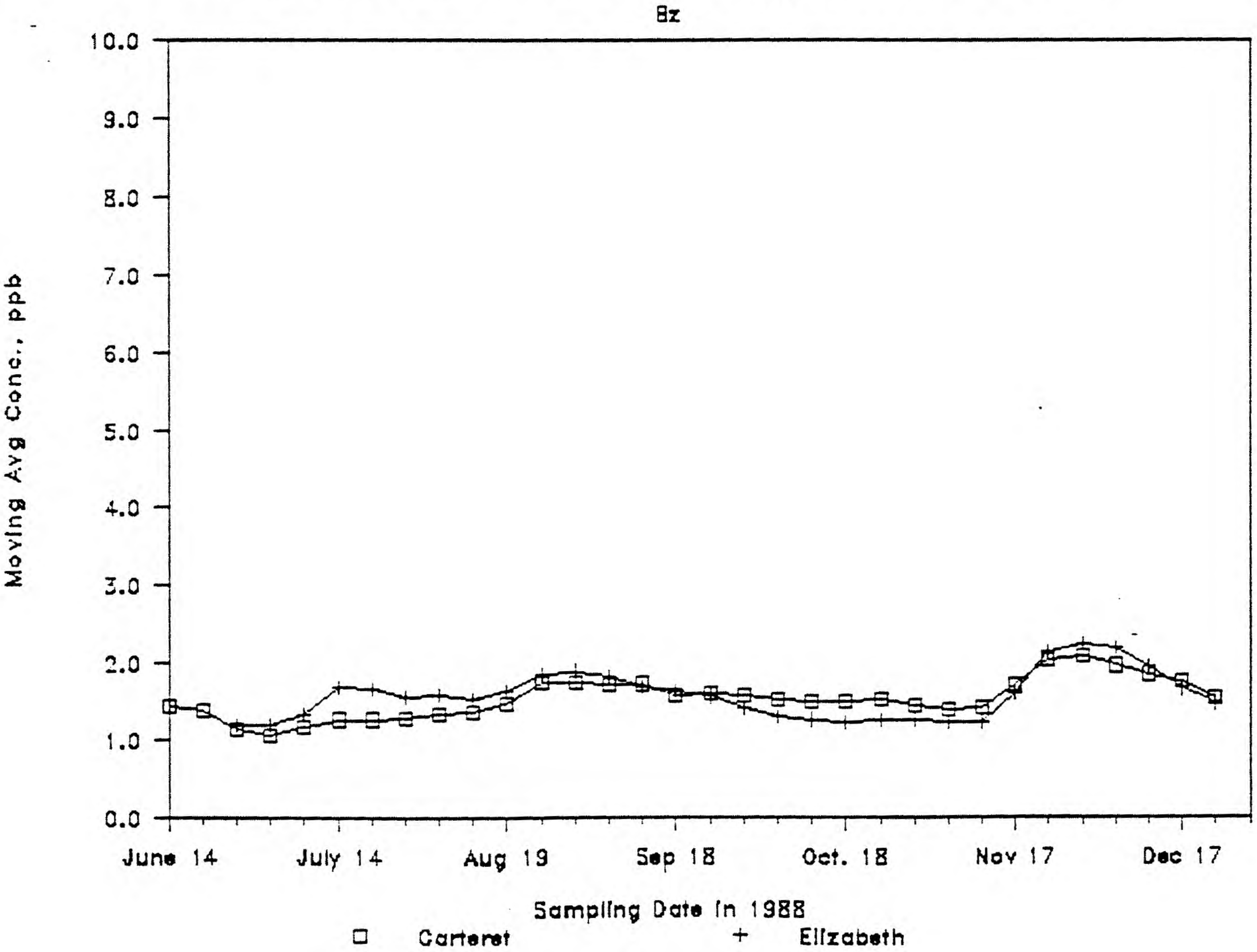


Figure 12 - 4 Comparison of Moving Avg Between Site

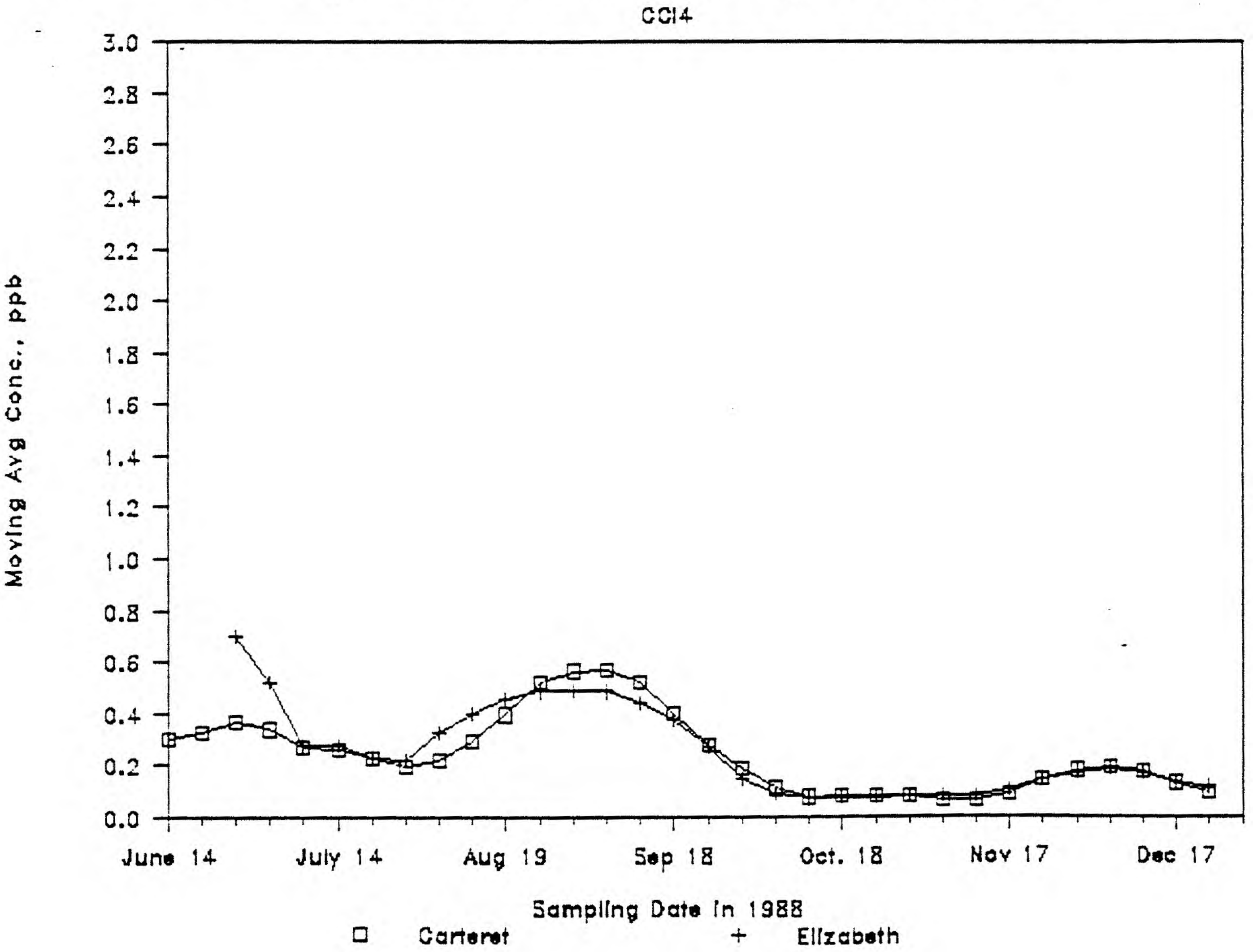


Figure 12 - 5 Comparison of Moving Avg Between Sites

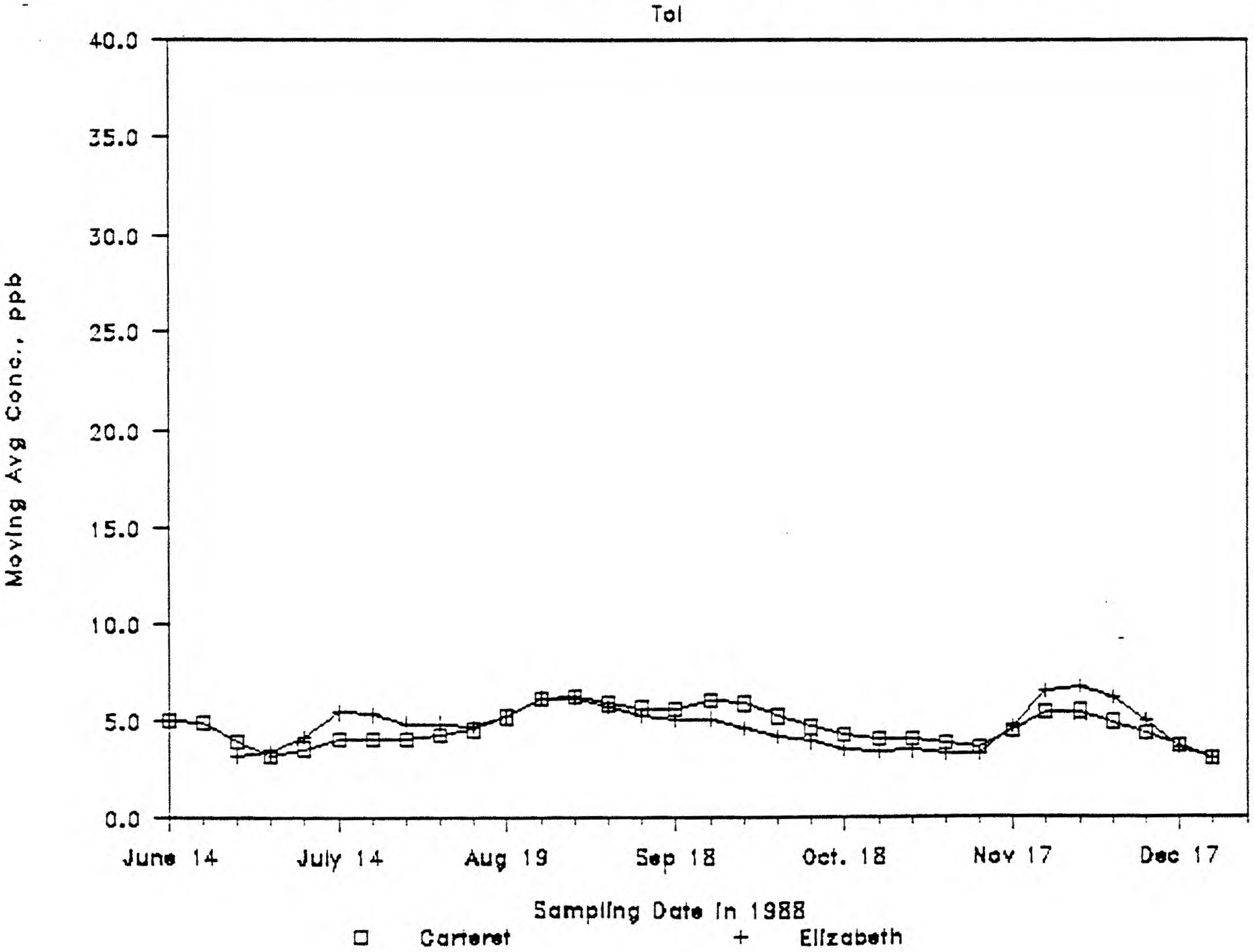


Figure 12 - 6 Comparison of Moving Avg Between Site

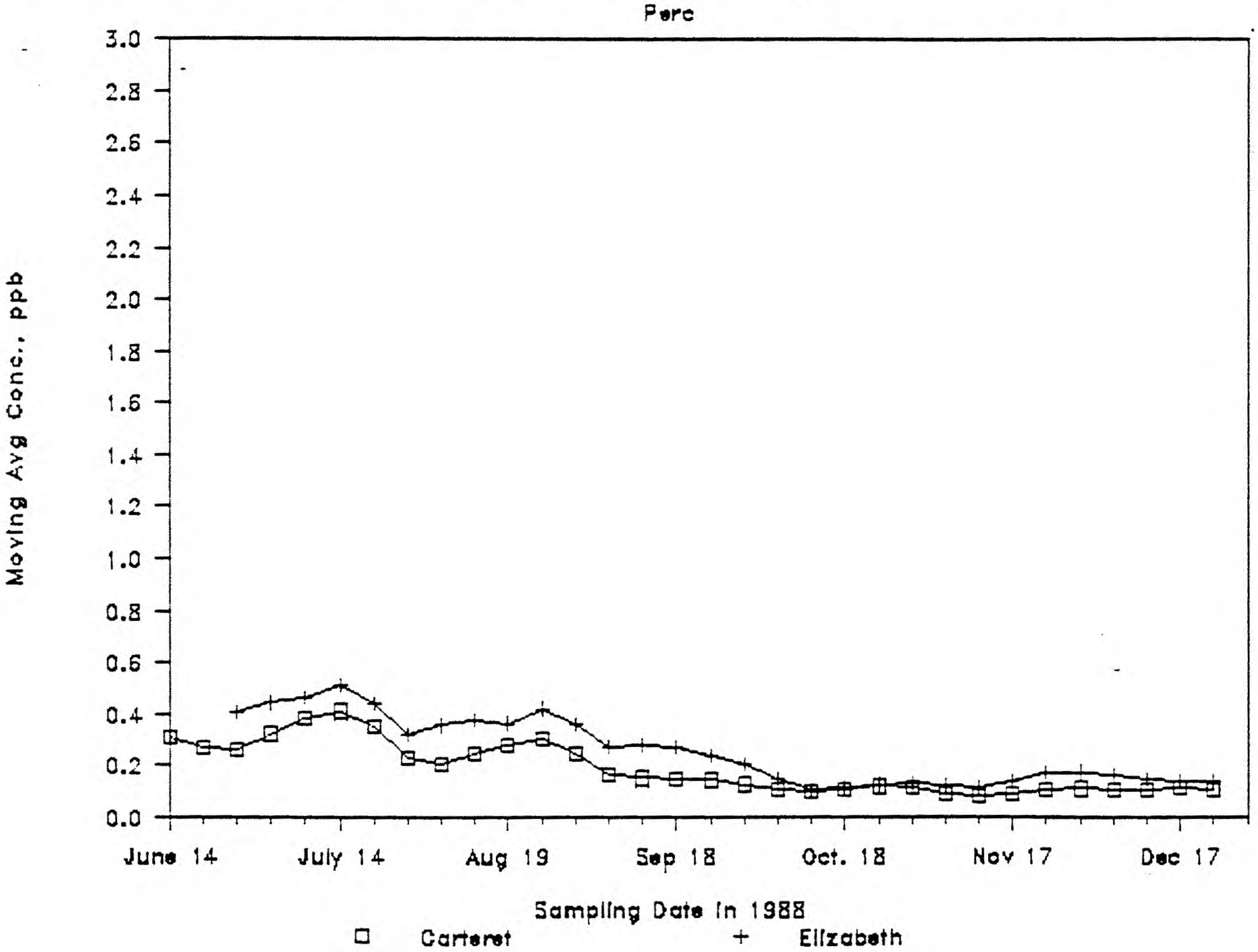


Figure 12 - 7 **Comparison of Moving Avg Between Site**

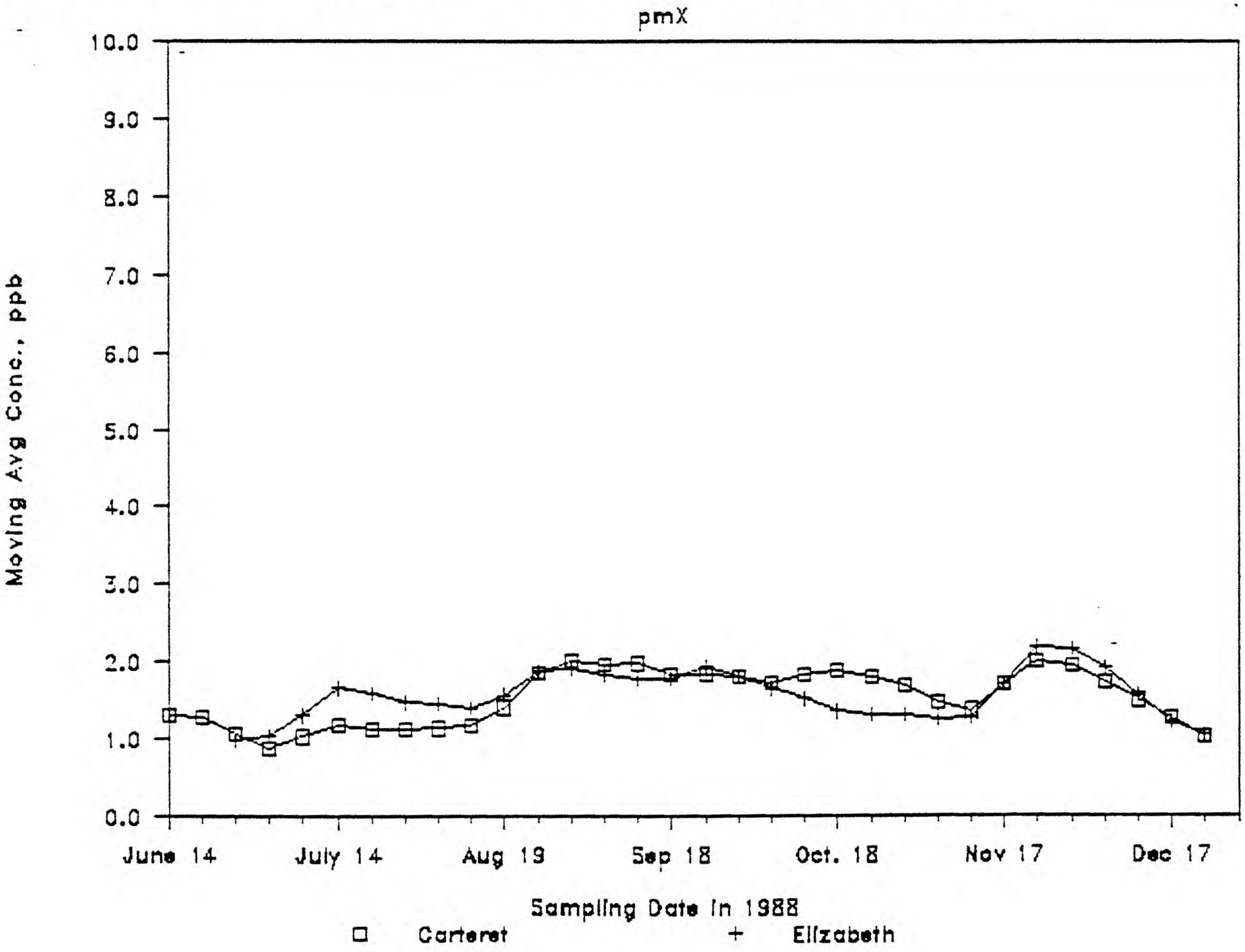
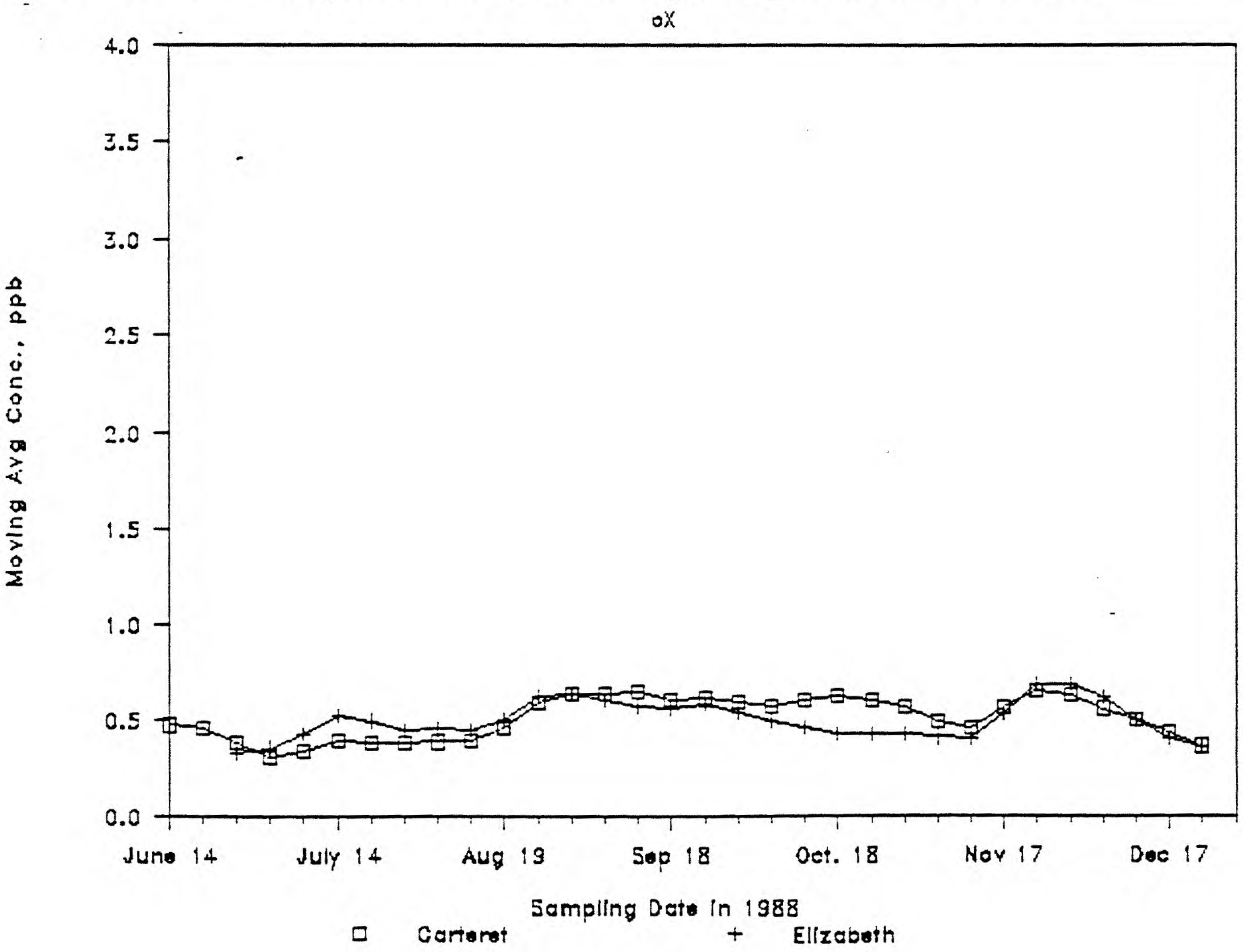


Figure 12 - 8 Comparison of Moving Avg Between Sites



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