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ENHANCED GC QUANTITATION OF  
ORGANO-CHLORINES

by  
B.F. Scott and M. Misunis<sup>1</sup>

Research and Applications Branch  
National Water Research Institute  
867 Lakeshore Road, P.O. Box 5050  
Burlington, Ontario, Canada L7R 4A6

<sup>1</sup> Hewlett-Packard (Canada) Ltd.  
1825 Inlister Blvd.  
Winnipeg, Manitoba R2X 1R3

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This manuscript is dedicated to the memory of Dr. P.D. Goulden

## MANAGEMENT PERSPECTIVE

The work described in this report was undertaken at the request of the National Water Quality Laboratory. They wished to optimize the operating conditions of their automated gas chromatography system. Individual components of the system were investigated separately and as part of the system as a whole. The ultimate aim was to achieve maximum response, good chromatography and high precision for organic compounds, using split/splitless and dual capillary column gas chromatography. Mixtures of 33 organo-halo compounds (including chlordane, hexachlorobenzene and p,p'-DDT) with serial dilutions of the mixtures, were used for this study, and over 45K data points were generated which were subjected to statistical analysis. As seen from the results presented here, maximum response for the compounds and a high degree of precision have been obtained using methods developed on the automated laboratory system of the NWQL. The initial request has been satisfied.

Dr. J. Lawrence  
Director  
Research and Applications Branch

## PERSPECTIVES DE GESTION

Le travail décrit dans le présent rapport a été entrepris à la demande du Laboratoire national d'analyses de la qualité des eaux, qui souhaitait optimiser les conditions de fonctionnement de son système automatisé de chromatographie en phase gazeuse. Les constituants du système ont été étudiés individuellement et en tant que composantes du système dans son entier. L'objectif ultime était d'obtenir une réponse maximale, une bonne analyse chromatographique et une haute précision pour les composés organiques, par injection à débit divisé/non divisé et utilisation d'une colonne capillaire double. Des mélanges de 33 composés organohalogénés (incluant le chlordane, l'hexachlorobenzène et le p,p'-DDT), et des dilutions sérielles de ces mélanges ont été utilisés pour l'étude; on a obtenu plus de 45K points, qui ont été analysés statistiquement. Comme en font foi les résultats présentés ici, une réponse maximale et un haut niveau de précision ont été obtenus pour les composés analysés grâce aux méthodes mises au point sur le système automatisé de laboratoire du LNQE. La demande initiale a donc été traitée de façon tout à fait satisfaisante.

## ABSTRACT

At the request of NWQL, a study was initiated to improve the reproducibility of the results obtained from the new automated gas chromatography laboratory, where the instruments are controlled by an HP 1000 computer. The initial criterion was that the coefficients of variance for each chromatographic peak was to be 5% or less for ten consecutive injections. The conditions of operation were splitless mode injection and dual column chromatography. The standard compounds were pesticides and chlorobenzenes contained in the hexane and benzene eluates of the clean-up stage of aquatic samples. Also bromobenzenes and hexa-chlorobutadiene were included as these were surrogates added to actual samples. The reproducibility of the automatic injector was first checked and CV for two test sequences was less than 1%. Operating conditions were then altered as was the integration parameters and this resulted in CV's of 5% or less for 33 of the 36 compounds.

Four column types were tested, SPB-1, SPB-5, thin phase SE-52 and the polar SPB608. The thin phase column and the polar columns exhibited apparent losses of resolving power when several of the peaks co-eluted. Serial dilutions were conducted over a decade range of concentrations. The resulting values gave correlation coefficients usually over 0.99 and calculated minimum of  $0.1 \times 10^{-12}$  g of compound impinging on the detector for many of the detectable amounts of chlorinated organics.

## RÉSUMÉ

À la demande du LNQE, une étude a été entreprise pour améliorer la reproductibilité des résultats obtenus grâce au nouveau laboratoire automatisé de chromatographie gazeuse, où les appareils sont commandés à l'aide d'un ordinateur HP 1000. Au départ, on voulait que les coefficients de variance pour chaque pic chromatographique soient de 5 % ou moins pour dix injections consécutives. Le chromatographe fonctionnait en mode injection à débit non divisé, avec colonne double. Les composés étalons étaient des pesticides et des chlorobenzènes contenus dans les éluats d'hexane et de benzène de l'étape de fractionnement des échantillons aquatiques. Il y avait également des bromobenzènes et des hexachlorobutadiènes, ajoutés aux échantillons réels sous forme de substituts. On a d'abord vérifié la reproductibilité pour l'injecteur automatique : les CV pour deux séries d'essais étaient inférieurs à 1 %. Les conditions de fonctionnement furent ensuite modifiées, tout comme les paramètres d'intégration, ce qui donna des CV de 5 % ou moins pour 33 des 36 composés.

Quatre types de colonnes ont été expérimentées : SPB-1, SPB-5, SE-52 à phase mince, et SPB608 polaire. La colonne à phase mince et les colonnes polaires accusaient une perte apparente de leur pouvoir de séparation lors de la co-élution de plusieurs des

pics. Des dilutions sérielles ont été effectuées pour une dizaine de concentrations. Les valeurs obtenues correspondaient à des coefficients de corrélation généralement supérieurs à 0.99 et à une quantité minimale calculée de  $0.1 \times 10^{-12}$  g de composé pour le détecteur dans le cas d'un grand nombre de montants décelables de substances organiques chlorées.

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## 1.0 INTRODUCTION

Gas chromatography is and has been since its beginning, a technique used for characterizing and quantifying organic compounds. Extensive modifications have been applied to the original method to expand the range of application and of quantification. The introduction of narrow bore columns has increased the efficiency, thereby allowing better separation of components in complex mixtures. The large number of bonded phases now available for capillary columns enables better resolution of the components of similar composition in multi-component systems. The use of automatic samplers should enhance the reproducibility of the results, as well as allowing unattended operation. The increased amount of data obtained from capillary column analysis and the control of the sampler and data collection invariably requires the use of an automated system which can also facilitate data interpretation. Indeed, a number of gas chromatographs each equipped with several detectors can be operated from a central computer, which will govern the injection of samples, ensure the gas chromatograph is operating with the correct method, collect the data, manage the data after the run is complete by invoking the necessary post-run programs, control the printout of the data, store the data and provide hard copies of necessary chromatograms.

This seems quite simple and straightforward, but this is not the case. The person operating the system must be quite conversant with modern gas chromatography, be able to effectively use the existing operating system and be familiar with the manufacturer's computer calls and terminology, as well as being capable of programming and/or using the sophisticated programs that can be used in post-run operations. Each of these aspects requires some specialization. Gas chromatography is still an art, which requires selecting the optimum conditions of the wide range of parameters under the chromatographer's control. Even the mundane tasks of changing needles, septa, positioning columns correctly for the type of injection and installing guard columns require some

expertise. If a new GC method is introduced to the laboratory, that method must be fine-tuned on the operating system so the linearity, reproducibility and minimum detectable amounts are within acceptable required limits.

This manuscript is the result of attempting to quantify a method for analyzing toxaphene, which using a similar but different gas chromatograph gave coefficients of variation (CV) less than 5% for the peaks of interest (Ryan and Scott) in splitless mode. When the method was transferred to the equipment discussed in this article, the CV's were greater than 15%. This report will outline the problems encountered and their resolution to produce results as good if not better than those reported in the literature.

For this work laboratory standards were used exclusively. If mixtures of standards cannot give reproducible results of high precision then extracted standards will give less precise results and the accuracy of the analysis of environmental samples would be suspect. In addition, the results presented here will serve as "bench-marks", permitting calculation of losses through the clean up steps and allowing calculation of the response factors for each of the components in the mixtures.

The criteria used for reproducibility will be the coefficient of variance, which is the standard deviation divided by the mean. This allows immediate comparison between results.

## 2.0 EQUIPMENT AND METHODS

### 2.1 Equipment

All chromatograms were run on an HP 5890 gas chromatograph equipped with a split/splitless injector and two Ni<sup>63</sup> EC detectors. Peripheral equipment included a HP 7673 automatic sampler and HP 3392A integrators, one attached to each detector for dual column work. The ends of two capillary columns were inserted through the bottom of the injector system so each would analyze the same sample. The gas chromatograph, the integrators and the injector were controlled by the

Laboratory Automation Systems (LAS) of the RTE-A software of an HP 1000 computer which has a 125K disc drive capacity. Peripheral equipment includes an HP 2780 tape drive, an HP 2934A printer, a 7550A plotter and a 2623a terminal.

## 2.2 Chromatographic Conditions

In all instances, hydrogen was used as the carrier gas and argon:methane (95:5) was the makeup gas introduced just before the detector. For the initial runs, the carrier gas column head pressure was 72 kPa and the makeup gas had a pressure of 107 kPa. Initially, the program employed for toxaphene analysis was used. This had an initial temperature of 80°C which was held for three minutes, then increased to 150°C at 20°C/min. The rate was then lowered to 2°C/min to a maximum temperature of 260°C. This final temperature was held for 10 min. The purge time was set at 0.8 min. As some compounds were retained on the column for too long a time resulting in peak broadening, other programs were used and the parameter changes are described in the text.

## 2.3 Columns

Three suppliers provided the columns used in this work. A megabore column (.53 mm x 15 m) was supplied by Hewlett-Packard and used exclusively for the initial work on injector reproducibility. SUPELCO (Canada Ltd.) provided capillary columns with liquid phases of SPB1, SPB5 and SPB608 (DB-1, DB-5 and DB-225). These were 30 m long and .22 mm i.d. with a film thickness of 0.25  $\mu$ . HIRESCO (Can.) supplied a 30 m long thin film (0.100  $\mu$ ) XE-52 (DB-5) column. These columns were conditioned prior to use in the gas chromatograph.

## 2.4 Standards

Toxaphene was obtained from the National Bureau of Standards and made up in iso-octane (B&D) such that a 1  $\mu\text{L}$  injection would correspond to an injection of a  $10^{-9}$  g/ $\mu\text{L}$  solution. The following chlorobzenes and organo-chlorine pesticides were used in all standards: 1,2-, 1,4- and 1,2-dichlorobenzene, 1,3,5-, 1,2,4- and 1,2,3-trichlorobenzene, tetrachlorobenzene, pentachloro- and hexachlorobenzene,  $\alpha$ -,  $\gamma$ -, and  $\delta$ -BHC, HEPTACHLOR, ALDRIN, HEPTACHLOREPOXIDE, alpha- and gamma-CHLORDANE,  $\alpha$ - and  $\beta$ -ENDOSULPHAN, MIREX, METHOXYCHLOR, DIELDRIN, ENDRIN, ENDRIN KETONE, p,p'-DDE, o,p'-DDT, p,p'-TDE, and p,p'-DDT. Calibration standards also included 1,3-dibromobenzene, 1,2,3-tribromobenzene, tetrabromobenzene, hexachlorobutadiene and 1,2,3,4-tetrachlorobiphenyl as these chemicals were used as spikes in other studies. These compounds were obtained as crystals from the ES EPA Depository, Research Triangle Park, EPA, N.C. A weighed amount of crystal was made up in a benzene solution, and aliquots of these were diluted in iso-octane solutions. the final composite solutions contained from 100 to 5 ppt in solution, which was nominally designated as the parent solution. Other solutions were then made up containing concentrations of 10, 20, 30, 40, 50, 60, and 70% of the components of the parent solution, all in iso-octane. For single column work, 1  $\mu\text{L}$  was injected and for dual column work, 2  $\mu\text{L}$  were injected. Retention times were established by injecting one component solutions of each standard. Initial work on the reproducibility of the injector was performed using lindane and aldrin in iso-octane. This three component solution was supplied by Hewlett-Packard.

Statistical analysis of the results were conducted by using standard formulae. The formula for least square fit of lines were taken from Long and Winefordner (1983), but the eight formulae given on page 716A of this reference contain at least three errors. The denominator for the slope should read the sum of the squares of the concentrations

multiplied by the number of values, minus THE SQUARE OF THE SUM OF THE CONCENTRATIONS. The second term of the numerator of the standard deviation ("s") should be multiplied by the slope "m", not the unidentified value "b". Thirdly, the formula for  $s_j$  is wrong. We found that this value should be calculated as  $s_m$  times the square root of the sum of the squares of the concentrations, all divided by the square root of the number of observations. These formulae, along with other formulae used in this study are listed in Table 1.

### 3.0      RESULTS

#### 3.1      Reproducibility of the Injection System

As the reproducibility of the injection system for single component systems should be below the maximum acceptable precision for subsequent analysis, this aspect was the first phase of the present investigation to be checked. The specifications for the injector are quoted for a flame ionization detector. Since electron capture (EC) detectors were used in this work, the manufacturer agreed that the test solution of lindane and aldrin would be suitable. Also the specifications required quantitation on a megabore column, using split mode (10:1) with the gas chromatograph operating isothermally (170°C). Listed in Table 2 are the results from ten separate injections from three vials. The first six came from one vial, the next two came from the second vial and the last two came from the third vial. These results were obtained from using vials that were loosely capped with the vial cap being removed before injection then replaced. The cap was removed just before injection as there was a small impurity introduced from the vial septum. The CV's of 1.6% and 1.38% for lindane and aldrin respectively were better than the manufacturer's specifications. The increasing values for the first six readings are caused by evaporation of the iso-octane solvent. By plotting the signal against the time of injection, a reasonable straight line can be drawn, whose slope predicts an enhancement of the signal by 409 counts/min for lindane and

and 500 counts/min for aldrin. Taking this factor into consideration, and using the six values, CV's of 0.5% and 0.44% are calculated for lindane and aldrin respectively. These values indicate two very important points: first, the injector does give reproducible results and second, the split of the sample is reproducible.

The next step was to introduce temperature programming. A simple program starting at 140°C and rising 2°C/min to 180°C was used. Two injections from each vial were made. Prior to this, four runs were made using the program with only the iso-octane blank used. There were no discernible peaks in the chromatogram resulting from material from the vial septa. The results from injecting the lindane-aldrin mixture are shown in Table 3. CV's of 1.4 and 1.5% for lindane and aldrin respectively were obtained.

The final step was to utilize the splitless mode, capillary columns and a temperature programmed run. The program had a 2 min initial hold at 80°C then programmed at 10°C/min to 140°C and the temperature increased at 2°C/min until 200°C and then ramped to 250 at 10°C/min with a 5 min final hold. Results from this procedure are given in Table 3. CV's of 2.4% and 2.7% were calculated. During this procedure the contents of each vial were injected twice. If the results from the first of the two injections were considered, CV's of 1.4% and 1.5% would be obtained but these values are based only on five points.

These results show that the injector does give reproducible results in both split and splitless mode, isothermal and programmed runs. The CV's are well below the maximum value acceptable for quantifying organic pesticides and halobenzenes on the gas chromatograph.

### 3.2 Single Column Mode

#### 3.2.1 Establishing the method

##### 3.2.1.1 Column head pressure

The request from NWQL was to develop a method that could analyze the organo-chlorine pesticides that elute in the benzene and

hexane eluates of normal column chromatography cleanup as well as the chlorobenzenes and the organo-chlorines that elute in the hexane fraction of the cleanup procedure (IWD). The coefficient of variance should be below 5%. This necessitated a three ramp temperature program whose parameters are listed in Table 5. For the studies on purge times and column head pressure only, the final temperature was set at 300°C with a 1 min final temperature hold. Otherwise, the method used in Table 5 was used for all subsequent work. As the retention times will alter as parameters are changed, the peaks are identified by letter and this coding is given in Table 6, as well as being included in several other tables. Table 7 lists the area response and the CV's for each of the components in the solution at the 100% level and at the 50% in the solution for a head pressure of 25 psi. The CV's are generally below 5%. Table 8 contains the results from altering the head pressure on the column, decreasing by 5 psi each series of runs using the nominal prime standard. The optimum result appears to be at 20 psi, the value that was used for the rest of the study.

### 3.2.1.2 Purge time

The next parameter altered was the purge time, which was decreased at intervals of 0.3 min. These results are shown in Table 9, and they show that there is little difference caused by altering the purge time. This can quickly be realized, by adding or subtracting the standard deviation to the mean and comparing the resulting value to a similarly treated mean for the same compound. By doing this to a series, one with an early retention time (less than 4 min), one with intermediate time (about 12 mins) and one late in the chromatogram (20 min), it is evident that increasing or decreasing the purge time does not favour the introduction of the volatiles over those compounds with lower vapour pressures.

The initial program used had a 20°C/min final ramp which caused a marked increase in the baseline either caused by excessive

bleed from the column or a decrease in the gas flow into the detector. Although the instrument can be run on a column compensation mode, this type of operation was not used as it assumes that the compensation will be identical for all runs, an unlikely prospect. The final ramp was decreased to 10°C/min. Also the final temperature was set at 300°C, slightly higher than the column manufacturer's recommendation. To a first approximation, the higher temperature should not affect the column, as very little oxidation should occur with the hydrogen carrier gas. However, resolution of the peaks decreased quite rapidly, so a maximum temperature of 260° was utilized. A typical chromatogram is shown in Fig. 1

### 3.2.1.3 Negative peaks

Another factor was the effect of the solvent on early eluting peaks. After the initial solvent peak, there was a pseudo-negative peak. The integrator would automatically set the baseline at the minimum of a negative peak giving false area values until the baseline was re-established at the minimum of positive peaks. Therefore, integration of the chromatogram was started after the initial negative peaks.

## 3.3 Single Column Mode

### 3.3.1 SPB1 column (OV-1 type)

After confirming that good reproducible results could be obtained using the SPB-1 column, all further work was carried out with a 1m guard column installed just before the working column. At this time the standard solution was changed. The new standard contained bromobenzene surrogate spikes of high concentrations. Use of this standard was beneficial for the following reason. The spikes were excessively high and partially overloaded the column, producing peaks with tails as shown in Fig. 2. The elution time for 1,2,4-trichlorobenzene was 0.08 min before the 1,2-dibromobenzene, with 1,2,3-dichlorobenzene being

eluted on the tail of the large peak, 0.35 min later. This condition would be similar to those encountered in an environmental sample where extraneous unknown peaks may occur in the chromatogram. The means for each component in the standard solution and the CV is listed in Table 10 along with the same values obtained from analyzing seven different dilutions of the standard which lists the mean responses and the CVs. The results are presented in Table 9. The 1,2,4-trichlorobenzene peak has a CV that is reasonable, averaging 4.4% over this series of diminishing concentrations. The values for methoxychlor are the only ones that continually exceed the desired level of 5% for the CV. Using linear regression analysis, whose principal elements are defined in Table 1, on the mean values contained in Table 10, the appropriate constants for each compound studied were calculated and are listed in Table 11. For 26 of the 32 compounds, the correlation coefficient is greater than 0.99, and this indicates the detector response is linear over the concentration range investigated. The minimum detectable amount ( $C_i$ ) is generally five times less than the lowest concentration injected. However, the smallest amount detectable is about a factor of 10 less than the lowest concentration injected. The  $C_i$  value is derived from a statistical treatment of the data, whereas the least detectable amount results from the deflection when drawing the chromatogram and the deflection is greater than the background noise.

### 3.3.2 SPB5 column (DB-5 type)

The SPB-1 column was replaced with an SPB-5 capillary column equipped with guard column and ten injections of the standard and each of its dilutions were made. These results are listed in Table 12. As shown on the chromatograms in Figure 3 and in this table, Aldrin co-elutes with the tetrachlorobiphenyl on this column. The means listed in Table 12 were subjected to a regression analysis and these results are shown in Table 13. As in the previous case, the high value of the correlation coefficient indicates that the detector response over the concentration range studied is linear.

### 3.4 Dual Column Mode

Both SPB-1 and SBP-5 columns were then installed with guard columns in the gas chromatograph (dual mode configurations) and the same series of standards were analyzed. The results are presented in Tables 14 and 15. The SPB-5 column results are slightly different from those listed in Table 12. This results from SPB-5 column being connected to Detector A and this detector gives a slightly different response than Detector B. However, the results in the two tables indicate that the injected sample is roughly split evenly between the two columns. The result for both columns were subjected to linear least squares analysis and these results are given in Tables 16 and 17. Chromatograms are similar to those shown in Figures 2 and 3.

The next step was to use an extremely polar column and a thin liquid phase column from another supplier. The polar column selected was a SPB-608 and with the other being a thin phase XE-52 column (DB-5 type). Results obtained from these columns are presented in Tables 18 and 19. Typical chromatograms are shown in Figures 4. The SPB-608 column does not effectively separate all the components of the solution as effectively as the SPB-1 column as there are two sets of peaks that are co-eluted. The SPB-608 column is the most polar column of the four tested. The CV's for these two columns are in the range that indicate good reproducibility of the system, especially in splitless mode. Linear regression analysis results for the contents of the standards run on these two columns are listed in Tables 20 and 21.

### 3.5 Aspects Which Influence GC Results

In the previous sections, the results presented were the culmination of many hours spent determining the optimum operating conditions. These include final sample preparation, instrumentation, sample injection and integration. It is important each aspect is

discussed at this point if the precision reported in this report is to be replicated.

### 3.5.1 Crimping of the vials

For unattended chromatographic runs, it is necessary to prepare a number of vials for injection that have properly crimped tops. This aspect is extremely important for good reproducibility. Several runs were made where the vial caps were crimped too tightly. The reproducibility was poor when this occurred, as compared to the degree of tightness which was used for low CV values. We found the optimum degree of tightness to be with the vial cap crimped snugly on the vial, with only a slight resistance to turning of the cap. On the other extreme is the instance where the caps are loose and allow evaporation of the solvent and/or sample. In a series of ten samples that are tightly crimped because of an improperly set crimper, two or three may produce extraneous results, but this is enough to increase the injections coefficient of variance. A comparison between the results of vials capped correctly and those which are crimped too tightly is given in Table 22.

Also, if the caps are crimped, as suggested here, with iso-octane as solvent and components with reasonable vapour pressures, they should be able to sit on the sample tray without noticeable alteration of composition for about a day. If the instrument or computer fails and analysis delayed, some of the contents of the vials may evaporate in a non-uniform manner, producing results which give poor reproducability.

### 3.5.2 Injector

Contamination of the injector can cause peak broadening or early humps in the chromatogram. This results in poor chromatography and difficulties in integrating the chromatogram. The injector should be stripped and rinsed as soon as this condition is observed.

### 3.5.3 Amount injected

Using splitless mode chromatography to introduce the sample to the column means the entire amount of solvent is being applied to the system. The larger the amount of solvent injected, the greater the distortion on the leading edge of the chromatographic peaks. This is shown in Fig. 5 where 1, 2, and 3  $\mu$ L of a standard were injected. Another factor that was apparent was the affect on retention times of the early eluting peaks. Table 23 lists the retention times of the three dichlorobenzenes, three tri-chlorobenzenes and a tetrachlorobenzenes. As the volume of the injection increases, so does the retention time. This is documented in Table 24. After six minutes, no effect is noticed. Similar behaviour was observed when 1, 2, and 3 $\mu$ L injections of 50% concentration of the standard solution were made. A sample size of 1  $\mu$ L per column is recommended.

### 3.5.4 Number of times a sample is injected

If a sample is injected more than once, more negative peaks can be anticipated in the resulting chromatogram. This results from small amounts of the vial septum being introduced to the vial contents when the needle penetrates this septum to withdraw a sample. The smaller the vial used (e.g., micro vial), the more apparent these negative peaks. Negative peaks have the effect of altering the character of the baseline, thereby affecting the integration and hence the reproducibility of the results.

### 3.5.5 Positioning of the column

This is usually adequately discussed in the manufacturer's material, however, it is useful to add a small section here. With the different types of injection that can be conducted on capillary columns (split, split/splitless and on-column) there is always a chance of a

mistake being made due to an oversight. When operating in split/splitless mode, the column should be positioned as low as possible in the injector. When the column was positioned where it should be for split mode (in the upper half of the injector) and ran in the splitless mode, the results were about half of those which were obtained from chromatograms where the columns were positioned correctly, as shown in Table 24.

### 3.5.6 Integration of signal

This aspect is perhaps the core of this report and as such is best discussed in a more extended manner.

Gas chromatography was initially used as a method to separate a mixture of organic compounds. When a trace of the eluant was obtained it was realized that this could be used to quantitate the compound under consideration. Initially, packed columns were used and the traces were quite broad. The quantitation could be carried out using the following techniques:

- counting area under the peak (if the peak was on graph paper);
- planimeter;
- cutting the peak out and weighing it on a balance;
- measuring the height;
- calculating area by multiplying height by width at half height.

Each of these methods relies on the chromatographer estimating the position of the baseline. Also the final two methods require that the peaks have Gaussian symmetry. All have a degree of subjectivity provided by the chromatographer.

With the advent of narrow bore columns, the wide peaks were eluted as thin spikes. The traditional methods of integration could not be used with a high degree of precision. This increased the need for automatic integrators. The built-in programs in these computers increased in complexity, taking the subjectivity out of the calculations.

By monitoring the electrical impulses, they could determine inflection points with better accuracy than human operators. They could also provide a position for the baseline based on electrical impulses. The more sophisticated the integrator, the better the baseline. However, at the time of this report, there are very few integrators on the market that will draw in the baseline in the chromatogram to show from where the peak is being integrated. This is important for chromatograms of complex mixtures where the peaks of interest may be sitting on a continuum of unresolved peaks or where there is a negative peak in the chromatogram which influences the position of the baseline. There are other techniques that a sophisticated integrator can utilize such as tangent skimming which is important when there is a peak on the leading or tailing edge of another peak. Variables such as peak width are important input parameters. The width of peaks in a temperature programmed run can vary, being extremely narrow near the beginning of the chromatogram and much broader near the end when the compound has been in the column for greater than 30 min. Although the manufacturer may suggest that a peak width setting of 0.4 min may be adequate, this may refer to peaks that elute early, whereas, a peak width of 0.8 min may be needed for a late eluting peak. Three peaks are shown in Figure 6, one that is early eluting, one that is late eluting and one that elutes between the two extremes. These are blowup representations of the actual peaks which appear in the original chromatogram as sharp spikes with a flat baseline under all three peaks. In (a), the baseline is slightly inclined under the peak, and if a flat baseline is drawn under the peak, the area difference between the two is less than 5%. For peak (b) the same procedure was followed and an error of 5% was determined. In case (c), the baseline followed by the integrator has the shape of an inverted "V". The error is calculated at 15%. Although the areas for each of the peaks differs from the idealized value, the reproducibility depends on how well the integrator will replicate its positioning of the baseline under each peak. presuming identical amounts were injected. A slight difference will affect its position and this is more important in

the case of (c) than (a) or (b). With the peak width set at a higher value, the inverted "V" disappears, but it should not be set so high so that the peak widths are similar to those related to peaks from packed column chromatography.

#### 4.0 DISCUSSION

The objective for this study was to ensure the system (GC and computer software packages) was working in a manner that would yield good reproducible results. The original agreement was that the Water Quality Branch staff would be responsible for the basic maintenance of the instrument but all other aspects, such as the GC methods would be under the control of the authors. This was done as the GC was one of many similar instruments and would receive the same treatment as the other instruments.

The outcome of this arrangement was to produce results, based on standards, that are extremely good. To achieve these results, considerable interaction had to occur between the NWQL and HP, the suppliers of the instrumentation. There was a significant time lag between the determination that the automatic injector gave reproducible results on the lindane and aldrin samples, and the reported quantitations of the standards. That was a learning time for both parties involved. Once the first set of reproducible results were obtained, the method and integration parameters were applicable to other standards of varying concentrations or other columns.

The reproducibility of the results as denoted by the CV's of 5% or less for all the pesticides and chlorobenzenes as well as the bromobenzenes in splitless mode is very good. Usually the repeatability is cited as much higher. The only exception is methoxychlor, with mirex occasionally having CV's greater than 5%. These two compounds are the last two eluted on the chromatograph. The columns selected for use are not perfect and there is some bleed at the higher temperatures

despite conditioning the columns according to manufacturer's specifications. The increasing background from the column bleed, especially at low concentrations, will lead to errors in the integration. Being the final two peaks, these compounds spend the greatest time in the column, so they would be most prone to broadening at the baseline. The particular liquid phase on the column may not give the desired chromatography for a particular peak, so that the integration may not be reproducible from run to run. It was decided that since all but one of the components give reproducible results, the method would be deemed acceptable.

Treatment of the results of the serial dilution of the standard by linear regression analyses produces several important findings. The first is the correlation coefficient result which is a measure how well a series of points define a specific equation. For the purposes of this study, we found it advantageous to consider a linear relationship. With correlation coefficients generally greater than 0.99, the assumption or consideration is as good as any other and indicates that the detector response is linear for these compounds. The next factor is the slope of the regression analysis, which is directly related to response and inversely to concentration.  $C_j$ , the minimal detectable amount, is primarily derived from the error associated with defining the intercept. Normally, a blank measure is involved as a term in the calculation of  $C_j$  but during this study, there was no response unless a compound peak was present. Accordingly, the blank value was set at zero.

All  $C_j$  values are tabulated in Table 25. The first two columns are for the two single column studies, and there is little difference between the  $C_j$  values for the two columns. When the two columns are run simultaneously, there is an increase in the  $C_j$  value, generally by a factor of 2. The other set of columns run simultaneously produced results similar to the first set of columns. Changing columns may result in a change in ability of the columns to separately elute the compounds, but does alter the minimal detectable amounts.

We have demonstrated the ability of the system to produce reproducible results in single and dual column mode, with several types of columns. Also, we have produced minimal detectable amounts for the OCs which are more than adequate for routine analysis. In short, the objectives of the study have been satisfied.

## 5.0 RECOMMENDATIONS

1. Crimping of vials: For good reproducible results, the caps on the sample vials should be snug, but not tight. There should be no distortions on the top metal surface. There must be some control on this seemingly unimportant aspect as the time collecting, extracting, cleaning up, and analyzing the sample will be wasted if the results are wrong.
2. No more than 1  $\mu\text{L}$  per column should be injected into the gas chromatograph, assuming an equal split into the columns. Amounts greater than this can cause distortion of the peaks.
3. No more than one injection per sample vial through the same vial septum should be made. If a second analysis is needed, a second septum should be inserted, or better yet, the sample should be placed in two vials. This is done to reduce the occurrence of negative peaks attributable to vial septum material.
4. Chromatograms of standards in which the peak shapes are less than ideal indicate that the columns need to be altered or the injector needs cleaning.
5. The peak width parameter should be wide enough to adequately integrate the peak regardless of temperature. As peaks tend to broaden with increasing time in the chromatograph, this parameter can be changed during the run. The actual amount can be determined from the standards.
6. If the coefficient of variance increases to a value greater than 10% for more than 10 of thirty peaks, the calibration data should be investigated.

7. The LAS package on the HP 1000 is user friendly but was probably developed for samples less complicated than trace organics in environmental samples. As such, the operation of the system is more complicated than simply using it as a hands-on operation. Accordingly, each operator should receive proper training from the equipment manufacturer.
8. Equipment manufacturers and their service and sales personnel often communicate in the jargon of the instrument. To uninitiated or partially-conversant personnel, this may not be comprehended as it should, and salient points could be glossed over. This is one of the main reasons the laboratory took so long to come on-stream. Now the competence exists in the laboratory. It is recommended that at least two people be familiar with the equipment so that if for some reason one leaves, it would not cause a major set-back while someone else was being trained.

#### ACKNOWLEDGEMENTS

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#### REFERENCES

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TABLE 1

$$(n \sum_{j=1}^n c_j x_j) - ((\sum_{j=1}^n c_j)(\sum_{j=1}^n x_j)) = A$$

$$(n \sum_{j=1}^n c_j^2) - (\sum_{j=1}^n c_j)^2 = B$$

$$(n \sum_{j=1}^n x_j^2) - (\sum_{j=1}^n x_j)^2 = C$$

$$\text{SLOPE} = A/B = M$$

$$\text{INTERCEPT} = \frac{(\sum_{j=1}^n x_j)}{n} - M \sum_{j=1}^n c_j$$

$$\text{CORRELATION COEFFICIENT} = \frac{A}{(BC)^{1/2}}$$

$$\text{STANDARD DEVIATION} = \frac{1}{\sqrt{n}} \frac{(C-MA)^{1/2}}{n-2} = S$$

$$S_m = \frac{S}{(\frac{B}{n})^{1/2}}$$

$$S_i = \frac{S(\sum c_j)^{1/2}}{\sqrt{n}}$$

$$\text{LIMIT OF DETECTION} = \frac{3}{M} (S_i^2 - (\frac{1}{M})^2 S_m^2)^{1/2}$$

TABLE 2

REPRODUCIBILITY OF INJECTION SYSTEM  
(SPLIT INJECTION, ISOTHERMAL, MEGABORE COLUMN)  
CRUDE RESULTS

RT	002	003	005	006	008	009	010	012	014	015
0.89	774044	781050	784630	789330	797550	813820	806670	814730	792218	800030
2.03	1048600	1061800	1062900	1073900	1080900	1098000	1091800	109390	1071000	1076680

AVERAGE:  $R_T$  0.89  $793276 \pm 13040$  (1.6%)  
 $2.03$   $1075500 \pm 14870$  (1.38%)

TREATED RESULTS

RT	002	003	005	006	008	009
0.89	774044	778600	774830	774630	773050	784420
2.03		1061800	1060400	1061400	1058400	1070500

AVERAGE OR MEAN: 0.89 =  $777106 \pm 4568.6$  (0.5%)  
 $2.03$  =  $1062500 \pm 4661.5$  (0.44%)

TABLE 3

REPRODUCIBILITY OF INJECTION SYSTEM  
(TEMPERATURE PROGRAMMED, MEGABORE COLUMN, SPLIT MODE)

RT	008	009	011	012	014	015	017	018	020	021
12.27	5777900	5613000	5631300	5578300	5506800	5617400	5530000	5440900	5410600	5295300
17.78	5626680	5493700	5476400	5437400	5294900	5440600	5403600	5292200	5255400	5129100
MEANS	RT 12.27	X = 5540150	$\pm$ 13555 (2.4%)							
	17.78	X = 5384798	$\pm$ 14275 (2.7%)							

TABLE 4

REPRODUCIBILITY OF INJECTION SYSTEM  
(SPLITLESS MODE, CAPILLARY COLUMN, TEMPERATURE PROGRAMMED)

RT	005	006	008	009	011	012	014	015	017	018	020	021
23.90	371800	322660	331200	309620	337420	355700	326380	312880	338220	331640	338820	359440
29.19	638940	527730	524680	492780	536180	548170	496280	469130	504520	491050	500720	523780
<b>AVERAGES OR MEANS:</b>												
RT 23.90	X <sub>11</sub>	= 353090	$\pm$ 15498 (4.7%)									
29.19	X <sub>11</sub>	= 510454	$\pm$ 23429 (4.6%)									

TABLE 5

INITIAL GAS CHROMATOGRAPHIC CONDITIONS

INJECTOR TEMP: 250 <sup>o</sup> C	DETECTOR TEMP: 300 <sup>o</sup> C
INITIAL TEMP: 80 <sup>o</sup> C	INITIAL TIME: 2 min
PURGE TIME: 1.50 min	DETECTOR: ELECTRON CAPTURE
INITIAL (A) RAMP: 10 <sup>o</sup> C/min	FINAL (A) TEMP: 170 <sup>o</sup> C
FINAL TEMP. (A) HOLD : 0.0 min	
SECOND (B) RAMP : 2 <sup>o</sup> C/min	FINAL (B) TEMP: 190 <sup>o</sup> C
FINAL TEMP. (B) HOLD : 0.0 min	
THIRD (C) RAMP : 20 <sup>o</sup> C/min (10 <sup>o</sup> C/min)	FINAL TEMP : 300 <sup>o</sup> C (280 <sup>o</sup> C)
FINAL TEMP (C) HOLD: 1 min	
CARRIER GAS: Hydrogen	PRESSURE: 25 - 10 psi
FLOW RATE : 5 mL/min	
MAKE-UP GAS : Argon/Methane (95/5)	
INSTRUMENT RUN ON INET (DL92)	PEAK WIDTH : 0.08
ATTENUATION : 4	SLICE WIDTH : 0.25
RANGE : 1	
<b>INJECTOR</b>	
No. of WASHES: 3	NO. of PRERINSES: 10
VOLUME INJECTED: 1 mL/COLUMN	VISCOSITY: 2
No. of POST RINSES: 3	No. of POST RINSES: 3

TABLE 6

Compound	Code
1,3-Dichlorobenzene	A
1,4-Dichlorobenzene	B
1,2-Dichlorobenzene	C
1,3,4-Trichlorobenzene	D
1,2,4-Trichlorobenzene	E
Dibromobenzene	F
1,2,3-Trichlorobenzene	G
Hexachlorobutadiene	H
1,2,3,4-Tetrachlorobenzene	I
Tribromobenzene	J
Pentachlorobenzene	K
$\alpha$ -BHC	L
Hexachlorobenzene	M
Tetrabromobenzene	N
$\gamma$ -BHC	O
$\delta$ -BHC	P
Heptachlor	Q
Tetrachlorobiphenyl	R
Aldrin	S
Heptachlorepoxyde	T
$\gamma$ -Chlordane	U
$\alpha$ -Endosulphan	V
$\alpha$ -Chlordane	W
Dieldrin	X
p,p'-DDE	Y
Endrin	Z
$\beta$ -Endosulphan	AA
p,p'-DDD	BB
$\alpha$ ,p'-DDT	CC
p,p'-DDT	DD
Endosulphan ketone	EE
Methoxychlor	FF
Mirex	GG

TABLE 7

PEAK AREA AND PEAK HEIGHT RESULTS  
 WITH COEFFICIENT OF VARIATION  
 (PRESSURE IS 25 PSI, PURGE TIME IS 1.5 MIN)

RT	IDENT.	100%		50%			
		PEAK AREA	HEIGHT	AREA	HEIGHT		
1.49	A	188679	0.7	45581	0.8	98875	3.1
1.65	B	120440	1.1	37491	1.0	60491	2.6
2.59	C	80812	0.6	26440	1.1	42595	1.8
3.06	D	70009	0.6	21529	0.8	34916	0.9
3.46	E	112883	0.6	36992	0.9	54903	2.0
3.72	G	153242	1.7	65959	2.3	64649	0.9
5.63	K	140312	1.0	41931	1.5	67291	1.8
7.52	L	172664	1.4	51732	2.3	81644	2.0
9.41	M	66213	1.7	23477	1.9	31403	3.7
9.80	O	180784	2.3	56976	4.1	99596	2.4
10.09	Q	60129	2.2	21505	3.0	27881	4.5
12.28	S	85938	3.0	23809	4.0	39631	4.0
13.42	T	114823	1.0	29350	2.0	55383	3.1
14.74	U	212688	2.4	48224	3.3	97415	5.4
15.66	V	255026	1.6	56528	3.0	117097	4.6
15.96	W	351177	2.5	64528	4.2	172468	3.2
16.22	X	336408	2.1	71779	3.5	148969	6.4
16.43	Y	276354	1.6	58973	3.1	127281	4.1
17.41	Z	198320	1.6	39640	3.2	94040	4.0
17.75	AA	139638	2.2	24740	3.8	68948	3.2
18.24	BB	253750	3.7	49337	5.1	116043	4.8
18.42	CC	453163	3.0	88309	5.4	189823	7.9
20.06	DD	575800	17	109848	5.5	256259	4.5
21.42	EE	536297	4.8	167056	7.5	209377	5.2
22.78	FF	304952	4.8	121855	7.6	122122	7.7
23.40	GG	468483	2.4	179320	3.5	224688	6.9

TABLE 8  
EFFECT OF CHANGING HEAD PRESSURE ON GC  
(AREA RESPONSE AND COEFFICIENT OF VARIATION)

Compound	psi									
	25	20	15	10						
	CV	CV	CV	CV	CV	CV	CV	CV		
A	188679	0.7	178670	2.3	260337	2.4	194252	1.8	180486	25
B	120440	1.1	158416	2.7	157913	5.3	129974	2.7	185486	1.9
C	80812	0.8	89958	2.3	92024	2.1	85757	2.3	86231	0.6
D	70009	0.6	76242	2.4	81745	1.8	77127	1.7	75345	1.6
E	112883	0.6	123983	2.5	132952	1.8	123350	1.8	121418	0.8
G	153242	1.7	250186	3.2	272545	2.8	237933	2.5	230245	1.0
K	140312	1.0	147356	3.7	162063	2.1	150184	2.1	142793	0.9
L	172664	1.4	185855	5.1	204713	2.0	190359	2.1	160684	2.2
M	66214	1.7	116673	5.3	123374	2.8	111403	2.4	99237	2.0
O	180784	2.3	187538	12	242319	2.7	224390	3.3	221882	2.2
Q	60129	2.2	110880	6.2	118839	3.2	108255	2.7	111968	4.3
S	85938	3.0	130911	5.5	131563	3.5	131244	3.7	142484	2.6
T	114823	1.0	172394	6.7	172306	2.3	160788	2.3	155336	1.0
U	212688	2.4	362382	5.0	385434	3.2	377231	2.7	267118	1.3
V	255026	1.6	358955	5.6	400866	2.8	378118	2.7	343830	1.8
W	351177	2.5	340512	18	524978	3.8	503538	3.8	507205	1.8
X	336408	2.1	577847	5.6	602594	2.9	552365	3.1	551337	1.3
Y	276354	1.6	429197	4.8	419008	2.7	387902	2.6	431292	1.3
Z	198320	1.6	289997	5.7	314708	3.5	299527	2.7	495436	1.3
AA	139638	2.2	194409	12	230220	4.3	214127	3.3	69068	3.2
BB	253750	3.7	443104	7.9	498838	4.4	468149	4.5	376309	2.9
CC	453163	3.0	757847	7.8	943061	3.4	929484	3.2	853416	1.7
DD	575800	16?	999601	9.9	928260	7.1	884743	5.7	995998	3.1
EE	536297	4.8	893299	15	811298	8.6	725214	6.5	866139	3.8
FF	304952	4.8	414509	18	386070	9.8	329245	6.8	439977	5.7
GG	468483	2.3	714433	7.5	712985	3.3	659089	2.3	666988	1.5

TABLE 9

EFFECT OF CHANGING PURGE TIME  
 (CONSTANT HEAD PRESSURE OF 25 psi)  
 REPORTING IN UNITS OF AREA AND CV

COM- POUD	1.5 MIN CV	1.2MIN CV	0.9 MIN CV	0.6 MIN CV
A	178670 2.3	200813 2.6	203452 5.0	218340 0.8
B	158416 2.7	176690 3.9	171364 5.3	174660 1.3
C	8958 2.3	88321 2.0	90348 5.6	85183 0.4
D	76241 2.4	77636 2.1	80724 5.9	76029 1.0
E	123983 2.5	126778 2.0	130545 5.7	123437 0.6
G	250186 3.2	256903 2.8	267932 7.4	251641 0.8
K	147356 3.7	153595 2.2	159880 6.6	154117 1.0
L	185855 5.1	196392 2.8	202899 7.6	187200 1.7
M	116673 5.3	122553 2.8	126496 8.4	115562 3.3
O	187538 1.2	216383 4.4	226614 12	216262 3.4
Q	110880 5.5	119012 2.9	123155 8.6	115545 3.0
S	130911 6.7	143498 2.9	153403 6.9	143729 3.8
T	172394 5.0	182523 4.5	199398 13	185095 8.9
U	362382 5.6	382485 2.8	400944 6.8	371917 3.0
V	358955 18	376728 3.0	396575 7.2	368468 2.6
W	340512 5.6	413788 4.8	451135 14	425680 5.8
X	577847 5.6	605583 3.6	633668 6.7	583115 2.8
Y	429187 4.8	442802 3.3	462068 6.5	425179 2.7
Z	289997 5.7	306151 3.0	321403 6.8	297270 2.8
AA	194409 12	226439 3.3	240925 9.1	219165 3.3
BB	443104 7.9	470697 3.8	506680 10	478502 4.9
CC	757847 7.8	815739 3.7	867930 8.3	801406 4.6
DD	999601 9.9	1113070 3.8	1198100 9.0	1045453 8.0
EE	893299 15	1059350 4.3	1173880 12.0	1006739 11
FF	414509 18	513053 4.8	573849 13	494300 14
GG	714433 7.5	747004 3.3	771504 6.6	714156 3.3

TABLE 10

**RESPONSE (AREA) AND COEFFICIENT OF VARIANCE OF STANDARDS  
CHROMATOGRAPHED ON A SPB1 CAPILLARY COLUMN**

CODE	CONC of 100% SOLN. ( $\mu$ g/ $\mu$ L)	100%		70%		60%		50%	
			CV		CV		CV		CV
A	50	97891	0.7	71085	1.8	63090	1.2	54007	1.1
B	50	103597	0.3	86905	6.0	80220	5.7	67747	1.9
C	50	122531	0.7	109095	10	95062	13	83073	3.8
D	5	66217	2.0	48374	2.1	43480	2.3	34414	5.0
E	5	34597	32	26487	3.1	23627	4.9	19321	5.0
F	50	1127344	0.9	764397	1.0	667477	1.5	513587	1.2
G	5	81654	0.6	57935	1.4	51871	1.0	40765	1.6
H	5	244630	0.9	157426	0.9	136594	1.0	103512	1.0
I	5	110270	0.9	69430	7.9	64560	10	49971	4.3
J	20	708502	1.4	427733	1.2	367318	2.4	282635	1.3
K	5	143118	1.5	100695	1.3	90445	2.1	71573	1.5
L	5	94256	4.2	64511	6.3	56588	5.5	46650	3.9
M	5	213118	2.7	149008	3.4	135372	2.8	103646	2.3
N	20	497187	3.7	290607	4.2	257771	3.9	204331	2.6
O	5	113927	2.0	89902	2.9	80234	2.4	60301	1.9
P	10	135522	2.9	95988	2.8	86750	1.9	68646	1.6
Q	5	138934	3.5	96399	2.8	87873	2.8	70976	1.7
R	20	397943	5.6	272535	6.1	236397	4.0	212335	10
S	5	188702	6.5	134599	3.1	114090	2.4	96879	3.7
T	5	140248	2.9	101415	2.4	89344	2.4	73240	1.6
U	5	127062	3.5	90870	3.1	79990	3.1	66148	2.0
V	5	114442	3.6	81830	3.2	72535	3.2	60126	2.1
W	5	132699	3.6	94108	5.6	83891	3.1	69326	1.9
X	10	213120	3.8	153194	3.5	135410	3.1	112367	2.4
Y	10	136845	6.1	100851	4.6	95090	4.3	76189	3.2
Z	10	171307	7.0	118543	5.5	103203	7.4	91115	2.8
AA	10	165009	6.2	118360	5.4	106671	4.7	88950	3.0
BB	15	199827	9.3	141654	6.7	132540	6.8	111460	4.3
CC	15	352788	8.5	239955	6.3	219289	5.7	180635	4.0
DD	15	276148	11	202897	9.1	168593	8.5	182481	6.3
EE	10	147660	7.7	109167	7.3	97767	5.7	81336	3.9
FF	50	222241	17	144996	15	141248	11	124794	8.8
GG	10	265738	6.4	189614	6.1	171993	5.8	144903	3.7

TABLE 10 (CONT)

RESPONSE (AREA) AND COEFFICIENT OF VARIANCE OF STANDARDS  
CHROMATOGRAPHED ON A SPB1 TYPE CAPILLARY COLUMN

CONC of

CODE	CONC (pG/uL)	100% SOLN.		40%		30%		20%		10%	
A	50	45161	1.3	31170	3.9	19813	6.4	8953	5.5		
B	50	64614	2.6	47267	9.1	34291	19	27253	12		
C	50	73477	7.2	57384	16	30807	28	22972	28		
D	5	32753	17	41833	9.0	14552	7.7	6556	8.7		
E	5	16647	2.7	10453	11	7462	7.2	3890	3.2		
F	50	436303	1.8	272919	2.3	180524	1.2	83340	1.3		
G	5	35956	1.4	23948	1.5	16896	2.4	7993	1.8		
H	5	86933	6.8	58009	1.1	40850	0.8	20258	0.7		
I	5	44617	6.0	30249	3.4	22317	3.9	11275	2.4		
J	20	234415	2.4	146308	2.2	99702	1.5	48635	0.9		
K	5	62767	3.1	40890	3.0	28420	2.3	13524	3.0		
L	5	39008	5.4	27162	4.8	21984	2.0	9849	4.3		
M	5	93215	5.3	59064	4.4	41718	2.4	19522	2.3		
N	20	171817	5.5	105119	4.9	69717	3.6	32705	3.1		
O	5	56612	4.2	39258	2.4	32394	2.3	16486	2.1		
P	10	62685	4.2	41106	4.0	32724	2.0	15924	3.2		
Q	5	61799	3.6	41285	4.5	29196	3.0	13956	4.1		
R	20	170962	6.0	117581	17	80592	16	42553	24		
S	5	84877	4.6	56432	4.9	39372	6.5	21084	5.1		
T	5	63969	3.5	42013	3.7	29746	2.4	13989	1.5		
U	5	57357	4.5	38022	4.4	26756	2.7	12524	1.5		
V	5	52240	4.9	34593	4.7	24332	3.1	11420	2.3		
W	5	60002	5.0	39737	4.6	27947	3.0	13191	3.6		
X	10	97721	4.9	64413	5.0	45369	3.3	21501	2.4		
Y	10	67526	7.2	43497	6.8	30430	4.3	14759	3.8		
Z	10	78178	7.1	50364	7.4	34608	6.5	16610	3.7		
AA	10	77938	7.2	49726	6.6	34721	4.9	16253	3.1		
BB	15	98746	9.3	62208	9.7	41260	8.8	19676	5.5		
CC	15	152711	8.5	94936	8.6	66512	5.9	30569	4.2		
DD	15	118669	10	70237	14	48655	9.4	25614	28		
EE	10	71323	8.5	44931	9.1	31197	6.0	14372	4.1		
FF	50	105566	16	55039	18	36489	18	16525	12		
GG	10	120404	9.4	77512	9.8	51197	5.1	22645	13		

TABLE 11

LEAST SQUARES TREATMENT OF THE STANDARDS AND THEIR DILUTION  
FOR THE SPB-1 COLUMN ON DETECTOR B

CODE	COMPOUND	SLOPE	INTERCEPT	STD DEV	$C_i \times 10^{-12} g$	COR COEF	Sm	Si
A	DICHLORO	2000.7	1269.0	2542.9	2.2	0.997	57.0	1472.3
B	DICHLORO	2008.9	14465.6	8412.3	7.3	0.971	188.6	4870.3
C	DICHLORO	2521.9	13430.5	10331.9	7.3	0.972	231.7	5981.8
D	TRICHLOR	12797.1	4994.7	7470.4	1.0	0.945	1675.1	4325.1
E	TRICHLOR	7161.9	713.3	1263.4	0.3	0.995	283.3	731.5
F	DIBROMOB	22934.2	-34623.4	23808.0	1.8	0.998	533.9	13783.9
G	TRICHLOR	16533.9	238.4	1477.2	0.1	0.999	331.2	855.2
H	HEXCHLBUD	48463.8	-8066.8	7037.2	0.2	0.996	1577.9	4074.2
I	TETRACHL0	21389.0	-411.3	2912.9	0.2	0.997	653.2	1686.4
J	TRIBROMO	34852.1	-37057.3	29916.6	1.5	0.992	1677.0	17320.5
K	PENTACHL	28971.3	108.6	2610.6	0.1	0.999	585.4	1511.4
L	a-BHC	18558.5	821.8	1375.7	0.1	0.999	308.5	796.5
M	HCB	43299.1	-1107.2	4680.3	0.1	0.998	1049.4	2709.7
N	TETRABROM	24323.2	-24367.7	21174.7	1.5	0.992	1187.0	12259.3
O	g-BHC	22982.0	5711.9	4834.5	0.4	0.992	1084.0	2799.0
P	d-BHC	13437.9	3189.3	3248.6	0.4	0.997	364.2	1880.8
Q	HEPTACHL	27937.3	897.9	2633.7	0.1	0.998	590.5	1524.8
R	TETCHBIPH	19758.3	3251.7	6826.3	0.6	0.999	382.7	3952.2
S	ALDRIN	37597.0	2587.4	3154.5	0.1	0.999	707.3	1826.3
T	HEPTCEPO	28435.2	1521.8	2926.9	0.2	0.998	656.3	1694.5
U	g-CHLORD	25657.6	1276.9	2382.7	0.1	0.998	534.3	1379.5
V	a-ENDOSU	23147.5	1238.0	2359.2	0.2	0.998	529.0	1365.9
W	a-CHLORD	26789.6	1273.8	2592.0	0.1	0.998	581.2	1500.7
X	DIELDRIN	21561.3	2598.2	4522.0	0.4	0.998	507.0	2618.1
Y	p,p'-DDE	14021.7	3608.3	5321.8	0.6	0.994	596.6	3081.1
Z	ENDRIN	17126.1	1537.9	3905.8	0.4	0.998	437.9	2261.3
AA	b-ENDOSU	16756.7	2333.0	4541.0	0.4	0.997	509.1	2629.1
BB	p,p'-DDD	12675.6	6755.1	12745.9	0.8	0.983	903.3	7286.7
CC	o,p'-DDT	22288.0	2515.6	19661.4	0.4	0.987	1393.0	11240.2
DD	p,p'-DDT	18108.4	2822.2	20575.4	1.2	0.978	1457.7	11762.7
EE	END KETON	15155.0	2499.0	4838.0	0.5	0.996	542.4	2801.0
FF	METHOXY	4550.1	-1958.1	11003.7	0.4	0.986	246.9	6370.7
GG	MIREX	27253.5	1137.8	7104.8	0.5	0.997	796.5	4113.4

TABLE 12

RESPONSE (AREA) AND COEFFICIENT OF VARIANCE OF STANDARDS  
CHROMATOGRAPHED ON A SPB5 CAPILLARY COLUMN

CONC of 100%		100%	70%	60%	50%				
CODE	SOLN. ( $\mu$ g/ $\mu$ L)		CV	CV	CV	CV			
A	50	119891	1.9	82821	4.6	70477	1.5	62837	0.8
B	50	92607	8.4	60292	12	46996	2.1	43591	0.9
C	50	125553	1.1	81267	6.1	65171	0.5	60341	0.7
D	5	69438	1.2	47643	4.0	40828	0.9	35974	5.9
E	5	47646	5.2	34510	3.7	30118	0.6	23876	5.5
F	50	1192810	1.1	758483	3.0	630521	0.8	523993	1.3
G	5	79834	1.6	54209	2.2	46097	0.7	39573	1.6
H	5	288750	1.7	174941	1.5	144858	0.7	119642	1.6
I	5	117971	1.7	78956	3.1	67424	0.8	57485	1.1
J	20	778291	2.1	464326	3.4	388618	0.9	307553	1.8
K	5	156065	2.4	104352	2.7	89340	0.7	75535	1.7
L	5	96900	3.2	70894	1.2	59790	0.5	50089	1.6
M	5	240538	4.1	167148	2.3	142426	1.1	118267	2.1
N	20	569869	4.6	354995	2.5	301043	1.6	226591	2.5
O	5	89499	5.1	70740	1.2	59643	0.6	49671	1.5
P	10	143197	5.1	107471	1.6	91780	1.2	73373	2.4
Q	5	112626	3.0	93345	1.4	77606	1.6	69087	2.0
R&S	(25)	677327	4.0	446430	6.8	381363	3.3	312265	3.4
T	5	123392	5.9	105138	2.1	87021	1.2	76692	2.0
U	5	138515	3.0	97872	2.3	82073	1.5	71333	2.4
V	5	117796	3.9	89689	2.0	75074	1.7	65131	2.5
W	5	144861	2.8	101469	2.1	85040	1.6	73725	2.4
X	10	178087	7.5	161512	2.1	132936	2.0	118159	2.7
Y	10	164629	3.5	115435	3.0	99482	2.7	83683	2.7
Z	10	162888	5.1	127206	4.2	103676	4.7	92069	5.5
AA	10	178055	4.9	137085	2.9	113498	2.7	98867	3.6
BB	15	150640	9.3	155529	4.8	124151	5.7	103866	6.1
CC	15	307683	5.4	287452	3.6	225469	4.1	204839	4.5
DD	15	187092	9.7	207365	4.8	144531	5.2	168820	7.3
EE	10	129930	9.5	120750	4.1	100125	5.6	86611	6.2
FF	50	128757	7.9	139716	6.8	102741	7.3	97280	8.8
GG	10	268456	4.5	206745	3.4	179707	4.7	148082	4.0

TABLE 12 (CONT.)

	CONC of 100% SOLN. (pG/uL)	40%	30%	CV	20%	CV	10%	CV	
A	50	53508	5.8	38640	3.5	26565	1.8	18303	8.3
B	50	40594	15	27351	3.6	17445	0.8	15831	5.9
C	50	54214	9.2	36118	9.6	22279	1.1	18435	3.9
D	5	30123	4.6	20965	2.8	15022	1.6	8824	10
E	5	21083	5.1	13856	16	9785	1.9	5887	20
F	50	417422	2.9	280056	1.2	166220	1.1	86690	2.7
G	5	31581	12	23659	3.2	14893	1.6	8884	7.4
H	5	96348	2.4	66465	3.0	44286	2.8	24421	7.8
I	5	47836	4.0	33215	2.0	21472	1.2	12332	7.5
J	20	239740	2.2	162324	1.5	97891	1.6	52299	2.5
K	5	62236	3.4	43931	1.8	28285	3.0	15790	1.6
L	5	42046	1.6	30598	1.1	19717	1.2	11215	3.9
M	5	94862	3.2	67386	2.6	41604	2.6	23094	2.2
N	20	179682	3.0	128604	2.5	73094	2.9	42492	2.3
O	5	41447	1.7	30221	1.4	19038	1.5	10584	2.3
P	10	61630	2.3	45587	1.6	28209	3.3	15786	2.2
Q	5	53494	3.2	38733	2.0	24914	4.2	13373	7.0
R&S	(25)	266048	7.8	192498	9.3	117717	8.6	75881	18
T	5	63071	2.5	44576	2.2	29586	2.5	15719	3.8
U	5	59796	5.0	41890	3.0	27460	3.3	14748	3.5
V	5	53597	3.1	38426	2.6	25418	3.9	13421	3.3
W	5	60835	2.8	43510	2.9	28543	3.3	15287	4.3
X	10	97683	2.6	68311	2.5	45599	3.2	24574	3.3
Y	10	68782	3.3	50137	3.8	31316	4.7	18040	5.0
Z	10	76764	3.9	54320	4.0	33857	7.0	21232	16
AA	10	81089	3.1	57594	3.4	37498	4.0	20139	4.2
BB	15	85437	5.8	62006	4.3	34410	6.9	20972	6.0
CC	15	160663	3.5	109076	3.9	76878	3.7	36542	4.3
DD	15	101448	7.3	69929	5.7	44369	7.8	20072	24
EE	10	70434	7.1	50776	4.8	37307	47	19147	21
FF	50	73107	10	48001	5.3	30979	6.2	14085	18
GG	10	121781	4.5	94460	4.3	63136	3.9	30247	5.7

TABLE 13

LEAST SQUARES TREATMENT OF THE STANDARDS AND THEIR DILUTION  
FOR THE SPB-5 COLUMN ON DETECTOR B

CODE	NAME	SLOPE	INTERC	STD DEV	Ci <sub>12</sub> (X10 <sup>-9</sup> g)	COR COEF	Sm	Si
A	DICHLOR	2342.1	2804.1	2700.3	1.7	0.998	55.2	1353.0
B	DICHLOR	1743.3	1347.6	3952.2	3.4	0.992	80.8	1980.2
C	DICHLOR	2394.3	845.3	4507.4	2.8	0.994	92.2	2258.4
D	TRICHLOR	13670.9	907.0	1116.8	0.1	0.999	228.4	559.6
E	TRICHLOR	9618.9	400.2	855.3	0.1	0.999	175.0	428.6
F	DIBROMO	23339.0	-37820.5	36935.8	2.4	0.996	755.5	18506.5
G	TRICHLOR	15754.1	-59.8	799.7	0.07	1.000	163.6	400.7
H	HEXCHBUD	55027.7	-8581.5	11523.4	0.3	0.993	2357.1	5773.8
I	TETRACHL	23247.1	-500.3	1592.0	0.1	0.999	325.6	797.7
J	TRIBROMO	37294.6	-3433.4	36550.6	1.5	0.990	1869.1	18319.5
K	PENTACHL	30771.9	-913.2	1947.1	0.09	0.999	398.2	975.6
L	a-BHC	19588.4	907.0	1242.8	0.09	0.999	254.2	622.7
M	HCB	48262.0	166.9	2273.6	0.07	1.000	465.1	1139.2
N	TETRABRO	27645.8	-22471.3	23382.2	1.2	0.993	1195.7	11715.5
O	g-BHC	18667.2	1616.7	2820.1	0.2	0.996	576.8	1413.0
P	d-BHC	14671.3	952.6	2593.7	0.2	0.999	265.3	1299.5
Q	HEPTACHL	24117.7	2684.2	5270.3	0.3	0.992	1078.0	2640.7
R&S	ALDRIN+	26429.2	-4124.3	12545.0	0.7	0.998	513.2	6285.6
	TETRCHBI							
T	HEPTEPXO	26456.7	4251.6	6786.6	0.4	0.989	1388.2	3400.4
U	g-CHLORD	27737.8	666.8	1622.4	0.08	0.999	331.9	812.9
V	a-ENDOS	24211.4	1853.4	2790.9	0.1	0.998	570.9	1398.4
W	a-CHLORD	28903.2	410.8	1298.5	0.06	1.000	130.8	640.7
X	DIELDRIN	19552.6	8386.2	13266.0	1.0	0.981	1356.8	6646.9
Y	p,p'-DDE	16486.8	500.6	1278.7	0.1	1.000	130.8	640.7
Z	ENDRIN	16810.0	3323.1	5066.4	0.4	0.996	518.2	2538.5
AA	b-ENDOS	18380.8	2535.4	4546.9	0.3	0.997	465.0	2278.2
BB	p,p'-TDE	11689.4	7072.7	16360.5	2.1	0.965	1115.5	8197.4
CC	o,p'-DDT	22827.7	10742.3	23830.1	1.6	0.980	1624.8	939.9
DD	p,p'-DDT	15149.0	8013.1	28590.6	2.8	0.939	1949.4	4325.1
EE	END KETO	14318.8	7096.6	10328.6	1.1	0.979	1056.6	5175.1
FF	METHOXY	3073.6	5068.6	16072.8	7.8	0.957	328.8	8053.2
GG	MIREX	27714.6	5945.9	7645.9	0.4	0.997	782.3	3832.4

TABLE 14

SPB1 COLUMN RESPONSE (AREAS)  
 AND COEFFICIENT OF VARIANCE  
 IN DUAL COLUMN MODE  
 (DETECTOR A)

CODE	100%	CV	70%	CV	60%	CV	50%	CV
A	75037	1.8	54357	2.7	55783	6.7	38556	2.3
B	104704	1.4	82778	3.6	97181	11	65211	3.5
C	103959	1.8	81355	6.0	78910	8.	58390	6.7
D	56283	1.8	39439	1.5	40164	10	26369	2.7
E	27602	10	19684	5.8	15424	9.4	11335	12
F	932521	2.7	597868	1.8	467288	2.5	352275	2.5
G	68473	1.9	47403	1.3	38276	1.8	29844	1.8
H	169779	2.4	104419	1.8	81883	2.2	64163	2.0
I	86835	1.8	59711	5.1	46526	10	37448	9.7
J	476142	3.0	280542	2.6	198525	2.4	149466	2.1
K	99206	1.7	68755	2.6	52353	2.4	41608	1.7
L	40448	2.1	27867	3.2	20820	1.5	17495	1.7
M	110614	1.7	75134	2.3	59231	1.7	47139	2.1
N	263130	2.5	160083	3.6	115339	2.0	90419	2.5
O	71754	1.3	51873	1.8	43232	2.0	35862	2.3
P	74536	1.5	53826	2.3	43157	3.1	35718	1.7
Q	24415	2.1	16460	3.9	12656	2.3	10489	2.4
R	246826	3.2	166499	7.1	120670	1.4	99390	5.8
S	98484	2.7	69468	5.6	51857	1.6	43846	3.4
T	61389	2.0	41225	2.9	30270	3.5	24706	3.9
U	51734	2.0	34730	3.2	25344	2.6	21146	1.5
V	47865	2.1	32307	3.7	23677	2.0	19939	1.9
W	63436	2.0	42679	3.7	30752	2.6	25477	1.9
X	89919	2.0	60261	3.4	42924	1.6	36134	2.1
Y	86376	2.2	60292	4.0	43306	2.7	35986	4.9
Z	57955	3.1	39171	6.5	24903	3.9	22242	5.2
AA	59077	2.8	39842	4.1	27546	3.3	24274	2.6
BB	62820	2.8	42107	6.3	29091	3.2	25156	4.8
CC	78770	2.6	52819	5.2	37486	3.0	30766	4.2
DD	45685	5.8	34589	9.0	22616	9.0	23298	7.5
EE	44171	3.5	29888	4.9	20045	4.7	17442	4.1
FF	21583	5.5					4903	48
GG	80297	2.9	53187	5.7	36762	3.0	30164	7.8

TABLE 14 (CONT)

**SPB1 COLUMN RESPONSES  
(AREAS)  
(DUAL COLUMN MODE)**

**SPB1 AREAS**

CODE	40%	30%	20%	10%
A	37048	23	24071	2.4
B	62065	13	47749	4.6
C	58767	12	40207	7.1
D	23346	3.6	16709	2.0
E	11145	9.1	7636	9.1
F	296553	2.9	197782	2.6
G	26664	2.2	19114	2.7
H	54277	2.3	38269	1.7
I	34168	2.9	25834	7.3
J	139617	2.1	97105	2.0
K	38679	3.0	28001	1.7
L	16451	2.1	11648	2.9
M	43386	2.6	30393	1.8
N	84076	2.5	58142	1.7
O	33587	2.6	25013	1.8
P	34846	3.9	25609	3.0
Q	9592	1.6	6799	2.8
R	93228	7.3	71591	10
S	42910	6.9	34691	11
T	23611	4.1	16285	2.4
U	20123	3.2	14308	2.7
V	19010	2.1	13460	1.0
W	24948	2.3	17492	2.3
X	35339	2.2	24864	1.8
Y	35526	2.2	25048	2.8
Z	22464	2.8	15683	2.0
AA	23532	2.8	16099	1.3
BB	24974	4.6	17093	3.6
CC	30981	3.9	21173	2.2
EE	18576	5.8	13603	7.6
FF	17907	3.8	12258	4.0
GG	29500	12	20981	9.8
			10488	13
				6220 3.0

TABLE 15  
RESPONSE (AREA) FOR SPB5 COLUMN  
IN DUAL COLUMN MODE (DETECTOR B)

CODE (pg/uL)	100%		70%		60%		50%	
A 50	128971	7.8	103578	14	93485	3.0	79047	2.5
B 50	79680	11	70680	2.0	50260	2.1	41422	1.9
C 50	98709	1.6	72610	2.4	53122	2.9	44132	3.1
D 5	57721	5.7	42137	3.1	32017	3.3	25140	3.1
E 5	40287	4.9	28437	2.9	21256	3.2	15689	17
F 50	950284	1.8	633858	2.4	429352	3.9	331727	3.0
G 5	65930	1.5	46203	2.0	33442	3.3	26758	2.5
H 5	231163	2.1	148995	2.1	103218	3.5	76771	13
I 5	101145	1.8	70580	2.4	51331	3.2	40951	3.0
J 20	639295	2.0	394946	2.7	261029	4.0	202107	2.7
K 5	138278	1.8	95283	2.5	69449	3.0	56339	2.0
L 5	91449	1.6	62612	2.2	44325	2.9	36976	1.6
M 5	202584	4.3	139435	3.0	105078	2.7	85497	1.8
N 20	429201	2.8	265421	3.7	186911	3.2	150807	2.0
O 5	90290	1.8	61364	2.2	43458	2.8	36105	1.9
P 10	130138	2.3	88331	2.8	62207	3.1	52999	1.5
Q 5	115867	2.1	81543	2.6	59993	3.1	48766	2.0
R&S (25)	561115	2.9	378113	4.1	269900	3.3	229498	3.2
T 5	138545	1.6	95974	1.9	68954	2.2	57874	2.4
U 5	124643	1.6	85471	2.2	62146	2.2	52049	1.8
V 5	115066	1.5	79328	2.2	57445	1.9	48217	2.0
W 5	130628	1.5	88638	3.1	64787	1.7	54163	1.9
X 10	214088	1.7	147305	2.3	104204	1.9	87344	1.7
Y 10	135483	2.1	96032	7.5	67389	2.2	57358	2.4
Z 10	165504	2.8	113291	5.3	66936	3.8	60161	4.3
AA 10	163442	2.1	112423	3.4	77891	2.1	67518	2.5
BB 15	132909	6.2	92132	8.4	66361	3.2	59741	4.2
CC 15	345855	2.9	236193	3.6	166783	2.0	139886	4.1
DD 15	190211	5.3	147108	7.4	94570	3.2	103189	3.4
EE 10	142085	3.5	95404	5.3	66166	4.8	57510	6.7
FF 50	122869	8.9	93353	13	63404	6.4	59029	8.7
GG 10	246028	1.6	171095	2.8	113981	4.4	95377	4.4

TABLE 15 (CONT)

RESPONSE (AREAS) FOR SPD5 COLUMN  
(DUAL COLUMN, DETECTOR B)

CODE	CONC 10% (pg/uL)	40%		30%		20%		
A	50	89870	4.6	65192	25	58156	3.1	50971 3.7
B	50	43288	4.6	33797	1.1	23761	1.6	(18266 2.7)
C	50	45621	3.5	32647	2.0	18861	2.4	11916 13
D	5	25358	8.0	17963	2.8	11145	5.4	5383 6.2
E	5	17312	6.2	9479	27	6338	16	2991 41
F	50	346749	2.7	223126	3.8	112376	6.1	57537 3.4
G	5	27879	2.0	19305	2.4	10689	13	5251 3.7
H	5	83459	2.3	56792	2.0	32078	5.0	16347 3.1
I	5	42344	2.5	29193	1.6	15498	4.1	7581 4.6
J	20	209266	3.0	136808	2.3	72763	3.5	37246 2.3
K	5	57164	2.4	39785	3.0	21710	3.4	10576 4.6
L	5	38499	2.8	27081	2.8	15304	3.1	7935 2.9
M	5	85058	3.0	57179	2.3	32186	3.9	16065 1.9
N	20	147608	3.2	100316	2.4	56104	3.6	28509 2.3
O	5	37193	2.4	25947	1.6	14769	3.4	7550 2.5
P	10	54017	2.7	37208	1.9	21367	4.4	10530 4.5
Q	5	51206	3.1	35654	2.1	18936	8.4	9108 6.7
(R&S)	(25)	226415	9.4	169077	8.3	83821	12	52033 14
T	5	59931	2.5	41862	2.3	24218	3.4	11814 4.1
U	5	52684	3.0	36854	2.6	21311	4.2	10614 2.4
V	5	48873	2.9	34196	2.0	19598	3.5	9658 2.7
W	5	55187	2.7	38452	2.1	21926	4.0	10783 2.6
X	10	90655	2.7	62883	2.3	35963	3.9	17618 4.3
Y	10	58216	4.2	40313	3.3	22420	5.1	11353 3.0
Z	10	67762	5.6	46666	2.9	23189	8.2	11461 3.7
AA	10	69526	3.9	47314	3.1	29110	4.5	13137 2.8
BB	15	55708	7.5	37904	4.4	22208	8.1	11146 4.6
CC	15	147383	4.5	100471	3.7	55093	5.3	27320 3.0
DD	15	84944	8.1	59515	5.3	29569	12	13522 2.1
EE	10	60704	7.2	41017	7.4	22511	11	9552 19
FF	50	57042	25	40833	6.0	20834	14	9123 11
GG	10	104122	4.1	77501	6.4	39941	5.5	18791 11

TABLE 16

LEAST SQUARE TREATMENT OF DATA GENERATED FROM SEQUENCES USING  
 DILUTIONS OF STANDARDS ON SPB1 COLUMN ON DETECTOR A  
 IN DUAL MODE (SPB5 ON DETECTOR B)

CODE	NAME	SLOPE	INTERC	STD DEV	$C_{12}$ ( $\times 10^{-2}$ g)	CORR COEF	Sm	Si
A	1,3DICH	1555.7	1480.7	3752.5	3.6	0.990	76.8	1880.2
B	1,4DICH	2234.7	8579.8	11091.7	7.4	0.961	226.9	5557.4
C	1,2DICHL	2132.6	6718.9	6781.9	4.7	0.983	138.7	3398.0
D	TRICHLOR	14201.2	-3457.5	10750.7	1.3	0.912	2410.6	6224.2
E	TRICHLOR	5546.6	-613.8	922.1	0.3	0.995	206.8	533.9
F	DIBROMO	17899.0	-35313.9	45671.6	3.7	0.991	904.0	22096.9
G	TRICHLOR	13727.8	-1572.0	1560.1	0.2	0.998	349.8	903.2
H	HEXCLBUDI	32975.9	-8482.4	8168.9	0.4	0.989	1831.7	4729.5
I	TETRACHLO	16676.4	315.2	2894.0	0.3	0.995	648.9	1675.3
J	TRIBROMO	22848.7	-34616.6	34190.7	2.6	0.976	1916.7	19795.1
K	PENTACHLO	19785.9	-2655.2	3217.2	0.2	0.995	721.4	1862.6
L	a-BHC	7935.5	-564.4	1372.6	0.3	0.995	307.8	794.7
M	HCB	22002.1	-2848.6	3151.4	0.2	0.996	706.6	1824.5
N	TETRABROM	12721.1	-16227.2	16105.5	2.1	0.983	902.8	9324.4
O	g-BHC	14248.7	277.9	5294.5	0.7	0.973	1282.0	3412.0
P	d-BHC	7360.7	1252.8	2292.6	0.5	0.996	257.0	1327.3
Q	HEPTACHL	4845.1	-669.6	774.0	0.2	0.996	173.5	448.1
S	ALDRIN	5546.6	-613.8	922.1	0.3	0.995	206.8	533.9
R	TETCLBIPH	18307.4	-48392.5	45452.1	4.1	0.990	979.8	25246.5
T	HEPTCLEPOX	12063.8	-1910.7	2532.6	0.3	0.992	567.9	1466.3
U	g-CHLORD	10145.0	-1454.1	2135.2	0.4	0.992	478.8	1236.2
V	a-ENDO	9384.9	-1151.4	1902.9	0.3	0.993	426.7	1101.7
W	a-CHLORD	12419.2	-1784.4	2808.6	0.4	0.991	629.8	1626.1
X	DIELDRIN	8771.0	-2470.8	4132.4	0.8	0.990	463.3	2392.5
Y	p,p'-DDE	8557.2	-2025.0	3632.4	0.7	0.992	407.2	2103.0
Z	ENDRIN	5659.7	-2290.7	3565.7	1.1	0.983	399.8	2064.4
AA	b-ENDOS	5787.8	-1791.5	2854.8	0.8	0.989	320.1	1652.8
BB	p,p'-DDD	3879.7	-1595.3	3798.7	1.6	0.984	269.1	2171.7
CC	o,p'-DDT	4698.2	-2368.2	4665.2	1.6	0.984	330.5	2667.0
DD	p,p'-DDT	2990.4	-1045.8	2080.7	1.2	0.991	147.4	1189.5
EE	END KETO	4323.0	-1440.0	2441.1	0.9	0.986	273.7	1413.3
FF	METHOXY							
GG	MIREX	7855.5	-3434.4	4327.1	1.0	0.987	485.1	2505.2

TABLE 17

LEAST SQUARES TREATMENT OF DILUTED STANDARDS FOR  
 SPB5 TYPE COLUMN USING DETECTOR B (WITH SPB1 TYPE  
 ON DETECTOR A) - USING 8 POINTS

CODE	NAME	SLOPE	INTER	STD DEV	C <sub>1</sub> (X10 <sup>-12</sup> g)	COR	S <sub>m</sub>	S <sub>i</sub>
A	DICHLORO	2055.3	30034.8	15505.3	12	0.930	332.7	8783.0
B	DICHLORO	1550.9	6778.9	6008.1	6.8	0.978	134.8	3565.6
C	DICHLORO	1940.5	264.3	3811.4	3.5	0.994	85.5	2261.8
D	TRICHLOR	11512.8	-526.4	1896.4	0.3	0.996	25.4	1125.4
E	TRICHLOR	8125.5	-1708.0	1808.0	0.4	0.993	405.6	1073.0
F	DIBROMOB	18839.4	-58054.4	60908.4	5.7	0.985	1366.2	36147.0
G	TRICHLOR	8125.5	-1824.8	2873.6	0.3	0.993	644.6	1705.4
H	HEXCLBUDI	44801.0	-12031.6	15677.9	0.6	0.982	3516.7	9304.3
I	TETRACHLO	20131.4	-3244.3	4373.7	0.3	0.993	981.1	2595.6
J	TRIBROMO	30909.8	-44708.7	49939.7	2.9	0.976	2800.4	29637.8
K	PENTACHL	27370.2	-4234.2	5096.2	0.4	0.993	1322.6	3499.2
L	a-BHC	17842.5	-2205.2	4407.5	0.4	0.990	988.6	2615.7
M	HCB	40256.9	-5792.8	7105.6	0.3	0.995	1593.9	4217.0
N	TETRABROM	20715.3	-23921.6	27943.2	2.4	0.983	1566.9	16583.8
O	g-BHC	27631.4	-2531.3	4398.3	0.4	0.990	986.6	2610.3
P	d-BHC	13022.4	-5711.7	6505.2	1.1	0.900	847.9	4796.6
Q	HEPTACHL	23025.6	-2696.0	4600.4	0.4	0.994	1031.9	2730.2
R+S	TETBIPHE	109099.5	-11435.5	26695.3	0.4	0.989	5985.9	15455.5
	+ ALDRIN							
T	HEPTEPOXI	27155.3	-2799.9	5995.3	0.4	0.993	1344.8	3558.0
U	g-CHLORDA	24428.2	-2774.0	5203.9	0.4	0.993	1167.3	3088.3
V	a-ENDOSUL	22597.5	-2581.4	4803.2	0.4	0.993	1077.4	2850.6
W	a-CHLORDA	25570.4	-3164.9	5529.3	0.4	0.993	1239.1	3278.5
X	DIELDRIN	20970.7	-5450.4	10023.7	0.9	0.991	1124.2	5948.7
Y	p,p'-DDE	13413.0	-3211.6	5959.6	0.8	0.993	668.4	3536.8
Z	ENDRIN	16071.7	-7403.8	12693.2	1.4	0.977	1423.6	4692.7
AA	b-ENDOSUL	15956.8	-3962.1	7907.2	0.9	0.990	886.8	4692.7
BB	p,p'-DDD	8611.2	-5549.1	10019.4	1.9	0.977	709.9	5728.0
CC	o,p'-DDT	21292.5	-4143.7	20563.7	1.6	0.984	1456.9	11756.0
DD	p,p'-DDT	12380.6	-897.90	9805.1	1.3	0.989	694.67	5605.5
EE	END KETO	13526.5	-3228.6	7984.5	0.9	0.990	895.1	4622.7
FF	METHOXYCL	2492.8	-1790.9	5698.5	4.0	0.992	127.8	3381.8
GG	MIREX	24004.5	-6679.9	14836.7	1.1	0.986	1664.0	8805.1

TABLE 18

RESPONSE (AREA) FROM DETECTOR FOR  
 SPB605 CAPILLARY COLUMN ON DETECTOR B  
 AND COEFFICIENT OF VARIANCE (DUAL MODE)

RT	CODE	100%		70%		60%		50%	
			CV		CV		CV		CV
3.15	A	113803	6.1	65482	2.4	75441	3.3	50215	2.7
3.31	B	69313	12	43978	4.0	43366	5.4	34527	23
3.71	C	80617	5.1	60509	2.8	46718	2.3	44058	2.9
4.82	D	44622	2.0	32969	3.3	27458	3.4	24549	11
5.80	E	19784	3.7	14825	3.7	12334	2.7	10888	3.6
5.87	F	157379	2.7	103818	3.2	88860	2.9	72022	2.7
6.21	G	650189	2.5	433661	3.6	350387	3.1	393492	2.9
6.53	H	51246	2.3	36827	3.0	29558	2.4	26631	2.6
8.98	I	69959	8.2	53650	3.3	44385	3.8	38188	2.7
9.68	J	440081	2.8	273371	3.6	230217	3.2	182704	3.2
10.77	K	102550	2.2	70132	2.9	60528	2.7	50566	3.0
13.71	L	138472	2.6	92479	2.9	81852	3.2	66497	3.2
14.38	M	66029	2.3	44945	2.5	40147	2.9	31964	2.4
15.14	N	327992	2.8	194826	3.0	179614	2.9	141187	3.6
16.06	O	66487	2.5	45664	2.7	40351	3.2	32502	3.0
17.88	P	72990	4.3	54665	2.7	45736	4.0	40237	3.9
18.40	Q	101376	3.0	67105	2.9	59735	3.3	49305	3.3
19.59	R	114243	2.3	80263	2.3	69881	3.0	56584	3.0
20.17	S	303907	3.2	194943	2.7	182851	4.4	151755	5.9
22.77	T	100806	2.9	70465	3.4	60336	4.1	50126	3.4
23.59	U	92430	2.8	62678	2.6	55585	3.5	42368	7.6
24.18	V+W	181139	3.0	123544	2.6	107613	3.0	88554	4.8
25.19	X+Y	253606	3.2	166245	2.6	148429	3.7	123495	6.2
26.19	Z	94614	5.6	61642	4.5	56737	6.2	52071	6.5
26.71	AA	166809	6.9	113366	4.8	98095	6.6	88990	7.8
26.79	BB	296684	5.0	178968	3.8	166334	5.3	144356	6.1
27.56	CC	117639	7.7	86735	4.9	71659	7.2	81751	9.8
29.49	DD	71537	18	26508	14	39733	16	33491	38
29.57	EE	84462	5.2	54172	3.9	48705	5.7	46590	21
29.64	FF	103575	5.1						

TABLE 18 (CONTINUED)  
RESPONSE (AREA) FOR THE SPB608 CAP. COLUMN  
IN DUAL MODE

RT	CODE	40%	30%	20%	10%
3.15	A	45648	2.5	38818	3.6
3.31	B	31310	11	27813	13
3.71	C	37500	5.4	31556	5.9
4.82	D	19200	3.0	16487	7.9
5.80	E	8530	4.6	7039	10
5.87	F	58290	2.6	49785	5.5
6.21	G	231647	4.2	192561	5.7
6.53	H	21250	2.7	17427	4.8
8.98	I	31144	3.8	25933	6.2
9.68	J	144381	3.2	119772	5.6
10.77	K	41020	3.0	34817	5.2
13.71	L	52392	6.7	45462	5.0
14.38	M	26583	2.8	22495	4.7
15.14	N	110760	3.3	96219	5.8
16.06	O	26908	2.6	22929	4.9
17.88	P	31908	4.1	28269	7.1
18.40	Q	40053	3.5	34317	4.8
19.59	R	47080	2.6	39684	4.8
20.17	S	114698	3.6	102117	7.0
22.77	T	41238	3.6	35040	4.9
23.59	U	35112	5.0	31881	5.6
24.18	V+W	72149	2.9	62264	6.2
25.19	X+Y	98363	2.8	89075	5.8
26.19	Z	37126	4.8	37014	7.4
26.71	AA	61626	3.8	59918	7.8
26.79	BB	103471	12	99837	6.4
27.56	CC	42483	7.0	45657	7.5
29.49	DD	297498	11	8278	98
29.57	EE	64753	3.1	46883	31
29.64	FF	134131	3.5	99759	31

TABLE 19

RESPONSE (AREAS) FOR THIN PHASE SE-52 COLUMN  
 ON DETECTOR A IN DUAL MODE  
 WITH COEFFICIENT OF VARIANCE  
 (WITH SPB608 ON DETECTOR B)

CODE	100%		70%		60%		50%	
		CV		CV		CV		CV
A&B		144323	12					
C		96119	4.0				61164	31
D		54009	1.4				37165	3.7
E	36530	4.8	26026	2.9	17035	2.8	18567	3.2
F	1182620	2.8	782314	2.4	612508	2.4	493149	2.5
G	62712	2.7	42527	1.8	35732	2.2	29478	2.6
H	287250	12	178535	2.2	139701	2.0	109195	2.0
I	106688	2.0	77411	1.5	62322	2.0	51888	3.3
J	638753	2.8	390082	2.0	312560	2.4	240490	2.7
K	133765	1.5	97428	1.7	80951	2.0	67383	2.4
L	60613	9.2	43096	3.0	32325	17	24282	20
M	147832	1.3	101079	2.1	87646	3.0	67417	2.9
N	387072	2.4	234197	2.3	204670	3.4	158171	2.7
O	55768	5.9	39676	2.1	33449	2.6	26480	2.4
P	98138	2.0	64245	2.1	57067	3.3	44849	2.3
Q	37622	2.6	31668	3.1	23595	4.0	21569	4.0
R&S	462319	2.3	300952	2.2	276417	3.4	220971	3.5
T	96780	1.7	73093	2.5	59516	3.2	49390	2.8
U	97750	2.2	63483	3.6	59273	3.2	43758	3.2
V	86345	2.2	61509	4.9	53502	3.2	42023	3.1
W	108809	2.1	73607	2.8	66133	3.1	50700	3.3
X	156847	2.3	113058	3.4	94829	3.4	78638	3.4
Y	119593	2.8	75328	4.0	73892	4.4	58362	7.4
Z	101015	3.4	75491	5.5	63164	5.1	56463	6.0
AA	114265	3.0	79254	4.5	69996	4.2	57918	4.4
BB&CC	244100	3.9	175348	5.4	150535	5.6	133218	5.3
DD	72911	5.6	63503	6.7	47570	7.1	58437	7.1
EE	84976	3.8	63110	5.1	53153	5.0	48677	5.4
FF	33539	9.4	27085	9.3	22361	11	24118	12
GG	170585	4.6	115570	5.6	110248	4.9	88243	5.9

TABLE 19 (CONT)

RESPONSE (AREA) OF SE-52 THIN PHASE  
CAPILLARY COLUMN ON DETECTOR A

CODE	40%		30%		20%		10%	
		CV		CV		CV		CV
A&B	94663	16	45648	2.5	102858	20	40928	79
C	57156	6.1	31310	11	71629	22	17785	76
D	33926	1.8	37500	5.4	30912	5.9	19686	17
E	14161	10	11922	4.3	6626	6.8	3891	4.8
F	405651	1.7	297331	6.8	162409	3.1	81211	2.9
G	25023	1.7	18967	4.8	11675	3.2	5705	9.2
H	92752	1.5	67310	5.2	41480	2.8	21662	5.7
I	44593	1.9	33374	5.7	19279	3.3	9335	4.1
J	201495	1.6	148357	5.9	90169	3.0	41671	17
K	58806	2.1	45744	4.9	30567	5.4	16072	6.2
L	25621	3.4	19197	13	14693	3.0	7269	13
M	58427	2.9	45128	6.4	30172	4.6	14364	7.1
N	131426	2.8	102645	6.4	68453	5.1	33223	5.5
O	23107	1.8	17665	5.7	13170	9.3	7157	11
P	38185	2.4	29703	6.7	20963	4.6	9306	4.2
Q	17777	3.0	13445	7.4	7838	9.1	4856	19
R&S	178518	4.9	138874	7.6	90882	5.3	42258	2.2
T	42636	2.4	32625	6.2	21389	5.5	10381	2.9
U	37398	3.2	29775	6.2	21087	6.7	9042	1.9
V	35765	3.1	28307	6.2	19287	6.4	8672	2.8
W	43113	3.3	34164	6.1	23396	6.5	10252	3.5
X	66398	3.7	52594	7.1	33299	10	16415	2.6
Y	45455	7.3	37189	8.1	28236	32	11068	13
Z	47867	12	38138	20	24681	8.5	11382	9.8
AA	47280	4.8	38940	8.4	26097	7.8	11706	4.3
BB&CC	102644	6.6	88339	10	53674	9.5	25441	6.8
DD	33291	8.7	29464	12	16451	10	7504	9.8
EE	38750	6.0	32379	9.1	19969	11	9165	13
FF	16207	10	14241	16	7825	17	4628	16
GG	68143	6.6	58565	10	38664	9.3	17295	7.0

TABLE 20

LEAST SQUARES TREATMENT OF STANDARDS AND THEIR DILUTIONS  
 FROM DATA COLLECTED FROM THE SPB-608 COLUMN ON DETECTOR B  
 (WITH A SE-52 COLUMN ON DETECTOR A)

CODE	NAME	SLOPE	INTER- CEPT	STD DEV	$C_{12}$ ( $\times 10^{-3}$ g)	COR	Sm	Si
A	1,3-DICHLORO	2098.4	3968.3	6968.0	5.7	.981	156.2	4034.2
B	1,4-DICHLORO	1266.8	4473.1	3335.4	4.5	.988	74.8	1931.1
C	1,2-DICHLORO	1570.9	3656.1	2990.8	3.0	.993	67.0	1731.6
D	TRICHLORO	8828.8	1490.0	1129.7	0.2	.997	254.3	654.1
E	TRICHLOROB	3639.2	1682.8	1339.9	0.6	.977	300.4	775.8
F	HEXCLBUDIENE	30675.5	-803.4	3455.2	0.2	.998	774.8	2000.4
G	DIBROMOBENZ	13008.0	-5917.4	33369.4	4.4	.989	748.2	19319.6
H	TRICHLORO	10247.7	331.1	1048.1	0.1	.998	235.0	606.8
I	TETRACHLORO	14150.4	1989.9	1946.0	0.2	.997	436.4	1126.7
J	TRIBROMOBEN	21429.3	-14987.5	15773.2	1.3	.994	884.2	9132.0
K	PENTACHLORO	20186.5	780.3	1646.2	0.1	.999	369.1	953.1
L	HCB	2098.4	3968.3	6968.0	0.6	.981	156.2	4034.2
M	a-BHC	12973.4	707.6	1144.4	0.1	.999	256.6	662.5
N	TRIBROMOBEN	15748.4	-6873.8	12328.9	1.4	.993	691.1	7137.9
O	g-BHC	13082.8	790.5	1136.9	0.1	.999	254.9	658.2
P	HEPTACHLOR	14902.4	2254.6	2971.4	0.3	.993	666.3	1720.3
Q	d-BHC	9864.0	951.2	2003.0	0.1	.998	224.6	1159.7
R	ALDRIN	22612.5	1581.3	1831.5	0.1	.999	410.7	1060.4
S	TETRCHBIPH	14589.7	4940.1	8133.5	0.9	.997	455.9	4707.0
T	HEPT EPOXI	19964.8	1023.5	1689.6	0.1	.999	378.9	978.2
U	g-CHLORDAN	18166.0	260.7	2282.4	0.2	.997	511.8	1321.4
V+W	a-CHLORDAN+	17837.3	1302.0	3216.4	0.3	.999	360.6	1862.1
	a-ENDOSULF							
X+Y	p,p'-DDE+	12390.1	875.8	7302.2	1.0	.996	409.3	4227.7
	DIELDRIN							
Z	ENDRIN	9202.3	2018.8	3891.2	0.7	.992	436.2	2252.8
AA	o,p'-DDT	10383.4	2253.0	9809.8	1.6	.985	695.0	5608.2
BB	p,p'-TDE	17641.5	3371.5	21685.9	2.1	.974	1536.4	12397.5
CC	p,p'-DD	7583.7	3373.2	10485.8	2.3	.968	742.9	5994.6
DD	METHOXY	1351.7	2765.9	14468.8	17.	.856	308.9	7729.6
EE	ENDRIN KET	6184.8	20100.2	13555.5	3.8	.838	1519.8	7848.0
GG	MIREX	4291.5	51037.8	59654.5	16	.342	4445.9	22958.4

TABLE 21

LEAST SQUARES TREATMENT ON STANDARDS FOR THE THIN PHASE SE-52  
 (DB5) TYPE COLUMN ON DETECTOR A  
 (SPB608 COLUMN ON DETECTOR B)

CODE	NAME	SLOPE	INTER- CEPT	STD DEV	C <sub>1</sub> (X10 <sup>-12</sup> g)	CORR COEF	Sm	Si
A+B	DICHLORO	2883.35	15031	20149.2	9.4	.934	450.55	9081.1
C	DICHLORO	2710.45	3513.6	12221.3	6.8	.970	257.32	6125.66
D	TRICHLOR	2484.74	-7129.4	16957.8	10.	.934	357.06	8499.78
E	TRICHLOR	6856.52	898.8	1956.5	0.5	.986	429.52	1107.28
F	DIBROMOB	23507.36	-51830	44308.1	3.3	.993	993.52	25652.6
G	TRICHLOR	12401.65	-396.7	846.1	0.1	.999	189.73	489.89
H	HEXCHBUDI	55925.0	-13882	13311.5	0.4	.990	2984.86	7706.86
I	TETRACHLO	21553.4	-432.2	1714.5	0.1	.999	384.44	992.62
J	TRIBROMO	31168.0	-34063.8	29947.	1.7	.990	1678.78	17338.4
K	PENTACHLO	29076.8	193.7	2441.3	0.1	.999	547.41	1413.40
L	a-BHC	11653.6	505.3	2752.8	0.4	.990	617.26	1593.76
M	HCB	29225.2	-398.3	2295.4	0.1	.999	514.71	1328.97
N	TETRABRO	18624.7	-10793.2	14524.1	1.4	.993	814.19	8408.92
O	g-HCB	10823.7	1347.0	1277.3	0.2	.998	286.41	739.52
P	d-HCB	5059.11	-5573.8	6395.8	2.2	.982	358.53	3702.90
Q	HEPTCHL	7781.6	1222.7	1758.6	0.4	.991	394.32	1018.14
R+S	TETCHBIP & ALDRIN	18296.8	-3807.8	7783.1	0.7	.999	349.04	4506.12
T	HEPTCHEP	19391.4	1768.1	2183.0	0.2	.998	489.49	1263.87
U	g-CHLOR	19283.1	-895.2	2298.4	0.2	.998	515.38	1330.70
V	a-ENDOS	17310.7	503.3	1161.1	0.1	.999	260.35	672.21
W	a-CHLOR	21626.9	-406.5	1726.4	0.1	.999	387.11	999.53
X	DIELDRI	15755.1	2371.9	3316.4	0.4	.998	371.81	1920.04
Y	p,p'-DE	11795.7	-671.3	3232.5	0.5	.997	362.41	1871.48
Z	ENDRIN	10098.0	3701.9	3211.0	0.5	.996	360.00	1859.05
AA	b-ENDOS	11451.7	1892.4	3913.4	0.6	.995	438.75	2265.68
BB+	p,p-DDE	8175.7	3853.6	5946.4	1.2	.997	222.23	3442.75
CC	o,p-DDT							
DD	p,p'-DT	5515.0	12807.9	13110.5	3.6	.896	966.25	6589.01
EE	ENDO KE	8503.34	3004.8	2750.1	0.5	.995	308.32	1592.18
FF	METHOXY	665.73	2803.0	2623.0	8.4	.970	68.01	1862.51
GG	MIREX	17083.7	1204.3	4351.1	0.4	.997	487.82	2519.09

TABLE 22

## INFLUENCE ON THE REPRODUCIBILITY FROM INCORRECT CRIMPING

COMPOUND CODE	TIGHT CRIMP (WRONG)		SNUG CRIMP (CORRECT)	
	HEIGHT	CV	HEIGHT	CV
A	89897	9.0	79047	2.5
B	47206	4.6	41422	1.9
C	52366	2.7	44132	3.1
D	30035	8.3	25140	3.1
E	18991	16.	15689	17
F	428399	6.5	331727	3.0
G	33143	5.3	26758	2.5
H	101534	5.8	76771	1.3
I	50736	5.5	40951	3.0
J	260448	6.4	202107	2.7
K	68149	6.4	56339	2.0
L	45384	6.4	36105	1.6
M	87700	35	85497	1.8
N	177114	7.8	150807	2.0
O	43683	6.0	36105	1.9
P	63027	6.1	52999	1.5
Q	61458	7.8	48766	2.0
R+S	274309	8.9	229489	3.2
T	69780	6.6	57874	2.4
U	60638	6.9	52049	1.8
V	56160	6.7	48217	2.0
W	63249	6.9	54163	1.9
X	103221	7.1	87344	1.7
Y	66814	7.8	57358	2.4
Z	80782	8.4	60161	4.3
AA	78596	7.9	67518	2.5
BB	63367	11.	59741	4.2
CC	167813	8.6	139886	4.1
DD	122518	11.	103189	3.4
EE	67924	11.	57510	6.7
FF	70276	14.	59029	8.7
GG	121321	32.	95377	4.4

The values in the two far right hand columns can be found in Table 15. Replacing those values with the results in the second column would greatly influence the linear gression analysis.

TABLE 23

INFLUENCE OF TOTAL AMOUNT INJECTED  
AS REFLECTED IN THE RETENTION TIMES

	1uL		2uL		3uL
R <sub>t</sub>	HEIGHT	R <sub>t</sub>	HEIGHT	R <sub>t</sub>	HEIGHT
1.49	46796	1.56	84460	1.62	117438
1.65	38880	1.72	74351	1.78	110741
2.59	27447	2.64	53475	2.68	77018
3.05	22804	3.10	44279	3.13	65111
3.46	40297	3.49	81434	3.52	120108
3.71	102321	3.75	273045	3.77	459009
5.62	47394	5.63	99186	5.63	150280
7.12	16506	7.13	23964	7.12	22901

Each of the remaining 18 components exhibit the same retention times independent of the total volume injected.

TABLE 24

EFFECT OF POSITIONING COLUMN IN INJECTOR  
USING SPLITLESS MODE INJECTION

MEASURED IN AREA COUNTS

$R_T$	5 cm into INJECTOR	2 mm into INJECTOR	FACTOR
1.79	51482	152050	2.95
2.00	67692	174110	2.57
3.04	36224	38906	1.07
3.53	17155	37641	2.19
3.95	23784	53351	2.66
4.20	39209	95782	2.44
6.13	31756	57325	1.81
8.03	37562	75313	2.01
10.65	24445	50360	2.06
13.09	23952	71544	2.03
14.38	29348	57547	1.90
15.84	63663	139560	2.19
16.86	59729	129370	2.17
17.16	77028	172030	2.23
17.47	79324	173890	2.19
17.70	59825	128010	2.14
18.77	47826	108370	2.27
19.11	35605	76238	2.14
19.68	64917	182710	2.81
19.87	95046	250190	2.63
21.18	156650	397850	2.54
22.11	125040	364130	2.91
23.26	67952	219050	3.22
23.88	104540	230590	2.21

TABLE 25  
 MINIMAL DETECTABLE AMOUNTS ( $C_1$ )  
 FOR EACH COLUMN AND CONFIGURATION  
 $(\times 10^{-12})$

Code	Name	Single Column		Dual Columns		Pair 2 (thin phase)	
		DB-1	DB-5	Pair 1			
				DB-1	DB-5		
A	1,3-Dichlorobenzene	2.2	1.7	3.6	12	9.4	
B	1,4-Dichlorobenzene	7.3	3.4	7.4	6.8	4.5	
C	1,2-Dichlorobenzene	7.3	2.8	4.7	3.5	6.8	
D	1,3,5-Trichlorobenz	1.0	0.1	1.3	0.3	10.	
E	1,2,4-Trichlorobenz	0.3	0.1	0.3	0.4	0.5	
F	Dibromobenzene	1.8	2.4	3.7	5.7	3.3	
G	1,2,3-Trichlorobenz	0.1	0.07	0.2	0.3	0.1	
H	Hexachlorobutdiene	0.2	0.3	0.4	0.6	0.4	
I	Tetrachlorobenzene	0.2	0.1	0.3	0.3	0.1	
J	Tribromobenzene	1.5	1.5	2.6	2.9	1.7	
K	Pentachlorobenzene	0.1	0.09	0.2	0.4	0.1	
L	a-BHC	0.1	0.09	0.3	0.4	0.4	
M	Hexachlorobenzene	0.1	0.07	0.2	0.3	0.1	
N	TetraBromobenzene	1.5	1.2	2.1	2.4	1.4	
O	g-BHC	0.4	0.2	0.7	0.4	0.2	
P	d-BHC	0.4	0.2	0.5	1.1	2.2	
Q	Heptachlor	0.1	0.3	0.2	0.4	0.4	
R	TetraChlBiPhenyl	0.6	0.7	4.1	0.4	0.7	
S	Aldrin	0.1		0.3		0.1	
T	Heptclorepoxide	0.2	0.4	0.3	0.4	0.2	
U	g-Chlordane	0.1	0.08	0.4	0.4	0.2	
V	a-Endosulphan	0.2	0.1	0.3	0.4	0.1	
W	a-Chordane	0.1	0.06	0.4	0.4	0.1	
X	Dieldrin	0.4	1.0	0.8	0.9	0.4	
Y	p,p'-DDE	0.6	0.1	0.7	0.8	0.5	
Z	Endrin	0.4	0.4	1.1	1.4	0.5	
AA	b-Endosulphan	0.4	0.3	0.8	0.9	0.6	
BB	p,p'-DDD	0.8	2.1	1.6	1.9	1.2	
CC	o,p'-DDT	0.4	1.6	1.6	1.6	1.6	
DD	p,p'-DDT	1.2	2.8	1.2	1.3	3.6	
EE	Endrin ketone	0.5	1.1	0.9	0.9	0.5	
FF	Methoxychlor	0.4	7.8		4.0	8.4	
GG	Mirex	0.5	0.4	1.0	1.1	0.4	
						16.	

FIGURE 1

Chromatogram of organochlorines on SPB-1 column.

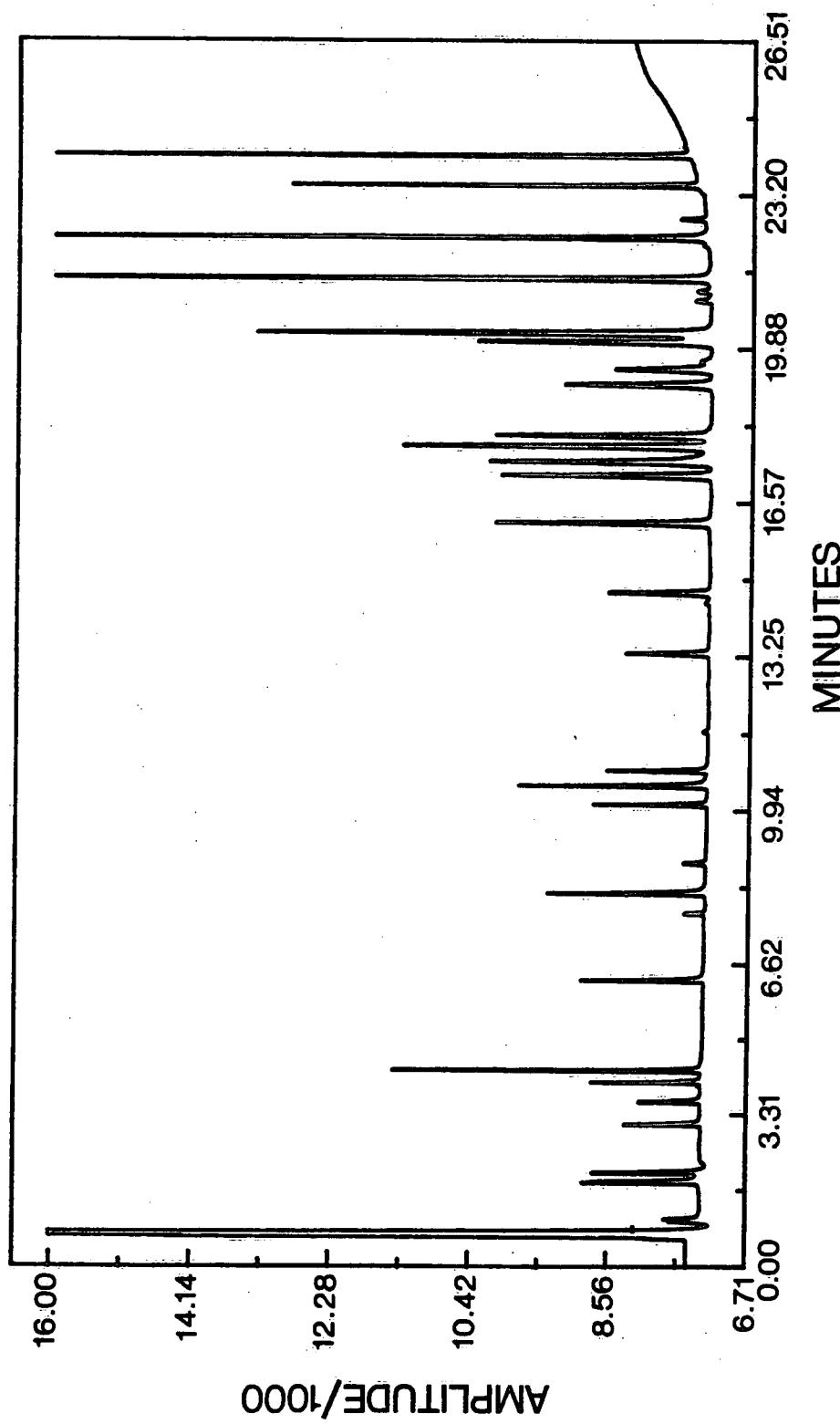


FIGURE 1.

FIGURE 2

Chromatogram of organochlorines and surrogates from SPB-1 column, in dual mode operation.

FIGURE 2.

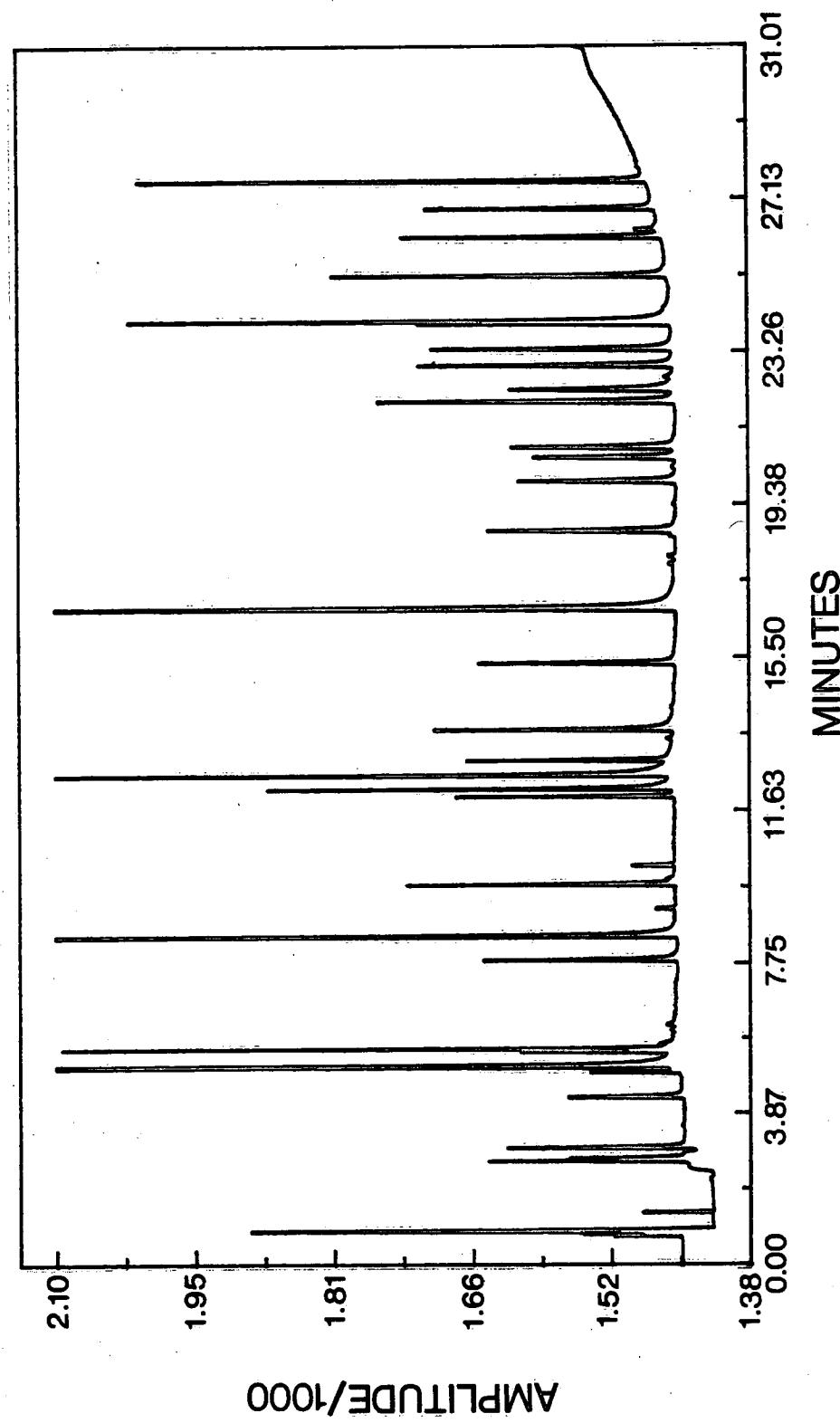


FIGURE 3

Chromatogram of organochlorines and surrogates from SPB-5 column, in dual mode operation.

FIGURE 3.

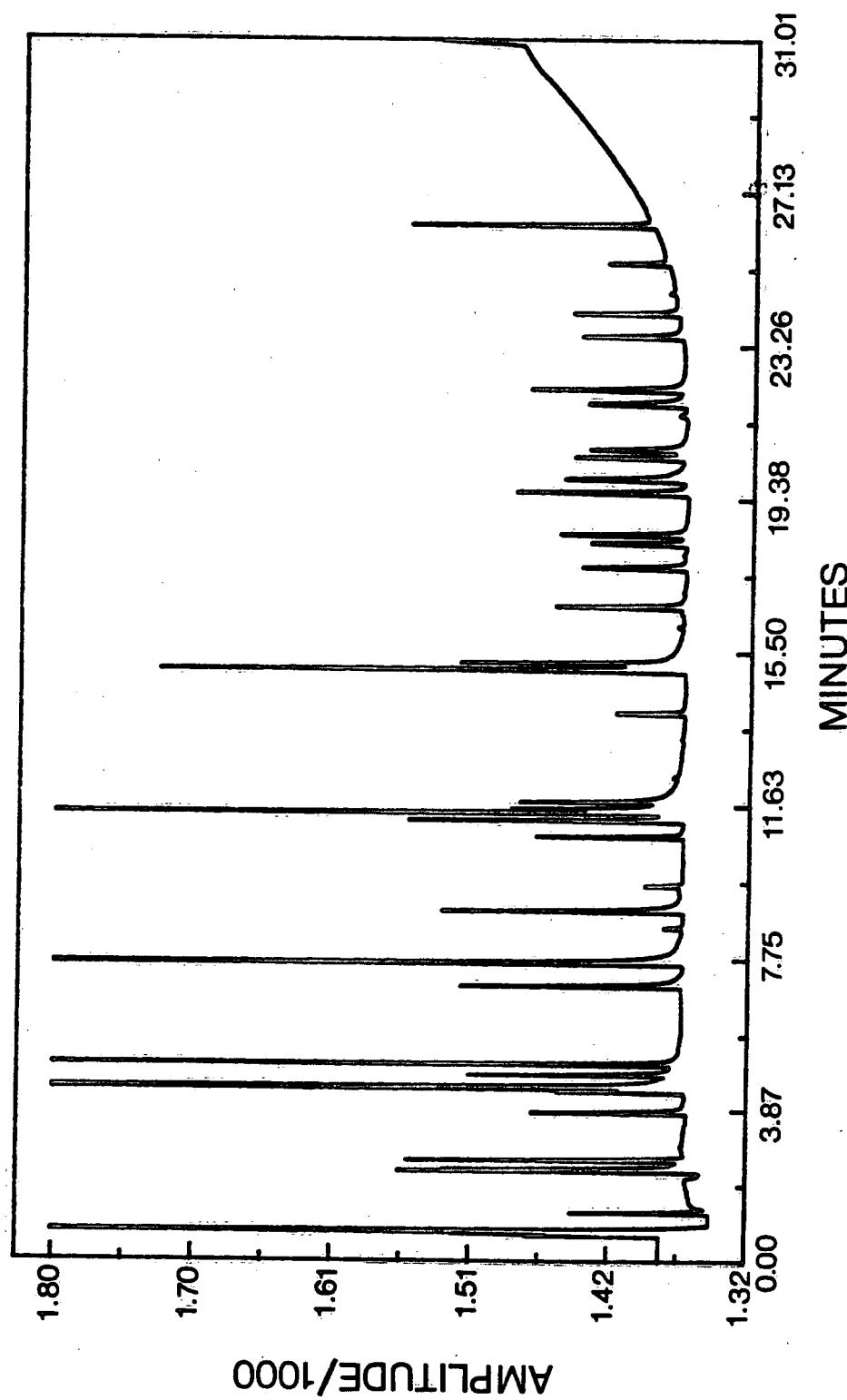


FIGURE 4

- a) Chromatogram of organochlorines and surrogates from SPB-608 column,  
in dual mode operation.
- b) Chromatogram of organochlorines and surrogates from XE-52 thin film  
column, in dual mode operation.

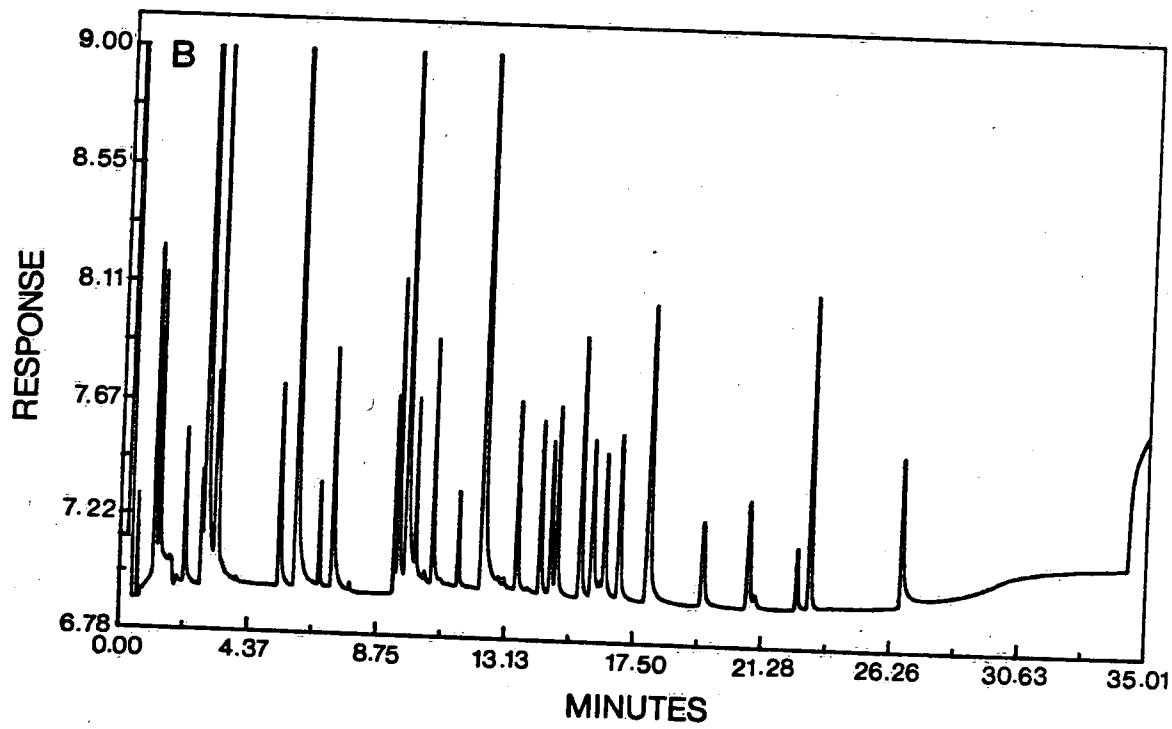
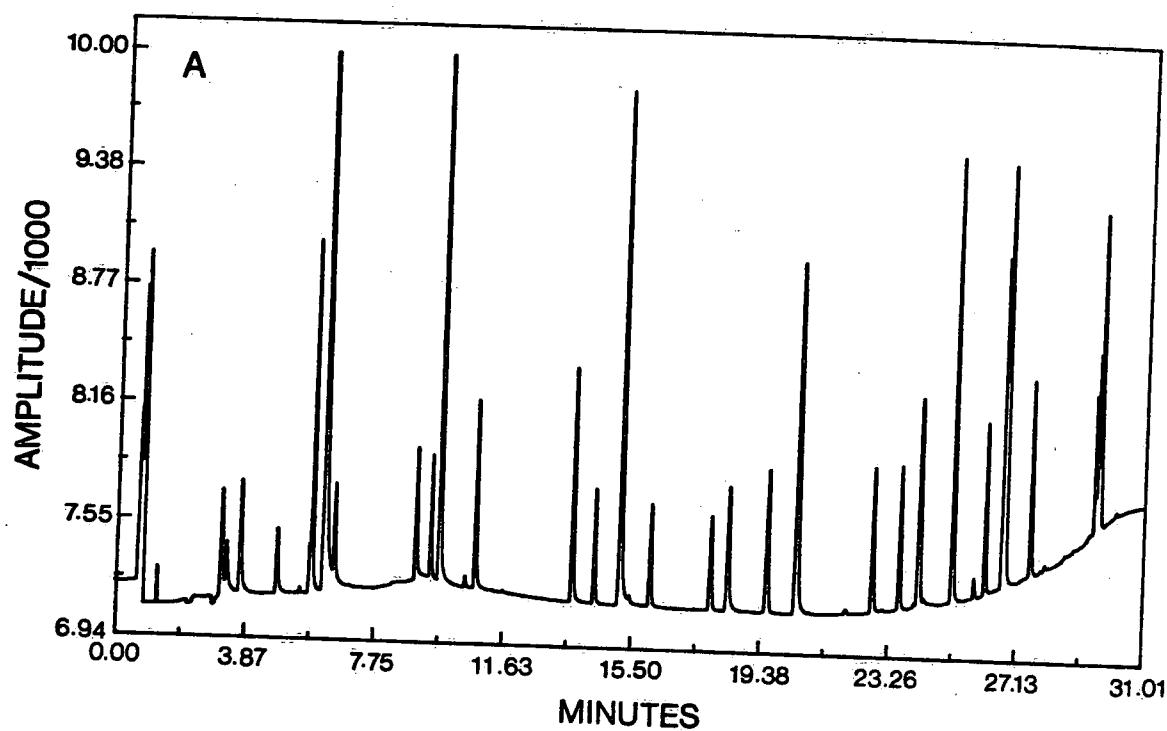


FIGURE 4.

FIGURE 5

Influence of the amount injected on the leading edge of peaks:

- a) 1  $\mu$ L of stock solution;
- b) 2  $\mu$ L of stock solution;
- c) 3  $\mu$ L of stock solution;

single column mode.

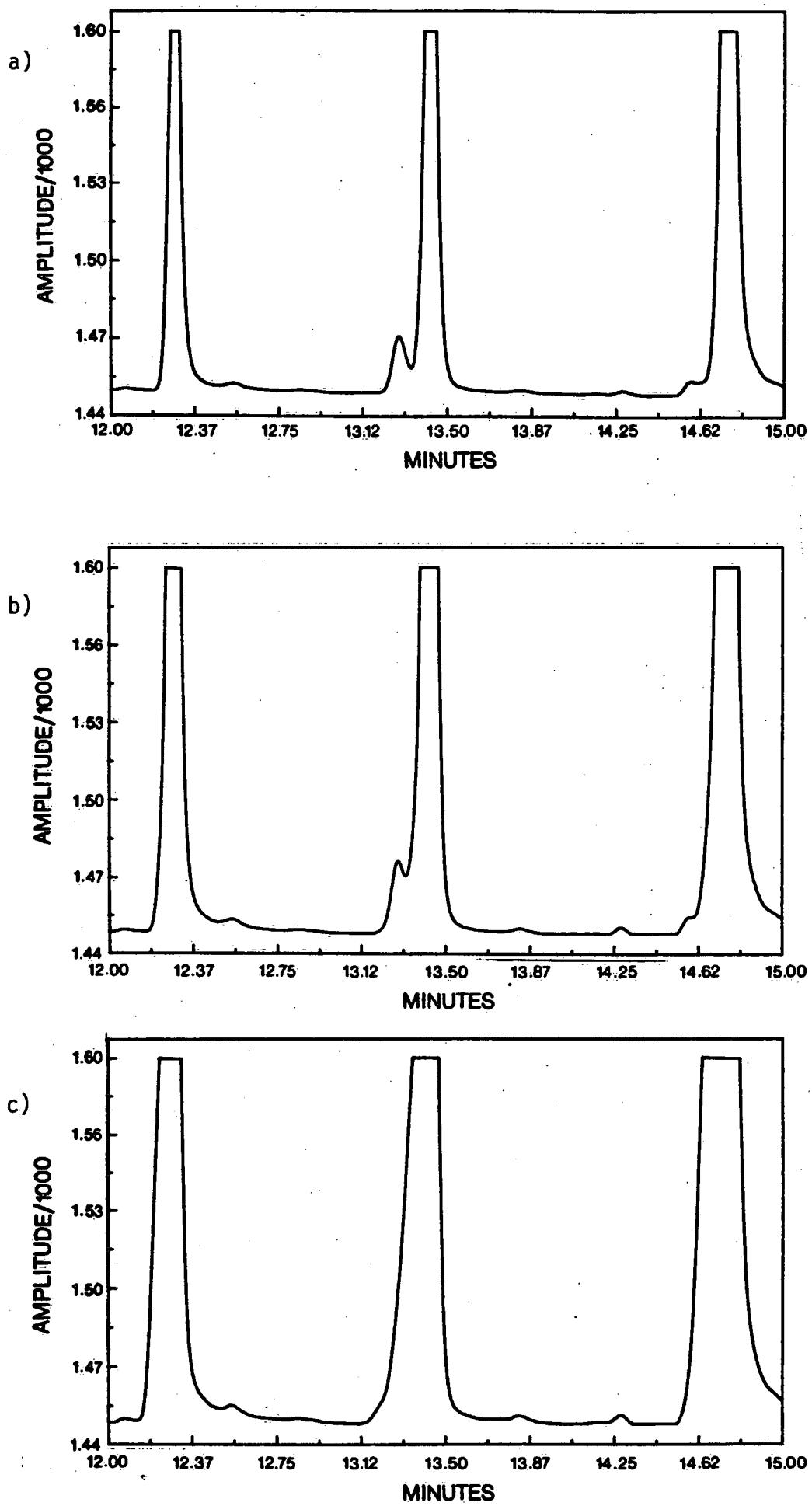


FIGURE 5.

FIGURE 6

Effect of peak spreading on position of baseline  
during integration of peak.

- a) early eluting peak: 2.9% error;
- b) intermediate eluting peak: 5.1% error
- c) later eluting peak: 12% error.

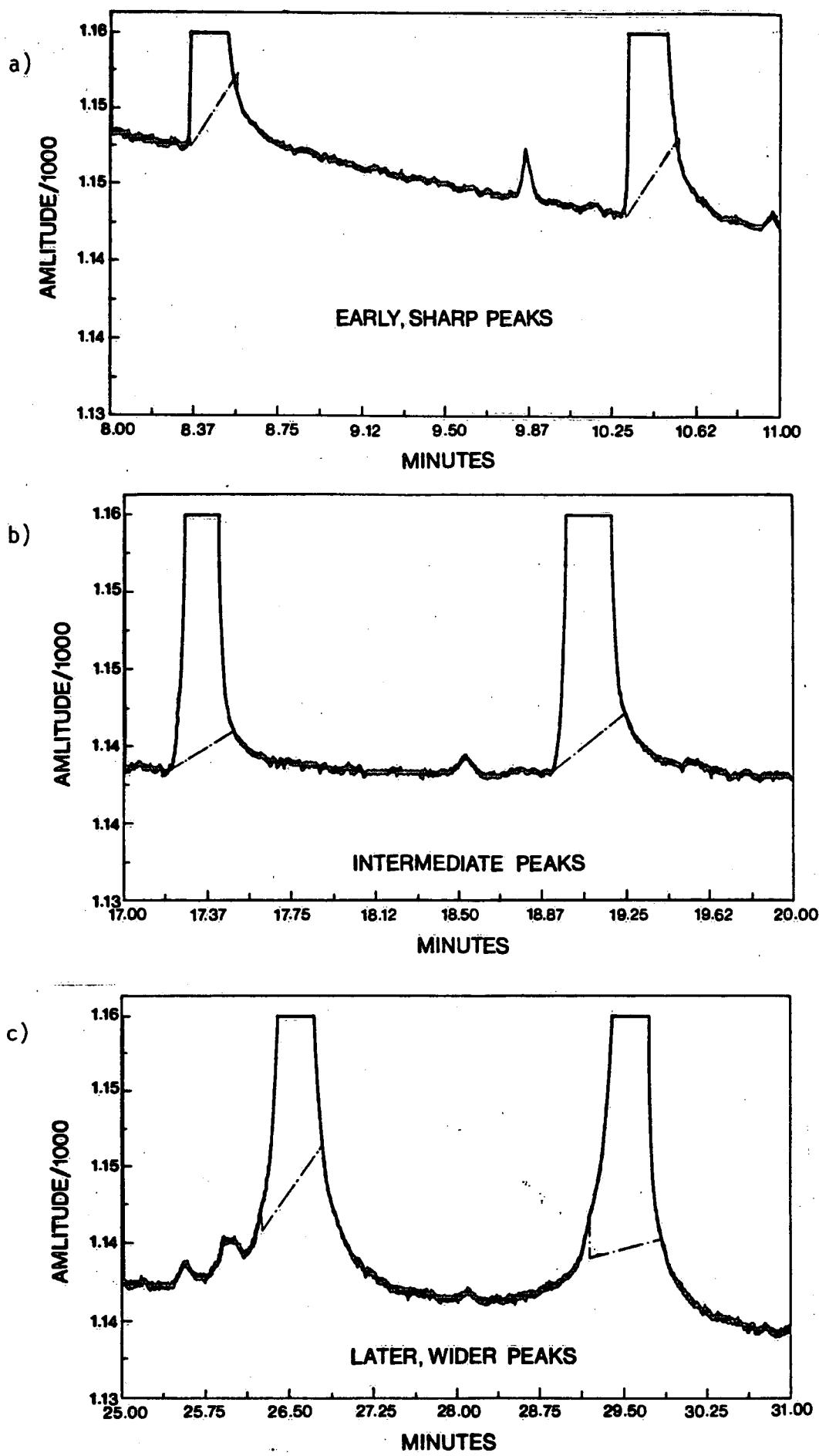


FIGURE 6.