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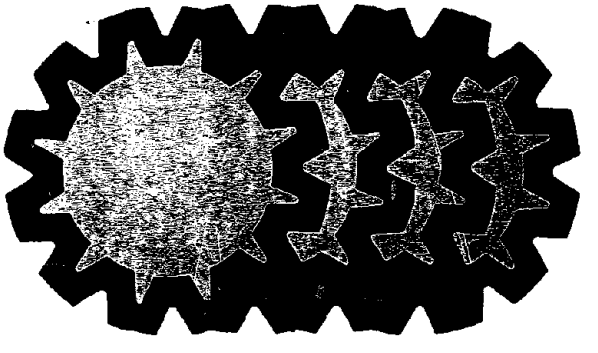
NIOSH

TECHNICAL INFORMATION

Collaborative Testing of Activated Charcoal Sampling Tubes for Seven Organic Solvents



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A charcoal tube sampling procedure and a gas chromatographic analytical technique are used in collaborative testing of activated charcoal sampling tubes for benzene, carbon tetrachloride, chloroform, dioxane, ethylene dichloride, trichloroethylene, meta-xylene, and two solvent mixtures. The study helps in fulfilling NIOSH's responsibilities under the Occupational Safety and Health Act of 1970 for the development of sampling and analytical procedures for toxic compounds for which standards have been set. Results indicate the overall error associated with the method and identify sources of error within each segment of the method.

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COLLABORATIVE TESTING OF ACTIVATED CHARCOAL SAMPLING TUBES FOR SEVEN ORGANIC SOLVENTS

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Contract No. HSM 99-72-98

U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
Public Health Service
Center for Disease Control
National Institute for Occupational Safety and Health
Division of Laboratories and Criteria Development
Cincinnati, Ohio 45202
June 1975

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ABSTRACT

The National Institute for Occupational Safety and Health has undertaken a program to provide reliable sampling and analytical methods for a number of industrial hygiene uses. Recommendation of methods to the Department of Labor for compliance testing and inclusion of methods in criteria for Recommended Standards for Occupational Exposures are two prime areas of utilization. This paper presents the results of our initial efforts in collaborative testing. The method tested is a charcoal tube sampling procedure and a gas chromatographic analytical technique.

Samples of seven single component solvents which included benzene, carbon tetrachloride, chloroform, dioxane, ethylene dichloride, trichloroethylene and xylene along with two solvent mixtures which consisted of a benzene-xylene combination and an ethylene dichloride-trichloroethylene combination were tested in two phases by 15 participating laboratories. The data analysis indicates the overall error associated with the method and identifies sources of error within each segment of the method.

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1.0 INTRODUCTION

The primary objective of this program was to determine whether the use of activated charcoal tubes should be presented by NIOSH as a recommended method of sampling for organic vapors. This study was performed to aid in fulfilling NIOSH's responsibilities under the Occupational Safety and Health Act of 1970 for the development of sampling and analytical methods for toxic substances for which standards have been set.

A method for the sampling and analysis of organic vapors using charcoal tubes had been developed at the NIOSH Cincinnati laboratory and it was being used routinely by NIOSH personnel. The most effective approach to determine the reliability of this method was through a collaborative testing program involving a representative cross-section of laboratories which would typically use such a method. The program objective was thus accomplished through a collaborative test program involving 15 participating laboratories and seven organic solvents.

In Phase I of the program the analytical method was evaluated by submitting 180 charcoal tubes with adsorbed organic solvents to each laboratory. The tubes were carefully prepared and monitored so that the amount of organic in each tube was very accurately known. The laboratories analyzed the tubes by the prescribed method and schedule, and the analytical error was determined through data analysis of the results. In Phase II both the sampling and analysis methods were evaluated. The participating laboratories collected charcoal tube samples using personal samplers. All of the participants sampled simultaneously from a large chamber at Scott's Plumsteadville laboratory. The samples were then analyzed and the method error determined as in Phase I.

The statistical data analysis provided estimates of errors for both sampling and analysis for each of the seven solvents. Separate estimates were made for replication error, between laboratory error and day-to-day error as well as total error.

2.0 PREPARATION FOR COLLABORATIVE TESTS

2.1 SELECTION OF COLLABORATING LABORATORIES

Fifteen laboratories were selected to participate in the collaborative testing. The NIOSH laboratories in Cincinnati and Salt Lake City participated on a volunteer basis. The remaining thirteen laboratories were selected on the basis of their being representative of the various types of laboratories which would utilize such a method, their capability and willingness to make available the required technical personnel and laboratory facilities, and the cost reimbursement required.

The participating laboratories included industrial hygiene laboratories, governmental laboratories, research and testing laboratories and a university laboratory. Some had direct experience with hydrocarbon analyses using charcoal tubes while others did not. However, all analysts were experienced in the use of gas chromatography for analysis of hydrocarbon mixtures.

2.2 DOCUMENTATION OF TEST METHOD

The sampling and analysis methods developed at the NIOSH Cincinnati laboratory were tested at Scott's Plumsteadville laboratory. The purpose was to determine if additional details or clarification were necessary in the method documentation to assure that all of the collaborators would carry out the sampling and analytical procedures similarly and in the manner practiced at NIOSH. Decisions were also made in consultation with the Project Officer and other NIOSH personnel as to those steps of the method which had to be done as specified, e.g., size of sample injection, and those which were optional with the analysts, e.g., the chromatographic separation column. The approved version of the sampling and analytical methods were then distributed to the collaborators. These methods are included in Appendix A.

2.3 TESTING OF CHARCOAL TUBES

Nine thousand charcoal tubes for organic vapors were purchased from Mine Safety Appliance Company. These tubes were prepared from a

single batch of charcoal by a procedure which had yielded satisfactory results for previous batches acquired by NIOSH.

Random samples of the charcoal tubes were selected to determine if they met NIOSH specifications. The most important specification is that the pressure drop across the charcoal tubes does not exceed one inch of mercury at a flow rate of one liter per minute. The pressure drop test was performed with the apparatus shown in Figure 2-1. The majority of tubes yielded pressure drops of 0.9 to 1.0 inches. Only one of 36 organic tubes exceeded 1.05 inches. Samples were submitted to the NIOSH Cincinnati laboratory and NIOSH concurred with Scott that the tubes should be satisfactory for program use.

Another property of the tubes which was measured was the weight of charcoal they contained. The specifications state that the adsorbing section should contain 100 mg. of charcoal and the back-up section 50 mg., but no tolerances are specified. Thirty-eight tubes were selected at random, broken and the contents of each section weighed. Of the 38 tested, 11 had adsorbing sections which deviated by more than 10% from 100 mg., 7 had back-up sections which deviated by more than 10% from 50 mg., and 4 had total weights which deviated from 150 mg. by more than 10%. Discussions with NIOSH personnel resulted in the conclusion that the charcoal weight variations should not adversely affect the tubes' ability to adsorb organics efficiently.

2.4 SEMINAR FOR ANALYSTS

A one-day seminar for the analysts from the collaborating laboratories was held at the NIOSH Cincinnati laboratory. The seminar was conducted by Scott with NIOSH personnel on hand to demonstrate techniques and answer questions on the method. The purpose of the seminar was to assure that the analysts fully understood the test method which had been submitted to them; and that they would perform the analyses in the specified manner. The seminar also presented the opportunity to determine if there was sufficient clarity in the method documentation.

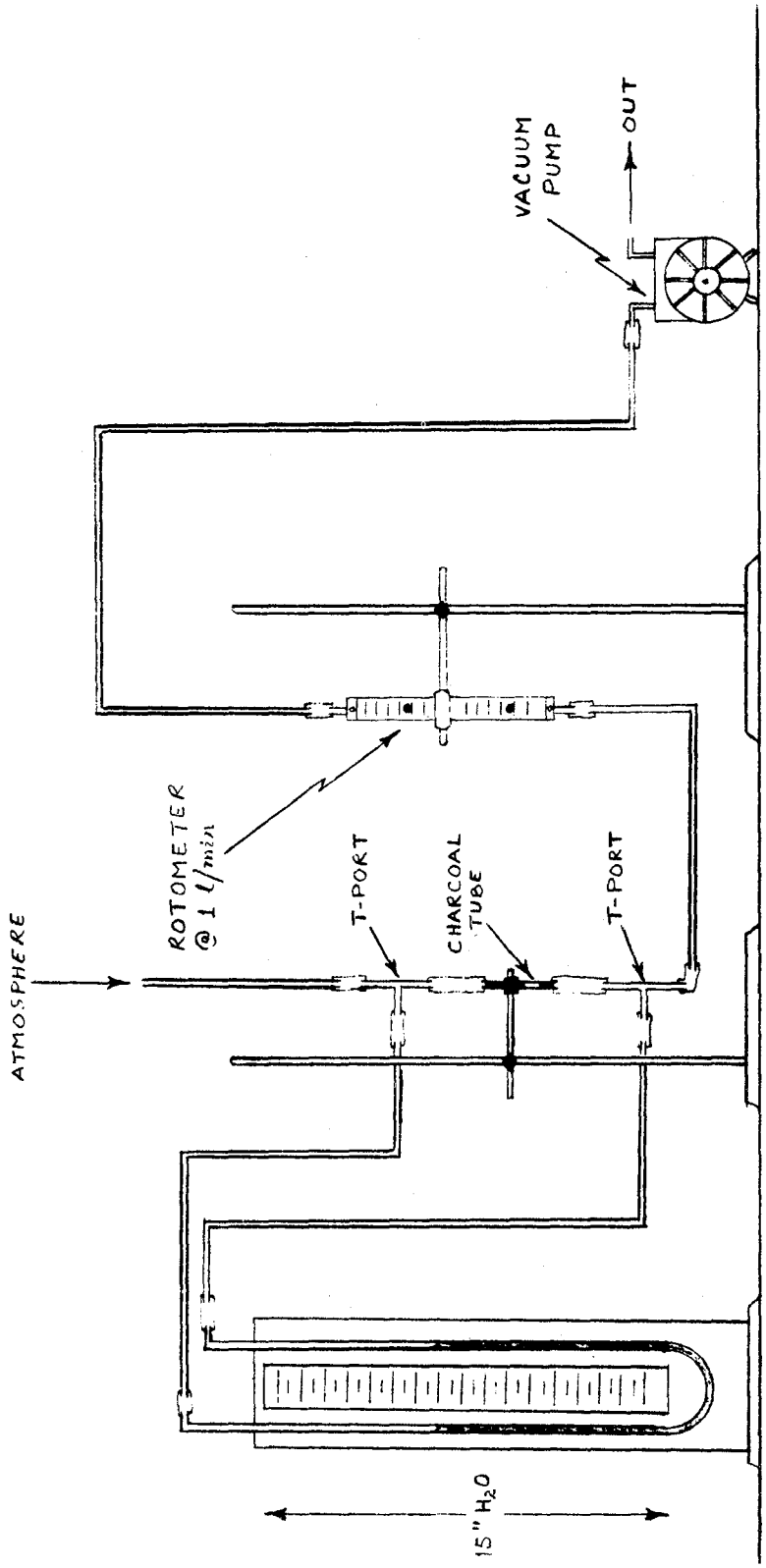

FIGURE 2-1 PRESSURE DROP APPARATUS

The theme of the seminar was that an analytical method had been developed by NIOSH and used successfully in-house. The object now was to determine in a realistic manner the quality of data which could be obtained by other analysts following the method precisely. It was recognized that some analysts had used similar methods with variations they believed could produce improved results. However, it was stressed very strongly that the object was to test the specific method presented and not to evaluate any modifications. The analysts were told that this was exclusively a collaborative test program, not a method development program.

The extent to which collaborators can be coached without biasing a collaborative program is subject to debate. In this case it was felt that the seminar was needed to assure that the analysts understood the method. In no case was any analyst permitted to perform any of the analytical steps at the seminar. Any such activity would constitute training and was strictly avoided because it would bias the results.

3.0 COLLABORATIVE TEST PROGRAM

The overall program was divided into two phases. In Phase I the collaborators analyzed samples prepared by Scott. In Phase II the collaborators collected samples at Scott's Plumsteadville, Pa. laboratory and analyzed the samples in their home laboratories.

The collaborative test program involved seven solvents present singly at up to five concentrations including 5%, 60%, 100% and 160% of the TLV, and the excursion limit. Two binary mixtures were included at the TLV for the two components. The 36 solvent/concentration combinations tested are shown in Table 3-1. Ten liter samples of each of these mixtures were specified for use in preparing the charcoal tubes.

3.1 EXPERIMENTAL DESIGN OF PHASE I ANALYSIS

The design of any data gathering experiment should be based on a knowledge of the information needed as input for the subsequent data analysis and on an understanding of any constraints imposed upon the program. In the case of the Phase I analysis this meant obtaining the data needed to perform the data analysis described in Section 4.1 within the magnitude of effort to which the collaborators had agreed.

Each of the 15 collaborators was to analyze 144 samples containing one or two of seven organics over a ten-day period. The 144 samples were composed of 4 replicates each of 36 compound/concentration combinations. It was desired to obtain data to calculate the systematic error (inter-laboratory error), replication error, and day-to-day error as well as accuracy at each level for each organic. It was also desired to determine the amount of organic, if any, adsorbed on the back-up section of each charcoal tube in addition to that adsorbed on the front section.

The method specified that standards for each organic be prepared fresh daily. If standards for all seven organics were prepared daily, excessive time would have been necessary. The design was therefore set to limit the analysis to two organics on any given day. It was felt that this would

TABLE 3-1 ORGANIC VAPOR ATMOSPHERES TESTED

Ref. No.	Component	Conc., ppm
A-10790	Benzene	0.543
A-10791	Benzene	6.25
A-10792	Benzene	10.3
A-10793	Benzene	19.3
A-10794	Benzene	48.7
A-10795	Carbon Tetrachloride	.436
A-10796	Carbon Tetrachloride	6.78
A-10797	Carbon Tetrachloride	11.02
A-10798	Carbon Tetrachloride	23.04
A-10799	Carbon Tetrachloride	245
A-10800	Chloroform	2.44
A-10801	Chloroform	31.9
A-10802	Chloroform	50.6
A-10803	Chloroform	98.6
A-10804	Dioxane	6.25
A-10805	Dioxane	59.6
A-10806	Dioxane	106.7
A-10807	Dioxane	162
A-10808	Dioxane	192
A-10809	Ethylene Dichloride	2.41
A-10810	Ethylene Dichloride	34.2
A-10811	Ethylene Dichloride	53.1
A-10812	Ethylene Dichloride	94.4
A-10813	Ethylene Dichloride	209
A-10814	Trichloroethylene	3.37
A-10815	Trichloroethylene	65.0
A-10816	Trichloroethylene	106.8
A-10817	Trichloroethylene	185.0
A-10818	Trichloroethylene	273.5
A-10819	m-Xylene	4.75
A-10820	m-Xylene	57.9
A-10821	m-Xylene	111
A-10822	m-Xylene	153
A-10823	m-Xylene	193
A-10824	Benzene, m-Xylene	10.9, 103
A-10825	Ethylene Dichloride, Trichloroethylene	51.1, 104

greatly increase the efficiency of the experiment without causing bias or limiting the utility of the data in any way. The analysis of all back-up sections would also require too much time, so it was decided to analyze the back-up section from only one of each set of four replicate samples.

The four replicate samples of each compound/concentration combination were divided into two pairs. Both members of a pair were to be done on the same day. The remaining parameters were randomized within the experimental design.

The analysis schedule for the ten days is shown in Table B-1 in Appendix B. The samples marked with an asterisk had the back half charcoal sections also analyzed. Table B-2 in Appendix B shows the randomized schedule of analysis days for each of the fifteen participating laboratories.

3.2 GENERATION OF PHASE I SAMPLES

The apparatus used for generation of the Phase I samples is shown in Figure 3-1. The gas mixtures were prepared in air in high pressure cylinders and analyzed to an accuracy of ±2% by Scott's Measurement Standards Division. The cylinder contents were analyzed by gas chromatography against primary standards prepared by Scott's Measurement Standards Division following proprietary procedures utilized by that Division in producing close-tolerance gas mixtures for sale to government and industry. Basically the primary standards are prepared by injecting a known amount of the trace component with a calibrated microliter syringe into a calibrated 5-liter glass flask and pressurizing the flask to a known pressure with high purity nitrogen.

The cylinders were attached to the sample generation apparatus. The gas passed through a pressure regulator, flow control valve, charcoal tube, flowmeter and wet test meter. Four samples were collected simultaneously in the four separate sampling legs. The wet test meters were calibrated both before and after the sample generation. The corrected wet test meter data were used to calculate the quantity of organics passed through the charcoal tubes. The flow meters were used for adjusting the sample flow rate to approximately one liter per minute.

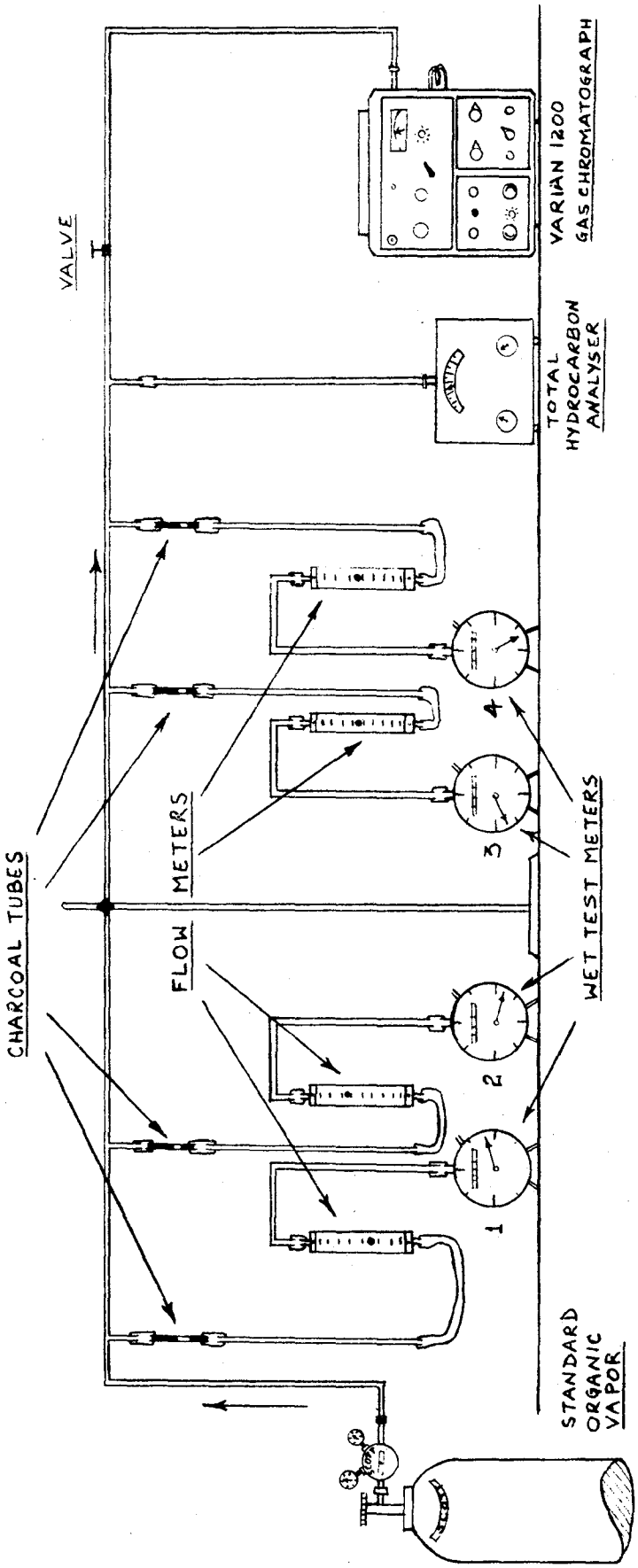

FIGURE 3-1 VAPOR COLLECTION MANIFOLD

The composition of the gas flowing through the system was monitored continuously with a flame ionization total hydrocarbon analyzer and periodically with a gas chromatograph. The operating details of these instruments are listed in Table 3-2. Analyses were made alternately directly from the cylinder and from the flow system until the two gave identical readings. This indicated that the system had equilibrated and collection in charcoal tubes commenced. During the collection period the instrument readings verified that the system concentrations did not change. The data recorded were similar to that shown in Figure 3-3 which relates to the Phase II sample generation.

Eighteen samples of each test gas mixture were collected on each wet test meter. This provided a set of samples for each of the fifteen laboratories as well as three spare sets. The program design called for analysis of four replicate samples from each mixture. A set of 18 samples was collected in each of the four sampling legs. The volume sampled in the four legs ranged from 9 to 11 liters, but was constant for each leg. A fifth set of 18 samples was then prepared to provide the collaborators with spares in case one of the four tubes scheduled for analysis was broken or the sample was lost. The manner of collection described above ensured that identical sets of samples would be submitted to each laboratory. This is most important in achieving a valid assessment of the systematic error (inter-laboratory error). The introduction of deliberate variations within each set of four replicate samples was made to eliminate the temptation of revising analytical data to reduce variations between identical samples. This cannot be allowed if the calculated within day and day-to-day errors are to be valid.

Each tube was capped with plastic ends provided by MSA and labeled with an eight digit identification number. A set of 180 tubes with adsorbed organics and 70 blank tubes for desorption determinations were shipped to each collaborating laboratory. Bulk samples of each organic solvent were shipped to each laboratory under separate cover from the sample tubes.

TABLE 3-2 HYDROCARBON ANALYSIS INSTRUMENT CONDITIONS

Gas Chromatograph

Model: Varian 1200

Fuel: Hydrogen @ 20 psi

Combustion Gas: Oxygen @ 30 psi

Carrier: Helium @ 40 psi

Detector Temperature: 200°C

Sample Size: 1 cc

Column: Carbowax 20M (20' x 1/8")

Isothermal Temperature: 80°C to 110°C (depending on the compound)

Total Hydrocarbon Analyzer

Model: Beckman 108A

Fuel: 40% H_2 in N_2

Purge Gas: Air

3.3 EXPERIMENTAL DESIGN OF PHASE II ANALYSIS

The experimental design and sampling schedule for Phase II was quite similar to that used in Phase I. Phase II was performed primarily to determine the magnitude of errors due to sampling by the specified method. This could be accomplished by comparing the errors in Phase II, which would include both sampling and analytical errors, to those from Phase I which were due solely to analytical error. This assumes that the Phase I sample generation errors were negligible. The drawback to this approach was that the errors due to analysis in Phase I and Phase II would have to be assumed to be identical or the sampling error could not be defined.

There were reasons to believe that analytical error might be significantly reduced from Phase I to Phase II. First of all, familiarity through use of the method would be expected to improve data. Second, it was learned that some collaborators who had noted deficiencies in their chromatographs in Phase I were taking steps to improve them. Finally, because of the poor results some laboratories obtained for the low concentrations (5% of TLV) in Phase I, it was decided to require standards within a factor of two of all samples, not just the TLV as in Phase I.

In order to estimate the relationship between analytical error in Phase I and Phase II, it was decided to analyze some of the spare samples prepared for Phase I, but not analyzed, as part of Phase II. Thus, 22 Phase I samples were scheduled for analysis by each laboratory in Phase II. In order to keep within the scope of effort, the analysis of back-up sections was omitted for the lower concentrations. At these levels little or no organics had been found on the back-ups in Phase I. Table C-1 in Appendix C shows the analysis schedule for Phase II samples. The samples marked with a single asterisk had the back-half charcoal sections also analyzed. The samples marked with a double asterisk were the Phase I spare samples. The randomized schedule of sampling days for each of the fifteen laboratories is presented in Table C-2.

3.4 GENERATION OF PHASE II SAMPLES

Collection of the Phase II samples by representatives from the 15 collaborating laboratories was performed at Scott's Plumsteadville laboratory. The sample collection was accomplished using apparatus illustrated in Figure 3-2. Gas mixtures from each of the 36 high pressure cylinders were flowed into one of two test chambers. The chambers were approximately 180 and 60 liters in capacity, and the internal surfaces consisted only of glass, Teflon and stainless steel. Each chamber was fitted with separate sampling lines for each sampler plus a line for monitoring the chamber contents. The chambers were first flushed with several volumes of the test mixture. Then a gas chromatographic analysis of the chamber was made and compared to a similar analysis of the cylinder mixture. More flushing time was allowed where needed, and the samplers were not given the signal to proceed until the chamber had reached equilibrium. During sampling the chambers were maintained at a positive pressure of 1" of water to prevent dilution by room air. The contents of the chamber were monitored continuously with a total hydrocarbon analyzer as well as intermittently by gas chromatography. A diagram of the monitoring data recorded during a typical sampling period is shown in Figure 3-3.

The sample pumps were all calibrated at the beginning of each day using the NIOSH procedure which had been made a part of the test method. No further calibration was permitted during the day. The only adjustments allowed were to bring the flow meter float back to its calibrated level. The flow rates were checked by Scott at the end of each of three days in order to estimate sampling error and deviations occurring during the day's use.

Once the signal to sample was given, each sampler collected five consecutive ten liter samples on tubes provided by Scott. Scott also provided the tube identification labels. When all of the samplers had collected five samples, the alternate chamber was moved into place, checked out and the next set of samples collected. Approximately one hour was required for each set.

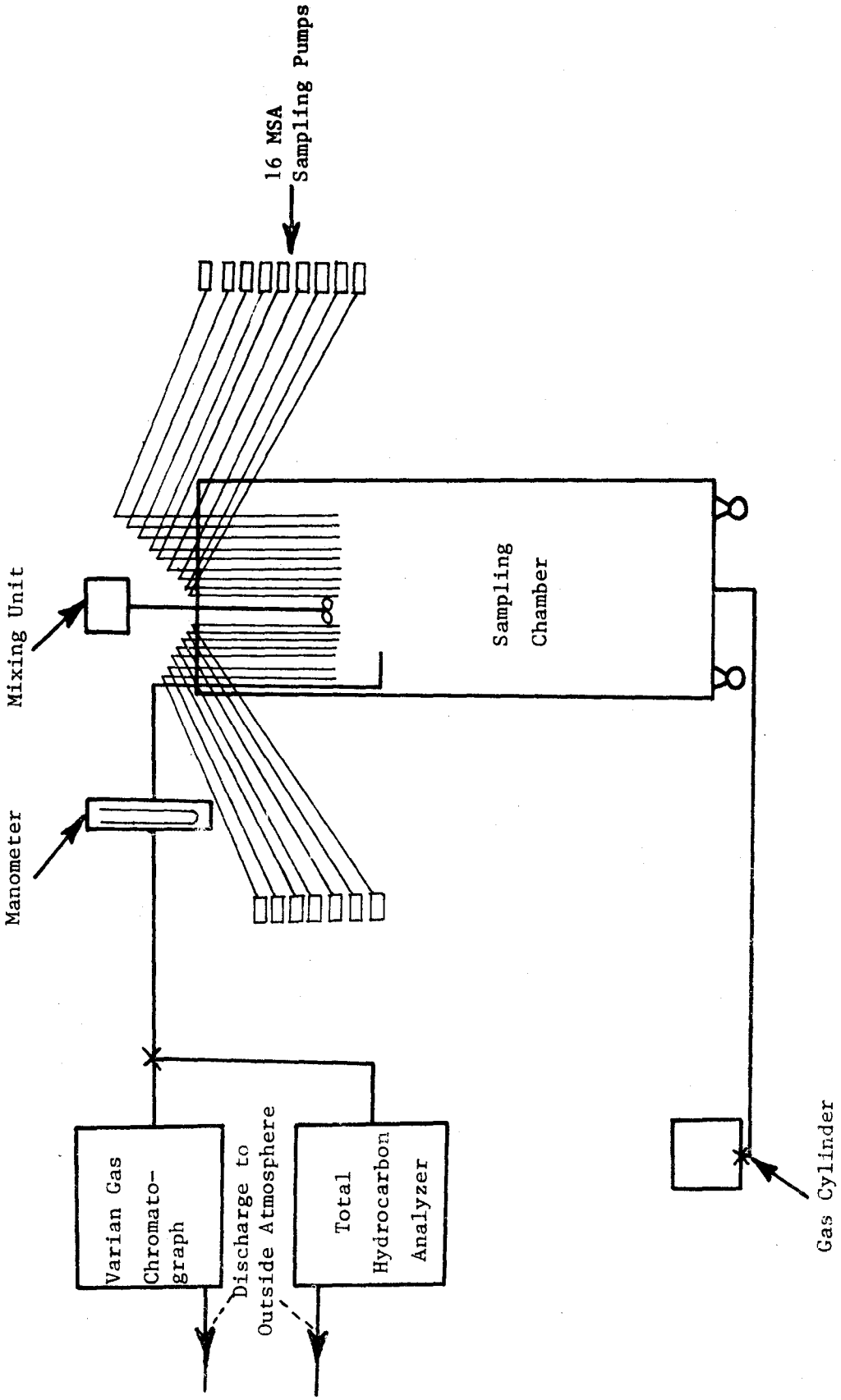

FIGURE 3-2 REPLICATE GAS SAMPLING UNIT

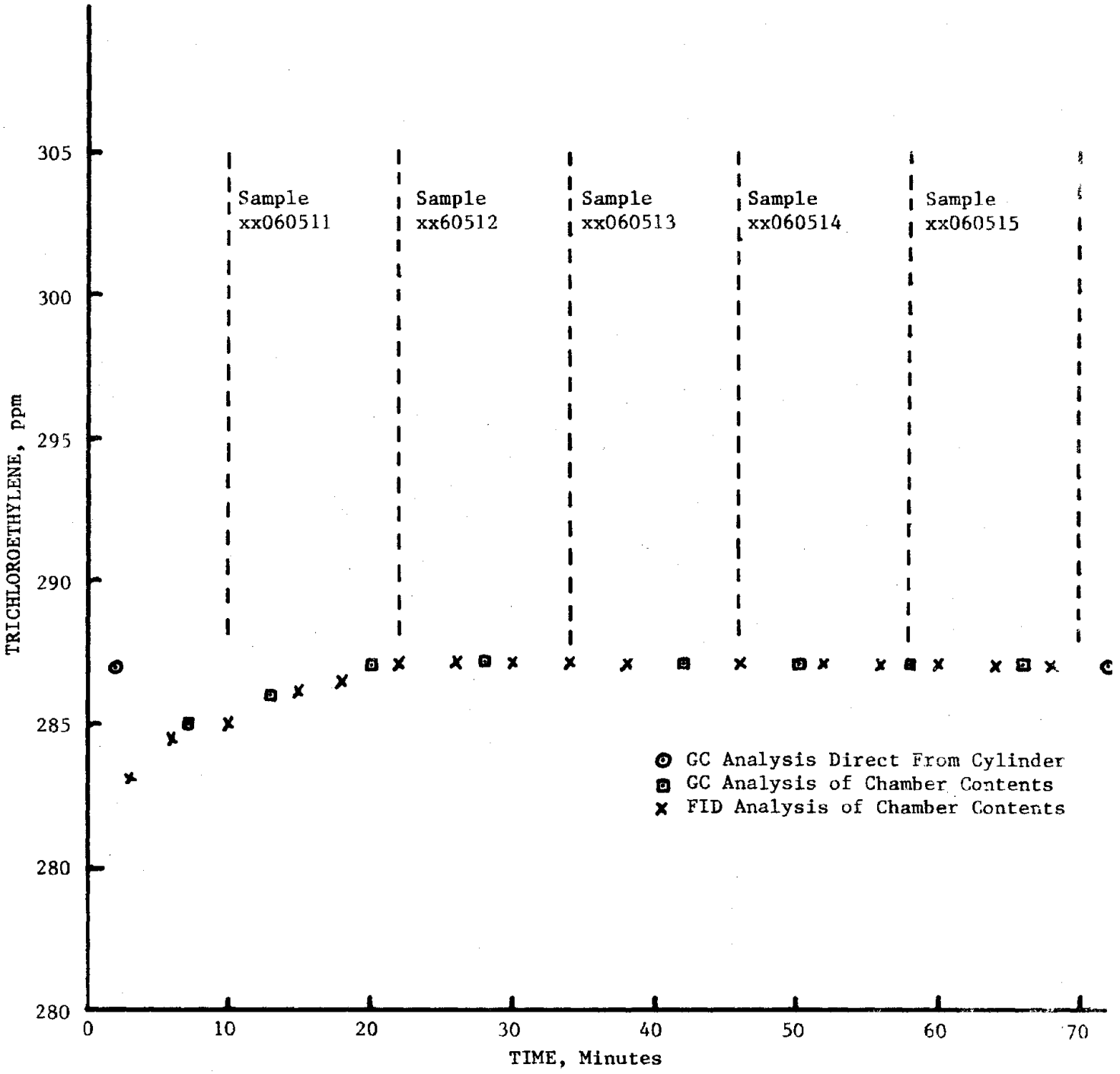

FIGURE 3-3 TRICHLOROETHYLENE MONITORING DURING PHASE II SAMPLE COLLECTION

3.5 ANALYSIS OF PHASE I AND PHASE II SAMPLES

The samples collected in Phase I and Phase II were analyzed in the collaborators' laboratories according to the schedules shown in Appendix Tables B-1 and C-1 respectively. The chromatographs and columns used by the various collaborators are listed in Table 3-3. The data acquired by the collaborators were entered on data sheets provided by Scott and then mailed to Scott upon completion of each phase.

TABLE 3-3

Chromatographs and Columns Used by Collaborators

Lab No.	Chromatograph Make/Model(s)	Column Stationary Phase(s)
1	Varian 2700	DC-200
2	Varian Aerograph 1520	Di isodecylphthalate, Carbowax 1540, FFAP
3	Hewlett Packard 700	FFAP, Porapak Q
4	Hewlett Packard 7620A	Durapak (Carbowax 4000)
5	Beckman GC-55, Barber-Colman 20	Apiezon L, Porapak Q
6	Hewlett Packard 5750	UC-W98
7	Hewlett Packard 7820 & 562	DC-200, PEG 600-Terephthallic Acid
8	Varian Aerograph 204B	FFAP
9	F & M Scientific 400	FFAP
10	Varian 1800	FFAP, Carbowax 20M
11	Barber-Colman 5000	FFAP
12	Perkin-Elmer 881	FFAP
13	---	---
14	Perkin-Elmer 900, Hewlett Packard 7600A	FFAP
15	Perkin-Elmer 900 & 990	FFAP

4.0 DATA ANALYSIS

The experimental design outlined in Section 3.0 lends itself very readily to the statistical analysis outlined by W. J. Youden in References 1 and 2. The replicates on any one day comprise what is known as a "unit-block" and can be studied both graphically (on a two-sample chart) and numerically in establishing the precision (within laboratory error) and systematic error (between laboratory error) of the method. By averaging the replicates on a day, the averages on two different days can be looked upon as another set of similar pairs "unit-block" and the analysis for precision and systematic error performed on this set of data. By studying these results and the results obtained from the unaveraged data, estimates of the day-to-day error component can also be evaluated. The precision errors obtained at each level can then be studied to ascertain if any homogenity exists in the errors from level to level. If homogeneity is seen to be present, pooled estimates of the error components can be established for each compound. These pooled estimates then provide estimates of the errors associated in analyzing a compound for the range of concentration levels under which the study was conducted.

Prior to subjecting the raw data to the analysis outlined briefly above, identification of outliers and missing data has to be considered. Considerable care has to be taken in this step of the analysis. A very stringent test for outliers can cause the very important degrees of freedom associated with laboratories to be drastically reduced, resulting in distorted estimates of error which do not reflect the true merit of the method. On the other hand, if data which lie well outside the general pattern exhibited by the laboratories are included in the analysis, grossly erroneous conclusions about the method may be reached. Moreover, inclusion of such outliers in an analysis can cause an unfair evaluation of the laboratories whose data reflected the true merit of the method.

In the Youden analysis, if one member of a pair is an outlier or missing value, the pair is excluded from the analysis. The exclusion of such a pair reduces by one the degrees of freedom available for that pair, but in no way complicates or hinders the statistical analysis of the data. Moreover, the "unit-block" concept and the two-sample chart illustrate very graphically the problems associated with outliers being included in an

analysis. This elegance and "robustness" of the Youden analysis merit themselves exceedingly well in data obtained from collaborative studies.

4.1 DATA ANALYSIS OF ANALYTICAL RESULTS FROM PHASE I

The analytical results received from the collaborating laboratories in Phase I are summarized in Appendix Table B-3. The numbers have been rounded off to two decimal places so that the results from all laboratories for a specific sample could be presented on a single sheet. In all of the data analysis, however, the complete set of digits reported by the collaborators was used. The asterisks indicate missing values and outliers as determined in the following section.

4.1.1 Identification of Outliers and the Two-Sample Chart

For each sample analyzed, replicate experimental measurements were made, averaged and then divided by a desorption factor to obtain the corrected amount of the compound present in milligrams. This corrected weight was then used as representing the amount present for a single sample in the statistical analysis. Some laboratories made as many as four repeat measurements for each sample; others performed (or reported) only the results from a single measurement for a sample.

Prior to testing the data for outliers, the numbers were carefully checked for gross computational or reporting errors. Once such obvious errors had been identified and the necessary alterations had been made, the results from the fifteen laboratories on each replicate on each day were subjected to the outlier test, "Recommended Criteria for Single Samples" (Ref. 3). A 2.5% significance level was used for the outlier test criteria and the test considers the possibility of outliers occurring either on the high side or the low side.

Initial outlier tests on the data indicated Laboratory #13 to have an unusually high number of outliers. Moreover, most of these outliers were well separated from the main cluster represented by the other laboratories. In fact, over 40% of the data reported by Laboratory #13 showed up prominently as outliers. A similar observation was noticed for Laboratory #1 for the dioxane samples. It was decided, upon consultation

with the Project Officer, to discard Laboratory #13's data from the analysis and Laboratory #1 from the dioxane analysis. It should be pointed out, however, that before rejecting any data points as outlying observations, the presence of such data points were discretely brought to the attention of the concerned laboratory to determine if such data were due to reporting errors or errors other than analytical errors. The total number of data pairs for each laboratory in which one or both of the members were determined to be outliers are given in Table 4-1. Data are included for both Phase I and Phase II.

TABLE 4-1 NUMBER OF OUTLIER DATA PAIRS

Lab. No.	Phase I	Phase II
1	15	19
2	0	0
3	3	1
4	9	10
5	4	2
6	1	0
7	1	15
8	1	0
9	3	1
10	6	13
11	0	0
12	0	0
13	40	35
14	0	1
15	5	5

Two illustrative examples of the outlier test are presented in Tables 4-2 through 4-5, and the corresponding two sample charts are shown in Figures 4-1 and 4-2. Tables 4-2 and 4-3 and Figure 4-1 are examples of the outlier test on the analytical results obtained for benzene on the two replicates on the second day at the fourth level. Similarly, Tables 4-4 and 4-5 and Figure 4-2 are examples for dioxane on the two replicates on the first day at the fifth level. The examples shown in these tables have data from Laboratory #13 and Laboratory #1 (for dioxane) already excluded.

TABLE 4-2 OUTLIER TEST ON FIRST REPLICATE
ON SECOND DAY OF FOURTH LEVEL OF BENZENE - PHASE I

.5467
.6000
.6450
.6535
.6648
.6681 BENZENE xx010403
.6810
.6829
.6850
.6907
.7080
.7230
.7836
.8490

1.0 1.0 .0 3.0 5.0 2.0 .0 1.0 .0 1.0

R	.5467 - .8490
Md	.6819
Mn	.6843
S D	.07247
S E	.01936
C V	10.58
P E	.04888

TABLE 4-3 OUTLIER TEST ON SECOND REPLICATE ON SECOND DAY OF FOURTH LEVEL OF BENZENE - PHASE I

.5428
.6142
.6452
.6770
.6920
.6993
.7060
.7131
.7147
.7280
.7380
.7450
.8200
1.5550

BENZENE xx010404

2.0 10.0 1.0 .0 .0 .0 .0 .0 .0 1.0

R	.5428 -	1.5550
Md	.7095	
Mn	.7564	
S D	.23877	
S E	.06381	
C V	31.56	
P E	.16105	

Out 1.5550 (10)

.5428
.6142
.6452
.6770
.6920
.6993
.7060
.7131
.7147
.7280
.7380
.7450
.8200

1.0 .0 1.0 1.0 1.0 3.0 3.0 2.0 .0 1.0

R	.5428 -	.8200
Md	.7060	
Mn	.6950	
S D	.06734	
S E	.01867	
C V	9.68	
P E	.04542	

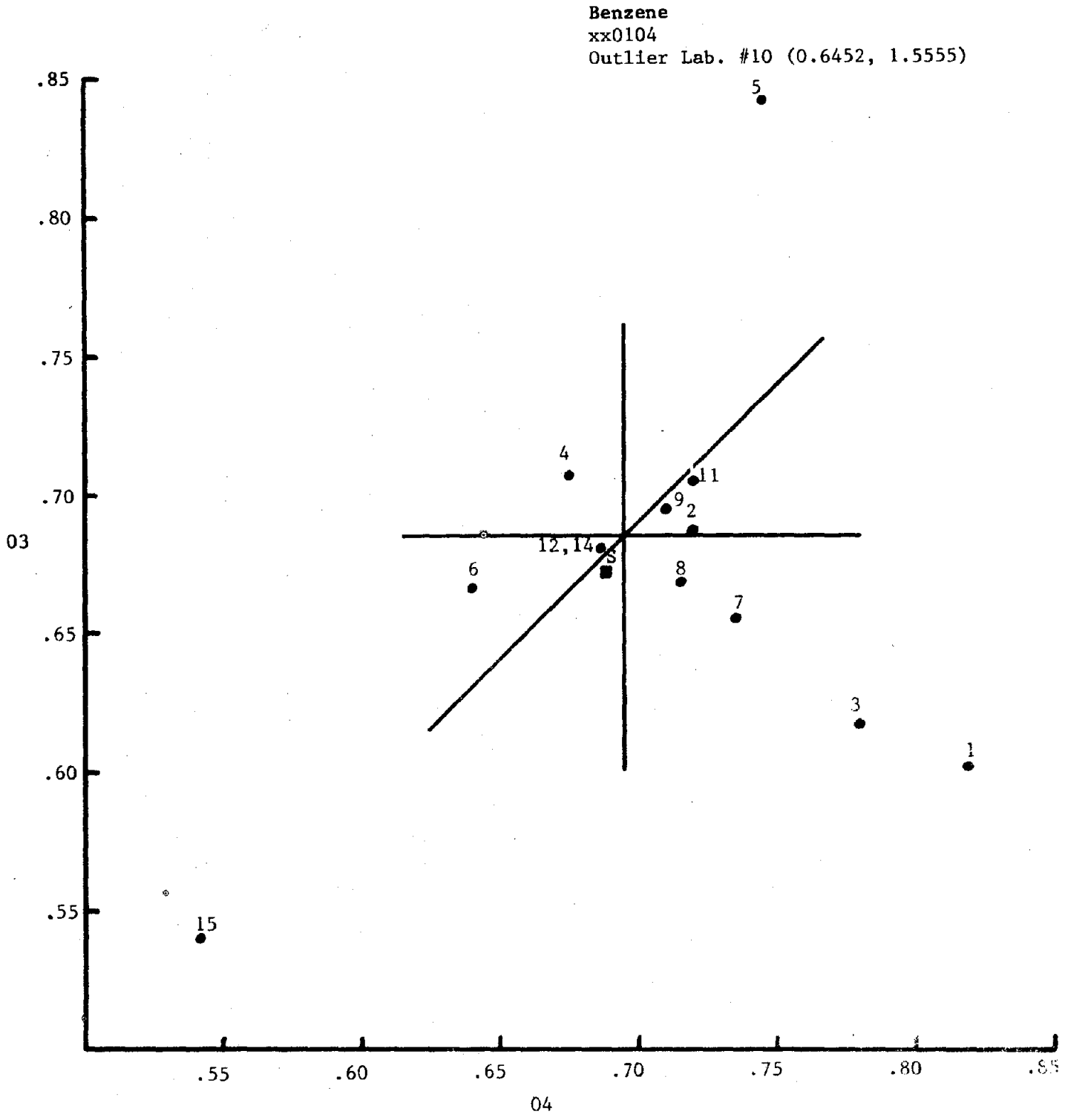

FIGURE 4-1 TWO-SAMPLE CHART OF REPLICATES ON SECOND DAY AT FOURTH LEVEL OF BENZENE - PHASE I

TABLE 4-4 OUTLIER TEST ON FIRST REPLICATE ON FIRST DAY OF FIFTH LEVEL OF DIOXANE - PHASE I

5.4500
5.5217
5.7000
5.7661
5.8520
5.9200
5.9331
6.1437
6.2813
6.3300
6.5987
6.6470
7.9740

DIOXANE xx040501

3.0 4.0 1.0 2.0 2.0 .0 .0 .0 .0 1.0

R	5.4500 -	7.9740
Md	5.9331	
Mn	6.1628	
S D	.66119	
S E	.18338	
C V	10.72	
P E	.44597	

Out 7.9740 (#5)

5.4500
5.5217
5.7000
5.7661
5.8520
5.9200
5.9331
6.1437
6.2813
6.3300
6.5987
6.6470

2.0 .0 2.0 2.0 1.0 1.0 1.0 1.0 .0 2.0

R	5.4500 -	6.6470
Md	5.9265	
Mn	6.0119	
S D	.39228	
S E	.11324	
C V	6.52	
P E	.26459	

TABLE 4-5 OUTLIER TEST ON SECOND REPLICATE ON SECOND DAY OF FIFTH LEVEL OF DIOXANE - PHASE I

6.0000
6.3527
6.5796
6.6000
6.6270
6.7020
6.8450
7.0421
7.1300
7.2321
7.2524
7.3102
9.5230

DIOXANE xx040502

1.0 5.0 2.0 4.0 .0 .0 .0 .0 .0 1.0

R	6.0000 -	9.5230
Md	6.8450	
Mn	7.0150	
S D	.84669	
S E	.23483	
C V	12.06	
P E	.57109	

Out 9.5230 (#5)

6.0000
6.3527
6.5796
6.6000
6.6270
6.7020
6.8450
7.0421
7.1300
7.2321
7.2524
7.3102

1.0 .0 1.0 .0 3.0 1.0 1.0 1.0 1.0 3.0

R	6.0000 -	7.3102
Md	6.7735	
Mn	6.8060	
S D	.40327	
S E	.11641	
C V	5.92	
P E	.27201	

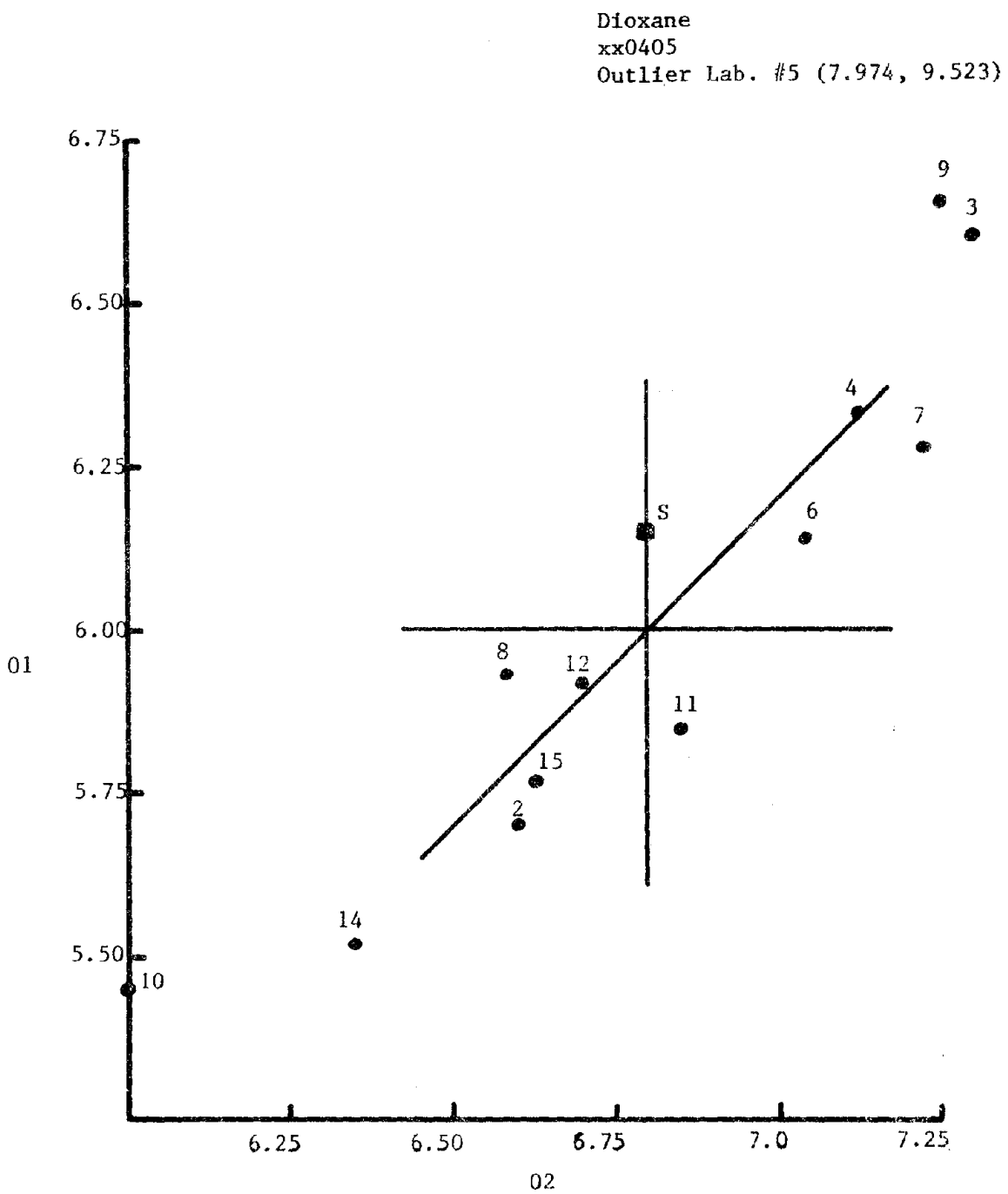

FIGURE 4-2 TWO-SAMPLE CHART OF REPLICATES ON FIRST DAY AT FIFTH LEVEL OF DIOXANE - PHASE I

In Tables 4-2 and 4-3, the column of numbers on the top of the page is a listing (in ascending order) of the replicates obtained from fourteen laboratories. Following this listing is an illustration of the frequency distribution for these numbers divided arbitrarily into ten groups. The numbers below this represent the following:

R:Range = x_{max} & x_{min}

Md:Median

Mn:Mean = $\dfrac{\sum_{i}^{n} x_i}{n} = \bar{x}$

SD = Standard Deviation = $\sqrt{\dfrac{\sum_{i}^{n} (x_i - \bar{x})^2}{n-1}}$

SE = Standard Error = $\dfrac{SD}{\sqrt{n}}$

CV = Coefficient of Variation (%) = $100 \times \dfrac{SD}{\bar{x}}$

PE = Probable Error = 0.6745 (SD)

The number which is an outlier is listed next and the number in parenthesis is the laboratory from which this outlier occurred. This process is repeated again with the outlier now removed and a new distribution and new set of statistics reported.

Figure 4-1 illustrates the two-sample chart for the replicates on the second day at the fourth level of benzene. The horizontal line represents the mean of one set of replicates and the vertical line the mean of the other set of replicates. The intersecting coordinates for the replicate pair from each laboratory are plotted and the true or "accepted" value for these replicates is represented by the darkened square.

If only random errors were the cause for the scatter in the data between laboratories, then the two determinations would have an equal probability of being both high, both low, first determination high and second low, or the second determination high and first low. Thus, the scatter should be a close cluster of points around the mean of the replications with a pattern which is, more or less, circular in nature. Due to the presence of non-random error components, the data tend to fall more into one set of quadrants than in the other. If we assume negligible errors in repeat determinations (good precision) among the individual laboratories and that the majority of the scatter (error) in the data is due to errors associated from one laboratory to another (systematic error), the results from repeat determinations in any one laboratory would tend to be then either both high or both low. Thus, the cluster of points would lie predominantly along the 45° line shown and the pattern appear elliptical in nature.

In the example shown for benzene, nine of the laboratories clustered fairly randomly around the mean. Laboratory #5 and Laboratory #15 were in the high-high and low-low quadrant respectively indicating the presence of systematic errors for this set of data. Laboratory #3 and Laboratory #1 were predominantly in the high-low quadrant showing that the precision for these two laboratories was not very good. The two-sample chart for dioxane in Figure 4-2 shows a very marked predominance of points strung out along the 45° line, obviously indicating a strong presence of systematic errors for this level of dioxane. In general, Laboratories #4, #5 and #10 tended to give higher results while Laboratories #7 and #15 had results which were lower than the random cluster around the mean shown by the other laboratories.

4.1.2 Analysis

As pointed out earlier, the set of data on two repeat determinations done on one day at one level comprised a "unit-block" suitable for the Youden analysis. The numbers reported by Scott for the same two determinations were considered to be the true or "accepted" value.

The Youden analysis obtains estimates of the replication error (precision) by analyzing the differences between repeat determinations made on the same day. Being a non-random component the systematic error will be the same for repeat determinations made on the same day within a laboratory. Thus by taking differences, the systematic error component drops out and the difference quantities can then be used to analyze the replication error:

$$S_r^2 = \frac{\sum_i^n (Di-\bar{D})^2}{2(n-1)} \qquad (4\text{-}1)$$

where: S_r^2 = Variance component of the replication error (precision

D_i = Difference quantity of repeat determinations in the ith laboratory.

$\bar{D}$ = Mean of the difference quantities.

n = Number of laboratories.

To obtain the standard deviation of the data itself we can use the results from either the first determination or the second determination; or a better method is to estimate this component from the sum of the two determinations:

$$S_d^2 = \frac{\sum_i^n (T_i-\bar{T})^2}{2(n-1)} \qquad (n-1) \text{ degrees of freedom} \qquad (4\text{-}2)$$

where: S_d^2 = Variance component of total error in data.

T_i = Sum of repeat determinations in the laboratory.

$\bar{T}$ = Mean of the sum quantities.

n = Number of laboratories.

The reason for using the totals in obtaining the standard deviation of the data is that the quantity S_d^2 can be used very conveniently in an F-ratio test for determining the presence of systematic error:

$$F_{n-1,n-1} = \frac{S_d^2}{S_r^2} \qquad (4\text{-}3)$$

Thus we are testing whether the estimates S_d^2 and S_r^2 differ significantly, i.e., whether they differ by more than can be reasonably explained on the grounds of errors in the estimates. The F-ratio must therefore reach a certain minimum value before we are willing to conclude that the ratio goes beyond what might happen solely from sampling. The minimum F-ratio values for various levels of significance at (n-1, n-1) degrees of freedom can be obtained from standard statistical tables.

The estimate of the variance component for systematic errors can now be obtained from the following equation:

$$S_d^2 = 2S_b^2 + S_r^2 \qquad (4\text{-}4)$$

where: S_d^2 = Variance component of total error in data.

S_b^2 = Variance component for systematic errors.

S_r^2 = Variance component for replication error (precision).

Another useful quantity obtained in this analysis is the coefficient of variation:

$$CV = 100 \frac{S_r}{\bar{x}} \ \% \qquad (4\text{-}5)$$

where: CV = Coefficient of variation (percent).

S_r = Standard deviation of the precision estimate.

$\bar{x}$ = Average amount present at the level in consideration.

The coefficient of variation is a useful quantity for ascertaining the relationship that exists between the replication error and the amount present. Thus at different levels of a compound, we can see how the precision estimate varies. This relationship then helps in determining the type of transformations that might be necessary to establish independence between the precision estimate and the amount present. If a transformation is able to make the mean and standard deviation of the transformed variate approximately independent, pooled estimates of the error components can then be obtained.

To test for the presence of any inherent systematic error in the method, we need to pass judgement on the difference between a reference value and the mean of the n values reported by the n laboratories. The reference value in our case is the value reported by Scott. Since we are dealing with pairs of data in our "unit-block" analysis, the sensitivity of the test increases if we combine the data from both members of a pair since they are both subject to the same systematic error. The test used here is the t-test which compares the difference between the mean and the reference value to the standard error of the mean:

$$t = \frac{(\bar{T}-R_T)\sqrt{n}}{S_T} \qquad (4\text{-}6)$$

where: t = Calculated t-test value.

$\bar{T}$ = Mean of the sum quantities.

R_T = Sum of the reference values.

S_T = Standard deviation of the sum quantities.

n = Number of laboratories.

Unless the value obtained for t exceeds some specified value, the observed difference between $\bar{T}$ and R_T is considered to be explainable because of the uncertainty in our estimate of the average amount found for that level by the n laboratories. Specified t values for various levels of significance are available from standard statistical tables.

Table B-4 in Appendix B summarizes the results of the analysis described above for each of the seven compounds and two mixtures. Column 1 of the table (Average Amount Found) is the mean of the results obtained by the n laboratories on the replicates of each day at each level. The second column (Average Amount Present) is the mean of the corresponding Scott values. The difference between the amount present and the amount found shows the systematic error of the procedure as estimated from the data available for each pair. This is shown in Column 3 as "Bias of Procedure". The fourth column (Precision) lists the variance component of the replication error, S_r^2, obtained from equation 4-1. Column 5 (Systematic Error) is the variance component for the systematic error, S_b^2, calculated from equations 4-2 and 4-4. Column 6 is the F-ratio test for significance of systematic errors, equation 4-3. The degrees of freedom (number of Laboratories minus one) is listed in column 7 and the coefficient of variation calculated from equation 4-5 is shown in column 8. Column 9 presents the calculated t-test values for systematic error in a method, equation 4-6.

By averaging the replicates analyzed on a day, the averages on two different days can be looked upon as another set of a "unit-block" and the analysis described above performed on this data. Table B-5 in Appendix B summarizes these results for each compound at each level.

Let us now assume, for the time being, that the estimates of the replication error (precision) obtained for each pair at each level in Table B-4 are homogeneous and can be pooled for that level. We will then obtain a table similar to Table B-5, but the quantities obtained for the precision and systematic error in these two tables would not be estimating the same variance components. The pooled systematic errors obtained from Table B-4 would now have to include the error due to analyzing on different days. But the systematic errors obtained from Table B-5 would be estimating the true between-laboratory error, since the day-to-day effect has been averaged out. Table 4-6 summarizes the relationships between the different estimates of variance components for results obtained by not averaging the replicates (Table B-4) and from results obtained by averaging the replicates (Table B-5).

TABLE 4-6 EXPECTED MEAN SQUARES FOR POOLED ESTIMATES

Analysis	Precision	Systematic Error
1. Replicates not averaged (Table A-4)	S_r^2	$S^2_{day} + S_b^2$
2. Replicates (Table A-5)	$\frac{S_r^2}{2} + S^2_{day}$	S_b^2

S_r^2 = Replication error

S_b^2 = Between laboratory error

S^2_{day} = Day-to-day error

If it can be established that homogenity exists in the precision estimates from level to level, pooled estimates of the variance components for each compound can be obtained by using the relationships described in Table 4-6.

The estimate of the day-to-day error, S^2_{day}, can be obtained either from the estimates in the first analysis or from those in the second analysis. If we had a completely balanced design for our analysis, with no missing cell values, these two estimates of S^2_{day} would be identical. Due to the fact that the Youden analysis can account for missing values and outliers, the two estimates of S^2_{day} do not necessarily come out to be exactly the same, but are nevertheless close enough to provide the estimate in which we are interested. For the purpose of this study, the estimates of the day-to-day error have been calculated from the estimates obtained in the first analysis.

From Tables B-4 and B-5 one sees a definite trend between the replication error, S_r^2, and the average amount found for a level. The precision values are seen to increase with the amount present for all seven compounds. Also, the coefficient of variation is approximately constant

for levels 2 through 5 indicating that S_r is proportional to the amount present at these levels. Level 1 on the other hand, deviates markedly from this trend except for ethylene dichloride and xylene where S_r is approximately proportional to the amount present at all five levels.

In Table B-4, the precision estimates are close enough to be averaged for each level. Similarly, the systematic error estimates can be averaged for each level. Using these averaged results and the results shown in Table B-5, one can obtain the various error components for each level, according to the relationships described in Table 4-6. However, if it can be shown that homogenity exists in the precision estimates from level to level, pooled estimates of the error components can be obtained for each compound. These error components would then describe the various errors associated in analyzing a compound for the range of concentration levels under which this study was conducted.

Before testing for any homogenity of variance, it is necessary to establish independence between the precision estimate, S_r, and the average amount found. To do this we need to perform a transformation on the data. In general, if a standard deviation, σ, is proportional to some function, $f(\mu)$, of the mean μ, the appropriate transformation is given by:

$$\int \frac{1}{f(\mu)}\, d\mu \qquad \text{(Ref. 5)}$$

In our case, $f(\mu) = \mu$ and hence a logarithmic transformation would establish the independence between S_r and the average amount found. Another advantage of a transformation which makes the standard deviation independent of the mean is that the transformed data become distributed more normally. Tables B-6 and B-7 correspond to Table B-4 and B-5 respectively. The only difference being a natural log-transformation of the data.

The test for homogenity of variance is taken from Reference 6 wherein the ratio of the largest to the smallest mean squares is compared against standard tables of these ratios. The upper 1% points table was considered for this study. The variation in the degrees of freedom from

one level to another was small enough so that an average value of about 12 could be used as an entry into the table. The upper 1% points of the ratio S^2_{max}/S^2_{min} for 12 degrees of freedom are shown in Table 4-7.

TABLE 4-7 UPPER 1% POINTS FOR THE RATIO $\frac{S^2_{max}}{S^2_{min}}$ AT 12 DEGREES OF FREEDOM

# of Mean Squares	Upper 1% Points
10	9.9
8	9.1
5	7.6
4	6.9
2	4.9

The test for homogenity of variance in the precision estimates was performed on the results shown in Tables B-6 and B-7. None of the compounds of Table B-6 showed any homogenity in the precision variance. In Table B-7 only chloroform, ethylene dichloride and xylene met the homogenity of variance criteria. The reason for the wide disparity in variance from level to level was mainly due to the variance in Level 1. Apparently, the results from Level 1 varied more from one replicate to another than the results from the other levels. By ignoring Level 1 and performing the test for homogenity on the remaining levels, almost all the compounds in both tables met the criteria. Only carbon tetrachloride and ethylene dichloride in Table B-6 did not meet the criteria stringently, but were close enough to warrant their inclusion for obtaining pooled estimates on all levels but the lowest concentration level.

The pooled estimates were obtained by taking the degrees-of-freedom - weighted average of the estimates:

$$S^2_{pooled} = \frac{\sum_i S_i^2 \, d_i}{\sum_i d_i} \qquad (4\text{-}7)$$

where: S^2_{pooled} = Pooled estimate of i variances.

S^2_i = Estimate of ith variance

d_i = Degrees of freedom for S_i

The S^2_{max}/S^2_{min} ratio and the pooled estimates from Tables B-6 and B-7 are shown in Tables 4-8 and 4-9 respectively. It should be remembered that the numbers reported in these tables are estimates obtained from log-transformed data, and represent relative-to-the-mean error estimates.

The individual error components: replication error, between laboratory error, and day-to-day error were then computed according to the relationships described in Table 4-6. Table 4-10 presents these error components as percent relative error. The percent relative error is computed from equation 4-8 as:

$$\% \text{ relative error} = \frac{s}{\bar{x}} \times 100 = \left(\sqrt{\exp(S^2) - 1}\right) \times 100 \qquad (4\text{-}8)$$

where: S = Estimate of an error component standard deviation.

$\bar{x}$ = Average level of concentration.

The total percent relative error is also presented in Table 4-10. and is computed as the root mean square value of the error components. This total error is the error associated with a single observation made in one laboratory on any one day. In general, the variance of a single observation made in one laboratory on any one day is given by:

$$S_r^2 + S^2_{days} + S_b^2$$

The variance of n observations on one day in one laboratory is given by:

$$\frac{S^2_r}{n} + S^2_{days} + S^2_b$$

TABLE 4-8 POOLED VARIANCE ESTIMATES ON LOG-TRANSFORMED DATA
FOR EACH PAIR OF REPLICATES AT EACH LEVEL
PHASE I

	All Levels Considered			Level 1 Excluded			Level 1		
	F_{max} Ratio	S_r^2 Precision	$S_{days}^2 + S_{labs}^2$ Systematic	F_{max} Ratio	S_r^2 Precision	$S_{days}^2 + S_{labs}^2$ Systematic	F_{max} Ratio	S_r^2 Precision	$S_{days}^2 + S_{labs}^2$ Systematic
Benzene	274.1	0.2919-01	0.2605-01	9.7	0.3947-02	0.7062-02	18.6	0.1243+00	0.9760-01
Carbon Tetrachloride	103.5	0.2889-01	0.9815+00	21.9	0.9385-02	0.1029-01	2.7	0.1016+00	0.4605+01
Chloroform	25.6	0.6480-02	0.1857-01	6.8	0.3325-02	0.8064-02	3.8	0.1690-01	0.5327-01
Dioxane	145.5	0.5409-02	0.9649+00	15.6	0.1512-02	0.4874-02	10.3	0.2018-01	0.4605+01
Ethylene Dichloride	19.6	0.3150-02	0.3816-02	19.6	0.3210-02	0.3206-02	1.8	0.2885-02	0.6505-02
Trichloro-Ethylene	1219.8	0.4315+00	0.4100+00	4.2	0.3794-02	0.2645-02	1.2	0.2060+01	0.1961+01
Xylene	15.5	0.3327-02	0.1541-01	6.5	0.2277-02	0.2843-02	3.1	0.7445-02	0.6470-01
Benzene (Mixture)	1.9	0.1053-02	0.2082-02						
Xylene (Mixture)	1.3	0.8785-03	0.3590-02						
EDC (Mixture)	2.3	0.3555-02	0.6815-02						
TCE (Mixture)	3.2	0.1821-02	0.1158-01						

TABLE 4-9 POOLED VARIANCE ESTIMATES ON LOG-TRANSFORMED DATA FOR EACH LEVEL (REPLICATE AVERAGED)

PHASE I

	All Levels Considered			Level 1 Excluded			Level 1	
	F_{max} Ratio	$\frac{S_r^2}{2} + S_{days}^2$ Precision	S_{labs}^2 Systematic	F_{max} Ratio	$\frac{S_r^2}{2} + S_{days}^2$ Precision	S_{labs}^2 Systematic	$\frac{S_r^2}{2} + S_{days}^2$ Precision	S_{labs}^2 Systematic
Benzene	89.6	0.3176-01	0.9263-02	6.2	0.3993-02	0.3374-02	0.1300+00	0.3010-01
Carbon Tetrachloride	94.0	0.6895-01	0.9849+00	4.5	0.7120-02	0.6968-02	0.2830+00	0.4370+01
Chloroform	7.1	0.1515-01	0.1511-02	2.0	0.8698-02	0.5953-03	0.3900-01	0.4900-02
Dioxane	32.1	0.6450-02	0.1004+01	2.6	0.1608-02	0.3387-02	0.2380-01	0.4590+01
Ethylene Dichloride	3.6	0.2663-02	0.2085-02	3.6	0.2499-02	0.2009-02	0.3400-02	0.2430-02
Trichloro-Ethylene	1552.2	0.7767+00	-0.1238+00	2.5	0.4110-02	0.2137-03	0.3570+01	-0.5720+00
Xylene	5.8	0.4445-02	0.1290-01	1.9	0.2662-02	0.1377-02	0.1130-01	0.5720-01
Benzene (Mixture)		0.1330-02	0.4770-03					
Xylene (Mixture)		0.4940-03	0.3530-02					
EDC (Mixture)		0.3840-02	0.2560-02					
TCE (Mixture)		0.1060-01	0.1870-02					

TABLE 4-10 SUMMARY OF PHASE I ANALYSIS

(Percent Relative Error Contribution of Variance Component)

SINGLE COMPONENTS

	Excluding Level 1				Level 1			
	Replication	Between Labs.	Day-to -Day	Total	Replication	Between Labs.	Day-to -Day	Total
Benzene	6.29	5.81	6.08	10.50	36.38	17.48	26.43	49.84
Carbon Tetrachloride	9.71	8.36	5.77	14.05	32.70	833.42	51.47	1047.26
Chloroform	5.77	2.44	8.66	10.69	13.06	7.01	22.26	26.96
Dioxane	3.89	5.82	3.86	8.00	14.28	987.39	12.29	1005.09
Ethylene Dichloride	5.67	4.48	3.46	8.01	5.38	4.93	6.39	9.71
Trichloroethylene	6.16	1.46	4.93	8.02	261.65	*	340.46	739.98
Xylene	4.77	3.71	3.83	7.15	8.64	24.26	8.68	27.35

MIXTURES

	Replication	Between Labs	Day-to -Day	Total
Benzene	3.25	2.18	4.01	5.60
Xylene	2.96	5.95	0.77	6.69
Ethylene Dichloride	5.97	5.06	6.53	10.21
Trichloroethylene	4.27	4.33	9.88	11.62

* Negative pooled estimate

The variance of n observations made on each of k days in one laboratory is given by:

$$\frac{s_r^2}{nk} + \frac{s^2_{days}}{k} + s^2_b$$

The variance of n observations made on eack of k days in each of 1 laboratories is given by:

$$\frac{s^2_r}{nkl} + \frac{s^2_{days}}{kl} + \frac{s^2_b}{l}$$

Thus, for example, in Table 4-10 the total percent relative error for benzene in the approximate concentration range of 0.20 mg. to 1.70 mg. (Level 1 excluded) is 10.5%. This means that if we were to analyze one sample of benzene in any one laboratory by the method used in this study, and the concentration was determined to be 0.600 mg., the one-sigma limits on our determination would then be:

$$0.600 \pm \frac{10.5}{100} \times 0.6 = 0.600 \pm 0.063 \text{ mg.}$$

4.2 DATA ANALYSIS OF ANALYTICAL RESULTS FROM PHASE II

The statistical analysis on the data obtained in Phase II was identical to the analysis performed on the data in Phase I. Outliers were identified and checked; a Youden analysis was performed on the pair of results obtained on each day, as well as on the average of the replicates obtained on different days; the data was subjected to a natural log transformation and the analysis repeated; pooled estimates of the error variances obtained for all levels, Level 1 excluded, and for Level 1; finally the percent relative error components estimated for the seven compounds and two mixtures. In addition, the results obtained in Phase I were compared with the Phase I samples analyzed in Phase II.

4.2.1 Identification of Phase II Outliers

The outliers were identified by the procedure described in Section 4.1.1 and, where necessary, the corresponding pairs excluded from the Youden analysis. Table 4-1 lists the number of data pairs obtained as outliers for each laboratory in Phase II. It is interesting to note that, except for Laboratory #7, each laboratory had almost the same number of outliers in Phase II as in Phase I. The raw data for the seven compounds and the two mixtures analyzed in Phase II are presented in Table C-3 in Appendix C. The asterisks indicate the missing values and outliers.

Although a very large number of outliers were obtained again from Laboratory #13, it was decided not to exclude this laboratory from the analysis. Thus, all results which were not rejected as outliers from Laboratory #13 were included in the Youden analysis of Phase II.

4.2.2 Analysis

The analysis of Phase II results was done according to the method outlined in Section 4.1.2. Table C-4 summarizes the Youden analysis performed with the replicates not averaged and Table C-5 summarizes the results obtained by averaging the replicates on each day. The corresponding analyses on the log-transformed data are presented in Table C-6 and C-7. The pooled estimates obtained from these two tables are shown in Tables 4-11 and 4-12 respectively.

Except for benzene and carbon tetrachloride, the other five compounds and mixtures seemed to exhibit homogenity at all levels, including Level 1. For the sake of comparison with the results from Phase I, however, the Level 1 pooled estimates have been separated from the other results. The percent relative errors obtained from these pooled estimates are shown in Table 4-13. A comparison of these errors with the percent errors obtained in Phase I points out the following:

a. The individual error components and the total percent relative error were generally greater by almost fifty percent or more in Phase II than in Phase I.

TABLE 4-11 POOLED VARIANCE ESTIMATES ON LOG-TRANSFORMED DATA
FOR EACH PAIR OF REPLICATES AT EACH LEVEL
PHASE II

	All Levels Considered			Level 1 Excluded			Level 1		
	F_{max} Ratio	S_r^2 Precision	$S_{days}^2 + S_{labs}^2$ Systematic	F_{max} Ratio	S_r^2 Precision	$S_{days}^2 + S_{labs}^2$ Systematic	F_{max} Ratio	S_r^2 Precision	$S_{days}^2 + S_{labs}^2$ Systematic
Benzene	563.4	0.9404-01	0.1779+00	21.29	0.8923-02	0.8733-02	112.6	0.4841+00	0.9532+00
Carbon Tetrachloride	879.0	0.1408+00	0.5320+00	23.22	0.8116-02	0.1372-01	35.41	0.7379+00	0.2864+01
Chloroform	28.54	0.1936-01	0.7371-02	5.84	0.8789-02	0.7365-02	6.28	0.5238-01	0.7391-02
Dioxane	9.40	0.5845-02	0.1955-01	6.81	0.5176-02	0.1923-01	4.24	0.8870-02	0.2102-01
Ethylene Dichloride	12.16	0.4267-02	0.1217-01	12.16	0.4620-02	0.9006-02	1.14	0.2869-02	0.2469-01
Trichloro-Ethylene	12.80	0.8798-02	0.9934-02	5.20	0.5263-02	0.6908-02	1.38	0.2438-01	0.2327-01
Xylene	13.32	0.3370-02	0.7058-02	13.32	0.3388-02	0.5681-02	1.32	0.3298-02	0.1279-01
Benzene (Mixture)	4.41	0.2557-02	0.1050-01						
Xylene (Mixture)	3.88	0.3255-02	0.9554-02						
EDC (Mixture)	2.89	0.7301-02	0.2173-01						
TCE (Mixture)	4.71	0.1201-01	0.1032-01						

TABLE 4-12 POOLED VARIANCE ESTIMATES ON LOG-TRANSFORMED DATA FOR EACH LEVEL (REPLICATES AVERAGED)

PHASE II

	All Levels Considered			Level 1 Excluded			Level 1	
	F_{max} Ratio	$\frac{S_r^2}{2} + S_{days}^2$ Precision	S_{labs}^2 Systematic	F_{max} Ratio	$\frac{S_r^2}{2} + S_{days}^2$ Precision	S_{labs}^2 Systematic	$\frac{S_r^2}{2} + S_{days}^2$ Precision	S_{labs}^2 Systematic
Benzene	282.90	0.2177+00	-0.2147-01	5.45	0.1345-01	-0.3517-03	0.1220+01	-0.1250+00
Carbon Tetrachloride	98.65	0.6118-01	0.5969+00	2.32	0.5798-02	0.7034-02	0.3050+00	0.3190+01
Chloroform	21.63	0.1566-01	0.1948-02	3.59	0.5049-02	0.6755-02	0.4490-01	-0.1130-01
Dioxane	5.40	0.1119-01	0.1064-01	5.40	0.1213-01	0.9398-02	0.6960-02	0.1630-01
Ethylene Dichloride	2.51	0.5260-02	0.8191-02	2.37	0.4631-02	0.6109-02	0.7780-02	0.1650-01
Trichloro-Ethylene	3.97	0.7598-02	0.6515-02	1.54	0.5338-02	0.3060-02	0.1730-01	0.2140-01
Xylene	2.39	0.4179-02	0.3822-02	2.39	0.3931-02	0.3426-02	0.5240-02	0.5510-02
Benzene (Mixture)		0.5230-02	0.6090-02					
Xylene (Mixture)		0.6350-02	0.4650-02					
EDC (Mixture)		0.6270-02	0.1380-01					
TCE (Mixture)		0.7190-02	0.9140-02					

TABLE 4-13 SUMMARY OF PHASE II ANALYSIS

(Percent Relative Error Contribution of Variance Components)

SINGLE COMPONENTS

	Excluding Level 1				Level 1			
	Replication	Between Labs.	Day-to -Day	Total	Replication	Between Labs.	Day-to -Day	Total
Benzene	9.47	0.00(1)	9.50	13.35	78.9	(2)	126.3	179.1
Carbon Tetrachloride	9.03	8.40	8.19	14.86	104.5	482.6	(2)	597.2
Chloroform	9.40	8.23	2.47	12.76	23.2	(2)	13.74	24.82
Dioxane	7.20	9.72	9.94	15.72	9.44	12.82	6.88	17.42
Ethylene Dichloride	6.80	7.83	5.39	11.71	5.36	12.90	9.07	16.72
Trichloroethylene	7.26	5.54	6.21	11.07	15.71	14.71	4.33	22.09
Xylene	5.83	5.86	4.75	9.54	5.75	7.43	8.55	12.74

MIXTURES

	Replication	Between Labs.	Day-to -Day	Total
Benzene	5.06	7.82	6.65	11.46
Xylene	5.71	6.83	7.01	11.35
Ethylene Dichloride	8.56	11.79	8.92	17.16
Trichloroethylene	10.99	9.58	3.44	15.03

(1) Very small negative estimate (-0.3517-03); assumed zero.

(2) Negative estimates.

b. The analyses at Level 1 in Phase II, especially for dioxane and trichloroethylene, appeared to have improved over the analyses at Level 1 in Phase I.

c. Although there was better homogenity in variances from one level to another in Phase II, the magnitude of the variances in Phase II were greater than in Phase I.

One obvious reason for the larger variances associated with the analysis of Phase II samples is the significant effect of errors associated in sampling the Phase II charcoal tubes. While in Phase I the sample sets were prepared to be identical, in Phase II fifteen different individuals were involved in calibrating and sampling through fifteen pumps. If one makes the inherent, but reasonable, assumption that the sampling error of Phase I was negligible compared to the sampling error of Phase II and the analysis errors in Phases I and II were identical, then an estimate of the sampling error of the method can be obtained from equation 4-9:

$$E^2_{PII} - E^2_{PI} = E^2_{sampling} \qquad (4\text{-}9)$$

where: E^2_{PII} = Total percent relative variance of Phase II (analytical error + sampling error)

E^2_{PI} = Total percent relative variance of Phase I (analytical error)

$E^2_{sampling}$ = Percent relative variance of sampling. (sampling error)

The first assumption cannot be checked by statistical methods. We can find no evidence in the data to indicate that it is not true. The second assumption can be checked by the data for the Phase I samples analyzed in Phase II to similar samples analyzed in Phase I.

Spare samples from the low, middle and high concentration levels sampled in Phase I were analyzed during Phase II. The amount of organic vapor collected in the Phase I spare samples (Code 00) was similar to the Code 02 replicates in Phase I. These two sets of data represent almost identical samples sampled by identical methods under identical conditions, but analyzed under different circumstances. By comparing the Phase I spare samples analyzed in Phase II with the corresponding Phase I 02

replicates, one can determine if there was any significant improvement in the analytical method of the two phases. Table 4-14 presents the two sets of data from the two phases. The rings identify the outliers obtained from each set of data. The corresponding Scott values, the laboratory means and the standard deviations are also listed. The F-ratio listed for each level is the ratio of the larger variance to the smaller variance. Standard F-ratio value at a 5% significance level is approximately 2.70.

A study of the F-ratio column shows that the only significant improvement in the analysis of Phase II samples was at the 5% TLV level (Level 1). This is especially noticeable in the case of dioxane and trichloroethylene. No significant improvement in the analytical method is seen for the other levels or in the analysis of the mixtures. Thus, one can reasonably infer that except for the 5% TLV level, the analytical error in the two phases was essentially the same. Thus, Equation 4-9 can be applied to estimate the magnitude of the sampling error.

Table 4-15 summarizes the percent relative error estimates of sampling and analyzing the single components and mixtures used in this study. The results shown exclude the 5% TLV level (Level 1). The analytical error estimates for the single components are fairly close and average around 9.5%. The sampling error estimates are also closely grouped, except for carbon tetrachloride (low end of the distribution) and dioxane (high side), and average about 8.3%.

Analytical and sampling error estimates for the benzene/xylene mixture are somewhat lower in comparison to the estimates for the ethylene dichloride/trichloroethylene mixture; but for both the single components and the mixtures, the sampling and analytical error estimates are of the same order of magnitude and lie in the range of approximately eight to ten percent.

TABLE 4-14 COMPARISON OF PHASE I SAMPLES (02) WITH PHASE I SAMPLES ANALYZED IN PHASE II (00)

Level		Lab 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Scott	Lab. Mean	Lab. Stnd. Dev.	F-Ratio
BENZENE																				
1	Phase II(00)	0.0190	0.0200	0.0160	0.0000	0.0130	0.0121	0.0137	0.0141	0.0148	0.0480	0.0147	0.0146	1.9028	0.0142	0.0125	0.0169	0.0148	0.0024	8.03
	Phase I (02)	0.0009	0.0310	0.0145	0.0250	0.0140	0.0135	0.0156	0.0195	0.0177	0.0180	0.0136	0.0140	0.1690	0.0121	0.0122	0.0170	0.0158	0.0068	
3	Phase II(00)	0.3400	0.3600	0.3430	0.3380	0.3140	0.3264	0.3707	0.3300	0.3298	0.4200	0.3357	0.3251	0.3303	0.3256	0.3362	0.3224	0.3360	0.0146	4.57
	Phase I (02)	0.3300	0.3450	0.3673	0.3540	0.3150	0.2918	0.3313	0.3046	0.3470	0.2730	0.3180	0.3335	0.3480	0.3189	0.2529	0.3255	0.3220	0.0312	
5	Phase II(00)	1.1000	1.8600	1.4636	1.6200	1.4510	1.5487	1.7045	1.4260	1.5838	1.2000	1.6027	1.4680	1.0125	1.5258	1.5537	1.5156	1.4746	0.2232	1.61
	Phase I (02)	1.7000	1.7290	1.5672	1.8790	1.9850	1.4227	1.6195	1.6909	1.6122	1.2750	1.5320	1.5690	1.5890	1.5451	1.4375	1.5422	1.6102	0.1759	
CARBON TETRACHLORIDE																				
1	Phase II(00)	20.5680	0.0900	0.0350	0.0000	0.0430	0.0372	0.0417	0.0366	0.0376	1.1200	0.0357	0.0387	0.0000	0.0335	0.0184	0.0266	0.0297	0.0152	3.39
	Phase I (02)	0.0000	0.0400	0.0336	0.0000	0.0520	0.0181	0.0742	0.0381	0.0259	0.0857	0.0450	0.0401	0.1990	0.0280	0.0918	0.0269	0.0408	0.0280	
3	Phase II(00)	0.3300	0.6500	0.9360	0.9900	0.8290	0.7453	0.3427	0.7860	0.7287	0.6770	0.7319	0.7564	0.6180	0.7501	0.6202	0.6741	0.6994	0.1801	1.99
	Phase I (02)	0.9600	0.7400	0.7589	0.4320	0.7300	0.7229	0.8333	0.7487	0.7228	0.7700	0.7700	0.8333	0.7370	0.7616	0.4863	0.6750	0.7337	0.1276	
5	Phase II(00)	1.3670	16.0000	13.1340	13.3700	14.3340	19.5544	15.1312	16.3500	12.7776	26.8000	15.4620	14.8400	35.5106	15.2973	17.2349	15.0937	15.2904	1.8929	1.06
	Phase I (02)	15.5250	15.2200	13.6952	11.6920	15.4510	19.9618	0.6722	15.0789	14.1302	2.7810	13.5940	15.9100	16.8230	16.1085	14.6560	15.2222	15.2188	1.9452	
CHLOROFORM																				
2	Phase II(00)	1.5090	1.7700	1.5300	1.4800	1.6980	1.4161	1.5210	1.7120	0.9860	1.4250	1.4860	1.5490	1.5996	1.4934	1.3098	1.5073	1.5356	0.1248	1.28
	Phase I (02)	1.1230	1.6160	1.5905	1.6100	1.3410	1.3708	1.7014	1.4993	1.4748	1.5980	1.3870	1.4960	2.2830	1.4336	1.4402	1.5142	1.5040	0.1102	
4	Phase II(00)	3.7000	4.8900	4.6420	5.0300	4.1940	4.9007	5.2064	5.0300	4.4966	4.5000	4.3260	4.3550	3.5008	4.0448	4.2718	4.6566	4.4725	0.4946	3.11
	Phase I (02)	4.9000	4.7300	4.5816	4.8700	4.4500	4.5256	5.2467	5.1644	4.6528	4.6000	4.5370	4.5490	4.2520	4.6665	4.2988	4.7028	4.6682	0.2804	
DIOXANE																				
1	Phase II(00)	0.2000	0.2100	0.2300	0.7470	0.2110	0.2046	0.1855	0.2060	0.2210	0.2180	0.2187	0.2030	0.2705	0.0014	0.0028	0.2209	0.2089	0.0124	77.5
	Phase I (02)	2.0000	0.2100	0.2371	0.4300	0.1850	0.2031	0.0000	0.2004	0.2485	2.8310	0.1840	0.2120	0.4540	0.1898	0.1743	0.2217	0.2247	0.1090	
3	Phase II(00)	4.2240	3.7500	3.9620	3.5000	4.4960	3.8293	3.6191	3.5400	3.8832	3.5110	3.7800	3.4710	2.9132	3.4891	3.9480	3.7466	3.7277	0.3693	1.62
	Phase I (02)	16.0000	3.6400	4.3526	4.0200	4.5710	3.6392	3.6469	3.8327	4.0287	4.9830	3.4280	3.4220	4.5690	3.8217	3.8510	3.7733	3.9861	0.4696	
5	Phase II(00)	6.2500	6.3100	6.9200	6.9900	6.9180	7.1983	9.6871	6.2800	6.9640	7.4750	6.5140	6.4470	5.1047	6.1268	7.6303	6.7233	6.6520	0.6466	2.28
	Phase I (02)	7.4700	6.6000	7.3102	7.1300	9.5230	7.0421	7.2321	6.5796	7.2524	6.0000	6.8450	6.7020	4.4720	6.3527	7.6270	6.8023	6.8571	0.4278	
ETHYLENE DICHLORIDE																				
1	Phase II(00)	0.0710	0.1400	0.1760	0.6000	0.1200	0.0884	0.0835	0.0980	0.1062	0.0980	0.1139	0.1102	0.0906	0.1007	0.1418	0.0938	0.1098	0.0275	14.19
	Phase I (02)	0.1100	0.1000	0.1006	0.2070	0.1030	0.0960	0.0959	0.1181	0.2594	0.1070	0.0938	0.1055	0.5930	0.0932	0.1005	0.0940	0.1019	0.0073	
3	Phase II(00)	2.6000	2.0800	2.0620	2.0900	2.2330	2.0791	2.0394	2.2100	2.0897	2.0660	2.1070	2.2780	1.4997	1.9592	2.3248	2.0788	2.1244	0.1046	1.02
	Phase I (02)	3.5000	2.0900	1.8863	2.0490	2.1320	2.1359	2.0827	2.0214	2.1201	1.9250	1.9350	2.0730	3.1370	1.9809	1.8100	2.0908	2.0185	0.1034	
5	Phase II(00)	6.6350	6.4100	7.2940	8.6000	6.5700	7.0373	7.8872	6.8600	5.8711	5.8880	6.6990	7.3920	5.6975	6.1896	8.8598	8.3542	6.9260	0.9489	9.76
	Phase I (02)	8.7000	7.3600	7.4949	9.5700	7.8580	7.8224	6.9884	7.3163	7.4315	6.9730	7.3340	7.9220	14.6030	7.4298	7.3767	8.2563	7.4422	0.3038	
TRICHLORETHYLENE																				
1	Phase II(00)	0.1630	0.2300	0.1970	1.1700	0.1510	0.1613	0.1946	0.1890	0.1791	0.1700	0.2042	0.1832	0.4630	0.1807	0.1820	0.1749	0.1834	0.0206	18.17
	Phase I (02)	0.1900	0.1900	0.3497	0.3970	0.1730	0.1675	0.1801	0.3240	0.1814	0.1800	0.1950	0.1843	0.0360	0.1780	0.1660	0.1765	0.2061	0.0878	
3	Phase II(00)	4.6000	5.8400	5.2600	6.0100	5.0060	5.6124	5.2977	6.0200	5.8135	4.4000	5.7310	5.6750	5.5645	5.4953	6.1983	5.6718	5.5015	0.5138	5.00
	Phase I (02)	5.9000	5.6200	6.0073	5.8800	5.8910	6.0433	6.0517	5.5234	6.0345	5.8000	5.4450	5.6950	4.9110	6.2271	3.5900	5.6863	5.8552	0.2298	
5	Phase II(00)	16.0000	16.0000	11.5420	15.1300	14.8910	13.4579	19.3876	16.2400	12.3557	8.2000	15.4150	14.2800	1.0492	14.4129	17.1446	14.3925	15.0966	2.0343	1.57
	Phase I (02)	15.6000	15.8000	17.7544	11.8690	15.2030	14.9073	14.9176	16.3882	14.6304	12.5000	14.7140	15.9800	14.0910	14.5616	17.8737	14.5072	15.1193	1.6227	
XYLENE																				
1	Phase II(00)	0.2160	0.2200	0.0710	0.1900	0.1550	0.1957	0.2026	0.2080	0.1816	0.2080	0.2036	0.1988	0.0000	0.2066	0.1659	0.2001	0.1962	0.0190	4.86
	Phase I (02)	0.2214	0.2100	0.1411	0.2700	0.0980	0.2006	0.2110	0.2025	0.2181	0.2240	0.1760	0.1942	0.1340	0.1858	0.1908	0.2016	0.1918	0.0419	
3	Phase II(00)	4.3470	4.6500	5.3900	4.9800	4.3750	4.8184	5.0650	4.4500	4.3334	0.9980	4.6970	4.462	5.3721	4.3760	4.8124	4.6063	4.7234	0.3669	1.36
	Phase I (02)	5.1000	4.3800	4.1067	4.3100	4.8080	4.6583	4.2747	3.8174	4.4262	5.8110	4.4250	4.6140	4.6640	4.2272	4.4466	4.6392	4.4470	0.3151	
5	Phase II(00)	8.2000	8.3600	8.5220	8.7100	8.0190	8.3760	7.4087	7.7000	12.1858	6.9080	8.4980	7.9700	6.9338	7.8304	9.0129	7.9981	8.0320	0.6292	2.11
	Phase I (02)	8.9000	8.1400	7.7002	8.3100	8.6050	8.4025	8.0232	9.2343	8.6337	8.2240	7.9490	8.0960	9.0560	8.0672	8.2428	8.0552	8.3722	0.4335	

TABLE 4-14 COMPARISON OF PHASE I SAMPLES (02) WITH PHASE I SAMPLES ANALYZED IN PHASE II (00)

(continued)

Level		Lab 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Scott	Lab Mean	Lab. Stad. Dev.	F-Ratio*
BENZENE (mixture)																				
1	Phase II(00)	(0.1500)	0.3620	0.3520	0.3440	0.2490	0.3162	0.4107	0.3180	0.3338	0.2600	0.3510	0.3398	0.4008	*	0.3286	0.3319	0.3358	0.0458	6.13
	Phase I (02)	0.3500	0.3470	0.3286	0.3300	0.3310	0.3160	0.3091	0.3147	0.3319	0.3190	0.3600	0.3338	0.2860	0.3266	(0.2302)	0.3347	0.3274	0.0185	
XYLENE (mixture)																				
1	Phase II(00)	3.4000	4.2700	4.5430	4.3900	3.8840	3.9805	4.9792	4.1800	4.0708	3.7280	4.0776	4.2990	2.9460	*	3.9626	4.2624	4.0507	0.4911	2.02
	Phase I (02)	4.4546	3.7700	3.8329	4.0300	4.0970	3.9338	3.8482	4.0236	4.1667	5.0160	4.0780	3.9090	4.4930	3.8992	3.6619	4.2988	4.0809	0.3453	
ETHYLENE DICHLORIDE (mixture)																				
1	Phase II(00)	2.5000	1.9900	2.0500	(20.7000)	2.0740	1.9412	1.8567	1.9260	1.8166	1.6530	2.0660	2.0030	1.6507	1.7259	2.1637	2.1092	1.9583	0.2228	1.36
	Phase I (02)	2.0000	2.1000	1.4587	2.7200	1.9110	2.1093	1.9805	2.1248	1.9724	2.0840	2.0720	2.1940	(4.1350)	1.9714	1.9852	2.1258	2.0488	0.2595	
TRICHLORETHYLENE (mixture)																				
1	Phase II(00)	5.4000	5.6500	6.0210	4.7700	5.8630	4.9422	5.6278	5.8100	5.1697	4.2000	5.3950	5.8170	4.5327	4.8569	5.7421	5.3834	5.3198	0.5493	1.44
	Phase I (02)	5.5000	4.7300	5.3690	4.3500	4.9980	4.9843	5.0427	5.0710	5.1868	4.2500	5.0800	5.1850	6.1580	5.0877	4.6874	5.4257	5.0453	0.4584	

* Sample Lost

TABLE 4-15 SUMMARY OF SAMPLING AND ANALYTICAL ERRORS
(PERCENT RELATIVE ERROR)

Single Components	Analytical + Sampling Error (Phase II)	Analytical Error (Phase I)	Sampling Error
Benzene	13.35	10.50	8.24
Carbon Tetrachloride	14.86	14.05	4.84
Chloroform	12.76	10.69	6.97
Dioxane	15.72	8.00	13.53
Ethylene Dichloride	11.71	8.01	8.54
Trichloroethylene	11.07	8.02	7.63
Xylene	9.54	7.15	6.32
Mixtures			
Benzene	11.46	5.60	10.00
Xylene	11.35	6.69	9.17
Ethylene Dichloride	17.16	10.21	13.79
Trichloroethylene	15.03	11.62	9.53

5.0 DISCUSSION OF RESULTS

The statistical analysis of the collaborative test data for the seven test compounds present singly at concentrations from 60% of the TLV to the excursion limit yielded an estimated relative total error of 12.7%. The analytical error was estimated at 9.5% and the sampling error at 8.3%. In Phase II, errors at the 5% of TLV concentration level were up to twice as large as for the other levels for the five compounds present at weights above 0.1 mg. and substantially greater for the two compounds present at less than 0.1 mg.

The % relative errors were calculated after removal of outliers. This is the statistically accepted approach to treating collaborative test data. However, from a practical standpoint we must consider the reasons for the relatively large number of outliers obtained. In Phase II, five of the fifteen collaborators reported data in which 10 or more of the 72 test pairs contained outliers. On the other hand, eight laboratories had either zero or one outlier.

This section investigates the various sources of error in the generation of the collaborative test data. Possible limitations of the method and means for improving the results are also discussed.

5.1 ANALYSIS OF BACK-UP CHARCOAL SECTIONS

A selected group of back-up sections of charcoal was analyzed in both Phases I and II. In Phase I a total of 36 back-up sections were analyzed by each laboratory. The 36 samples included one sample from each set of four replicates. In Phase II the analysis of back-ups involved two samples of each compound or mixture at the highest concentration only. The schedule change from Phase I to Phase II was made because in most cases nothing was detected on the back-ups from the lower levels.

The results for the analyses of the back-up sections in Phase I and II are tabulated in Tables 5-1 and 5-2, respectively. The mean reported by all laboratories for the front sections (less outliers) is also shown. The amount found on the back-up sections was very small in most cases. The median value for the back-up exceeded one percent of the mean of values reported for the front section only for the excursion limit equivalent of ethylene dichloride. The average amount of ethylene

TABLE 5-1 WEIGHT OF ORGANICS FOUND ON BACK-UP CHARCOAL SECTION IN PHASE I, MG.

Compound	Level	Mean Front End	Laboratory 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Benzene	1	0.015	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.008	<0.001	0.000	0.000	0.000	0.000	0.000
	2	0.167	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	<0.001	0.000	0.000	0.000	0.000	0.000
	3	0.290	0.001	0.005	0.000	0.000	0.000	0.000	0.000	0.000	0.000	<0.001	0.000	0.000	0.000	0.000	0.000
	4	0.558	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.001	0.001	0.000	0.000	0.000	0.000	0.000
	5	1.738	0.000	0.000	0.000	0.000	0.000	0.002	0.000	0.000	0.003	0.004	0.000	0.000	0.000	0.000	0.000
Carbon Tetrachloride	1	0.049	0.000	-	0.001	0.000	0.000	0.000	0.000	0.000	0.000	<0.001	0.000	0.000	0.000	0.000	0.015
	2	0.474	0.000	0.000	0.000	0.000	0.000	0.002	0.009	0.000	0.000	0.040	0.000	0.003	0.000	0.000	0.000
	3	0.843	0.000	-	0.003	0.000	0.000	0.005	0.018	0.000	0.058	0.110	0.000	0.001	0.358	0.000	0.069
	4	1.341	0.000	0.000	0.000	0.000	0.000	0.004	0.012	0.000	0.000	0.019	0.000	0.003	0.000	0.000	0.000
	5	13.687	0.000	0.050	0.070	0.531	0.138	0.043	0.169	0.293	0.262	0.022	0.078	0.077	0.000	0.095	0.181
Chloroform	1	0.140	0.000	0.000	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.003	0.000	0.000	0.000	0.000	0.000
	2	1.427	0.000	0.000	0.000	0.000	0.000	0.002	0.000	0.000	0.000	0.003	0.000	0.000	0.000	0.000	0.000
	3	2.246	0.000	0.000	0.000	0.000	0.000	0.002	0.000	0.000	0.000	0.002	0.000	0.000	0.000	0.000	0.000
	4	5.042	0.000	0.000	0.000	0.000	0.000	0.381	0.000	0.017	0.191	0.006	0.010	0.104	0.089	0.014	0.131
Dioxane	1	0.233	0.000	0.060	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.005	0.000	0.000	0.000	0.000	0.000
	2	1.844	0.000	0.010	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.008	0.000	0.001	0.000	0.000	0.003
	3	3.328	0.000	0.050	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.002	0.000	0.003	0.000	0.000	0.000
	4	5.074	0.000	0.080	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.024	0.000	0.000	0.000	0.000	0.000
	5	6.012	0.000	0.030	0.063	0.000	0.000	0.012	0.000	0.000	0.108	0.153	0.007	0.004	0.271	0.005	0.002
Ethylene Dichloride	1	0.117	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.003	0.000	0.000	0.000	0.000	0.000
	2	1.648	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.088	0.002	0.000	0.000	0.000	0.000	0.000
	3	2.241	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.003	0.000	0.000	0.000	0.000	0.000
	4	4.061	0.000	0.030	0.105	0.000	0.119	0.063	0.000	0.000	0.131	0.011	0.018	0.000	0.502	0.000	0.005
	5	8.371	0.590	0.500	0.640	0.485	0.635	0.297	0.336	0.825	1.182	0.556	0.519	0.819	1.804	0.045	0.197
Trichloroethylene	1	0.173	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	<0.001	0.000	0.000	0.000	0.000	0.000
	2	3.698	0.000	0.000	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.002	0.000	0.000	0.000	0.000	0.000
	3	5.110	0.000	0.000	0.156	0.000	0.000	0.005	0.000	0.000	0.002	0.007	0.000	0.000	0.000	0.000	0.000
	4	9.271	0.000	0.000	0.261	0.278	0.000	0.005	0.000	0.000	0.035	0.001	0.000	0.000	0.000	0.020	0.000
	5	14.049	0.329	0.040	0.039	-	0.496	0.260	0.214	0.000	0.300	0.044	0.055	0.167	0.003	0.099	0.228
Xylene	1	0.175	0.006	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
	2	2.128	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.001	0.000	0.002	0.299	0.000	0.003
	3	4.035	0.016	0.020	0.000	0.000	0.000	0.001	0.000	0.000	0.000	0.000	0.003	0.000	0.109	0.000	0.000
	4	5.963	0.010	0.000	0.000	0.000	0.000	0.002	0.000	0.000	0.000	0.003	0.000	0.000	0.000	0.000	0.002
	5	7.392	0.004	0.000	0.000	0.000	0.000	0.002	0.000	0.000	0.000	0.004	0.006	0.002	0.197	0.000	0.007
Benzene (Mixture)	3	0.357	0.001	0.005	0.000	0.000	0.006	0.000	0.000	0.000	0.000	<0.001	0.000	0.000	0.206	0.000	0.000
Xylene (Mixture)	3	4.454	0.011	0.060	0.000	0.000	0.000	0.007	0.000	0.001	0.000	0.049	0.000	0.000	0.110	0.000	0.000
Ethylene Dichloride (Mixture)	3	2.250	0.052	0.070	0.058	0.000	0.069	0.004	0.000	0.000	0.000	0.017	0.042	0.013	0.000	0.075	0.000
Trichloroethylene (Mixture)	3	5.543	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.143	0.011	0.000	0.002	0.000	0.006	0.000

TABLE 5-2 WEIGHT OF ORGANICS FOUND ON BACK-UP CHARCOAL SECTION IN PHASE II, MG.

Compound	Level	Mean Front End	Laboratory 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Benzene	5	1.543	0.005	0.000	trace	0.000	0.000	0.000	0.000	0.000	0.000	<0.020	0.000	0.000	0.000	0.000	0.000
		1.668	0.005	0.003	trace	0.000	0.002	0.000	0.000	0.000	0.000	0.031	0.000	0.007	0.000	0.000	0.001
Carbon Tetrachloride	5	15.736	4.032	0.009	0.016	0.845	0.007	0.086	0.053	0.306	0.059	1.904	0.030	0.177	0.000	0.016	0.026
		15.790	0.322	0.000	0.199	0.340	0.037	0.095	0.033	0.225	0.333	0.851	0.039	0.022	0.000	0.192	0.065
Chloroform	4	5.149	0.690	0.020	0.000	0.000	0.000	0.007	0.036	0.015	0.294	0.095	0.018	0.023	0.000	0.039	0.008
		5.031	0.220	0.000	trace	0.000	0.035	0.001	0.019	0.000	0.256	<0.001	0.013	0.114	0.000	0.003	0.035
Dioxane	5	6.781	0.043	0.020	0.000	0.000	0.000	0.000	0.000	0.003	0.668	0.181	0.008	0.009	0.000	0.001	0.000
		6.778	0.048	0.000	0.042	0.240	0.000	0.005	0.000	0.007	0.368	0.113	0.005	0.014	0.000	0.000	0.000
Ethylene Dichloride	5	8.080	0.530	0.050	0.176	0.277	0.437	0.063	0.000	0.092	1.084	0.486	0.056	0.091	0.000	0.167	0.103
		8.013	0.150	0.310	0.075	0.460	0.597	0.010	0.049	0.364	0.481	0.077	0.037	0.227	0.000	0.018	0.233
Trichloroethylene	5	15.253	0.005		0.000	0.000	0.101	0.000	0.011	0.060	0.583	0.021	0.108	0.103	0.000	0.078	0.043
		15.802	0.140	0.000	0.000	0.850	0.000	0.000	0.027	0.036	0.903	0.130	0.068	0.114	0.000	0.012	0.266
Xylene	5	8.651	0.094	0.010	0.000	0.000	0.000	0.009	0.013	0.012	0.029	0.042	0.029	0.015	0.000	0.065	0.005
		8.385	0.037	0.000	0.000	0.090	0.000	0.002	0.043	0.018	0.029	<0.044	0.008	0.007	0.000	0.024	0.008
Benzene (Mixture)	3	0.352	0.005	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	<0.043	0.000	0.001	0.000	0.000	0.001
		0.358	0.000	0.000	0.000	0.000	0.097	0.000	0.000	0.000	0.000	<0.020	0.000	0.000	0.000	0.000	0.000
Xylene (Mixture)	3	4.510	0.007	0.000	0.000	0.000	0.191	0.000	0.002	0.004	0.004	<0.001	0.003	0.004	0.000	0.005	0.002
		4.262	0.004	0.000	0.000	0.000	0.287	0.000	0.000	0.000	0.008	<0.044	0.002	0.000	0.000	0.002	0.005
Ethylene Dichloride (Mixture)	3	2.058	0.086	0.004	trace	0.090	0.233	0.000	0.000	0.000	0.232	0.055	0.012	0.011	0.000	0.023	0.000
		2.362	0.340	0.000	0.000	0.000	0.000	0.049	0.026	0.000	0.188	0.088	0.000	0.004	0.000	0.026	0.042
Trichloroethylene (Mixture)	3	5.340	1.273	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.002	0.013	0.017	0.011	0.000	0.000	0.000
		5.724	0.380	0.000	0.000	0.000	0.000	0.000	0.020	0.000	0.008	<0.001	0.000	0.007	0.000	0.000	0.000

dichloride found on the back-up was 8% in Phase I and 3% in Phase II. The mixture of ethylene dichloride and trichloroethylene showed more ethylene dichloride on the back-up section than for the same level of ethylene dichloride alone.

Because of the wide variations from lab to lab for the individual back-up sections and the small amount found on most back-ups, the data analysis was limited to amounts reported for the front ends. The possible causes of the variations in back-up values are variations in tubes, analytical errors and transfer from front to back-up sections during transport. An examination of the data patterns indicate that tube-to-tube variations are probably the major contributor to these variations. Analytical errors, which were found to be highest at low concentrations similar to those found on the back-ups, are believed to have also contributed significantly to the variation. There is no evidence that mode or distance of transport contributed to the variations in back-up values.

The analytical method recommends the analysis of back-up sections. We believe that even though this amount may be insignificant in many cases, it is necessary to analyze the back-ups if valid data are to be assured. This will also guard against errors when the sample flow through the tubes has been inadvertantly reversed.

5.2 DETERMINATION OF DESORPTION FACTORS

As part of the analytical method, each laboratory was required to determine desorption factors for each of the seven organic compounds. At least five replicate tests were made for each compound. The desorption factors determined by each laboratory for each compound are shown in Table 5-3.

The average desorption factors found for all compounds except dioxane were 0.96 ± 0.01. Dioxane, which would be expected to desorb less readily because it contains an oxygen atom, had an average desorption factor of 0.91. The data obtained by individual laboratories, however, show wide variations from the averages. For example, the 15 desorption factors reported for benzene ranged from 0.87 to 1.00. Since the desorption factor is applied to each analytical result to arrive at a corrected value,

TABLE 5-3 DESORPTION FACTORS OBTAINED BY COLLABORATORS

Compound	Laboratory															Avg.
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
Benzene	0.958	0.931	0.989	0.927	0.950	0.988	0.984	0.988	0.921	1.000	1.007	0.973	0.871	0.977	0.915	0.959
m-Xylene	0.926	0.974	0.974	0.978	0.970	0.953	0.981	0.958	0.961	0.912	0.992	0.985	0.852	1.000	0.901	0.954
Trichloroethlyene	0.982	0.993	0.953	0.947	0.895	0.979	0.985	0.940	0.964	1.000	0.999	0.985	0.966	0.990	0.912	0.966
Carbon Tetrachloride	0.863	0.989	1.004	0.899	0.921	1.074	0.970	0.984	0.977	0.893	1.007	0.984	0.999	0.970	1.010	0.970
Chloroform	0.976	0.979	0.964	0.918	0.964	0.978	0.902	0.955	0.962	1.000	1.016	0.976	0.889	0.983	0.936	0.960
Dioxane	0.876	0.959	0.896	0.968	0.815	0.885	0.974	0.949	0.842	0.883	0.951	0.927	0.911	0.981	0.880	0.913
Ethylene Dichloride	0.991	0.959	0.994	0.950	0.924	0.946	0.990	0.976	0.944	0.968	0.992	0.924	0.915	0.985	0.881	0.956
Average	0.939	0.969	0.968	0.941	0.920	0.972	0.969	0.964	0.939	0.951	0.995	0.965	0.915	0.984	0.919	

the variations in desorption factors will directly result in variations in the final data. Therefore, a significant portion of the between lab analytical error can be attributed to errors in determining desorption factors.

The analytical method was designed to minimize systematic errors in desorption factors. Each laboratory was provided with charcoal tubes from the same lot, and they were also provided with bulk samples of each organic compound from the same lot. The method specified that the same syringe be used for the addition of the organic to the charcoal as for the preparation of the standard against which it was compared. Thus, systematic errors such as inaccurate syringe delivery should have been cancelled out. A study of the five or more replicates for each compound reported by each laboratory indicates good agreement among the replicates for most laboratories. The few laboratories which had problems in replicating desorption efficiency determinations were generally the same laboratories for which a large number of outlier data points were found (see Table 4-1).

We can conclude that errors in desorption efficiency determinations contributed significantly to overall analytical error. The errors were primarily systematic but their source cannot be determined.

5.3 ANALYSES AT 5% TLV

The data analysis shows that the analytical errors at the 5% of TLV concentration were substantially greater than at the higher levels, especially when the weight of organic present was less than 0.1 mg. At first this may appear to be of relatively minor concern since the amount present is well below the maximum allowable. This is not true because to determine compliance with OSHA regulations, the concentration of each individual component must be determined and divided by its TLV. The sum of all these values must not exceed 1.0. Most industrial atmospheres contain a mixture of many organic compounds. Thus, if there are twenty compounds present, each should not exceed 5% of its TLV on the average.

The errors at the 5% of TLV level are most likely related to the gas chromatograph. Excessive noise, drift, spurious peaks and system hang-up would all cause much larger errors at low levels than at

higher levels. We believe that performance specifications for the gas chromatograph must be included in the method if accurate data at low levels are to be assured.

5.4 OUTLIER RESULTS

The relatively modest errors found at all concentration levels except the 5% of TLV must be tempered by the fact that the errors were calculated after removal of outliers. It is statistically correct to remove the outliers, but from a practical viewpoint we must recognize that such outliers would no doubt occur if the method were recommended for general use.

If limitations in the method were the cause of the outliers, we would expect a normal distribution of outliers from each laboratory about the mean for all laboratories. In Phase II, five of the fifteen laboratories accounted for more than 90% of the outliers. This strongly indicates that the method is satisfactory, but that the basic analytical techniques used by five laboratories were inadequate. The collaborative data show that the method can yield satisfactory results when used by some analysts who have no prior experience with the charcoal desorption method, but the occurrence of poor results is of much greater probability than for analysts with specific experience with this method. We believe that the best way to minimize erratic data (outliers) is to require or strongly recommend that analysts receive training in use of the method before performing routine analyses in their own facilities.

5.5 SAMPLING ERRORS

The estimates of sampling error shown in Table 4-15 are of the same order of magnitude as the errors in pump calibration shown in Table 5-4. The latter data were measured by Scott at the end of three sampling days. The Scott measurements were made using a single bubble meter and charcoal tube. The data should be free of systematic error, and thus depict the true variation from one sampler to another. Large differences, both positive and negative, between the rate reported by the collaborators and that measured by Scott are apparent in many cases.

TABLE 5-4 PUMP CALIBRATION DATA

Lab No.	4/3/73 Calib.* Flow Rate at Start 1/min.	4/3/73 Calib.* Flow Rate at End 1/min.	4/3/73 No. of Samples Collected	4/3/73 Meas.** Flow Rate at End 1/min.	4/4/73 Calib.* Flow Rate at Start 1/min.	4/4/73 Calib.* Flow Rate at End 1/min.	4/4/73 No. of Samples Collected	4/4/73 Meas.** Flow Rate at End 1/min.	4/5/73 Calib.* Flow Rate at Start 1/min.	4/5/73 Calib.* Flow Rate at End 1/min.	4/5/73 No. of Samples Collected	4/5/73 Meas.** Flow Rate at End 1/min.
1	1.00	-	40	1.166	1.00	-	50	1.15	1.0	-	50	1.026
2	1.0	-	35	.992	1.0	-	50	.842	1.0	-	50	.847
3	1.00	1.0	40	1.006	1.00	-	50	1.02	1.0	-	50	1.07
4	1.00	-	24	1.012	1.0	-	50	1.036	1.0	-	50	.993
5	1.00	1.00	40	.988	1.0	1.0	50	.914	1.0	1.0	50	.917
6	1.0	-	40	1.006	1.0	1.0	50	1.0	1.0	-	50	1.11
7	1.01	-	40	.946	1.01	1.01	50	1.11	1.01	1.01	50	.817
8	1.00	1.00	40	1.014	1.00	1.00	50	.834	1.0	1.0	50	1.005
9	.974	1.027	40	1.152	.993	-	50	1.05	1.0	-	25	1.024
10	1.01	1.00	40	1.14	.975	.960	50	1.29	.993	.914	50	.958
11	1.01	.996	40	.972	1.00	1.00	40	1.036	1.0	1.0	50	1.128
12	1.00	.841	40	.960	1.00	-	-	1.026	1.0	-	50	1.135
13	1.00	1.00	40	.998	1.0	-	50	.992	1.0	-	50	.997
14	1.00	1.00	40	.946	1.00	1.00	50	.936	1.0	1.0	50	.923
	1.01	1.01	40	1.020	1.01	1.01	50	1.016	1.01	-	50	1.036
15	1.00	1.00	40	.982	1.00	1.00	50	1.006	1.0	1.0	50	.979

* Reported by Collaborator
** Measured by Scott

The average measured flow rate was close to the 1.00 l/min. desired, and many of the individual flow rates were reasonably close to this value. On the other hand a significant number of pumps gave flows either less than 0.9 or greater than 1.1 l/min.

These facts vindicate the calibration method and tend to indicate that the pumps were adequate. The problem appears to be that some participants did not perform the calibration correctly. This demonstrates the need for a brief training course in sampling procedures and sampler calibration. This was not done in the Phase II sampling because it would have biased the data.

Some laboratories, notably Laboratory No. 2, included the estimated sample size on their analytical report sheets. By correcting for sample size, the results could possibly have been improved. However, since the method specifically stated that a ten liter sample be taken, and estimated sample size data were reported in a limited number of cases, no corrections were made.

5.6 COMMENTS FROM COLLABORATORS

The collaborators were asked to comment on the method. These comments and suggestions are summarized below.

1. The repeated injection of 5µl samples of carbon disulfide causes detector fouling which leads to poor results and unusually high percentage of down time. Several laboratories felt they obtained better results with the 1 or 2 µl samples they used in their own work.
2. Some laboratories also noted detector problems when the chlorinated compounds were analyzed.
3. "Ghosting" occurred when high concentrations of solute were followed by much lower concentrations.
4. Heat is generated when carbon disulfide is added to the charcoal, and some vapor may be lost.
5. Better quality control in the manufacture of the charcoal tubes is necessary. Some tubes had the urethane divider in the wrong place or not clearly dividing the two sections.

6. The tube caps were hard to remove and hard to replace for a good seal.

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Martha Seymour Jim Woodfin Dave Sundin Alexander Teass Chuck McCammon Richard Kupel Ann Saalwaechter Kathy Schulte	National Institute for Occupational Safety and Health Cincinnati, Ohio
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APPENDIX A
SAMPLING AND ANALYTICAL METHODS

TENTATIVE METHOD FOR THE SAMPLING OF ORGANIC COMPOUNDS WITH CHARCOAL ADSORPTION TUBES

1. Scope

A method is described for collecting organic vapors on charcoal adsorption tubes.

2. Principle of the Method

A known volume of air is drawn through a charcoal adsorption tube by means of a battery-powered sampling pump. Any organic vapors present will be adsorbed onto the charcoal.

3. Apparatus

3.1 Sampling Pump: An approved coal mine dust personal sampling pump or any vacuum pump whose flow can accurately be determined at 1 liter per minute.

3.2 Charcoal Tubes: Glass tube with both ends flame sealed, 7 cm. long with a 6 mm OD and 4 mm. ID, containing 2 sections of 20/40 mesh activated charcoal separated by a 2 mm. portion of urethane foam. The adsorbing section contains 100 mg. of charcoal, the back-up section 50 mg.

3.3 Calibration apparatus for pump flow rate.

4. Sampling Procedure

The personal sampling pumps to be used should not be run continuously for more than 4 hours unless necessary. These pumps will run 8 hours continuously, but after the battery starts to run down, the flow will slowly start to drop. To be safe in running for a full day, run one pump for 4 hours and then substitute with another pump for the remaining 4 hours. The first pump can be recharged while the second pump is in operation in case a back-up pump is needed. All pumps should be charged overnight before use the following day. In reiteration, one pump can be used for a full 8 hours, but it would be more accurate to use one pump only for a total of 4 hours running time.

All pumps should be calibrated before departure to the site of sampling. When sampling over a period of days, it is recommended that the pump (or pumps) be calibrated immediately before and immediately after use each day. A calibration check should take a maximum of 5 minutes.

During actual sampling, the length of tube connecting the charcoal tube to the pump should be as short as possible and there should be no tubing ahead of the charcoal tube. After all sampling for a day has been completed, check the flow rate on the pump(s) to see if it had radically changed. If a noticeable change has occurred, record the change for future correlation to sample analysis.

Both ends of the charcoal tube should be broken to provide an opening of at least 2 mm., which is one-half the ID of the tube. A smaller opening causes a limiting orifice effect which reduces the flow through the tube. The smaller section of charcoal in the tube is used as a back-up section and should therefore be placed nearest the sampling pump.

One liter per minute is the recommended sampling rate. A 10 liter sample is normally adequate. The tube must be sampled in a vertical position. One charcoal tube should be treated in the same manner as the sample tubes (break, seal, ship) with the exception that no air be drawn through it. This tube will serve as a blank.

5. Calibration Procedure

The equipment used to calibrate a personal sampling pump consists of a pump, a 1000 ml. calibrated buret (bubble meter), several varied lengths of ¼" ID Tygon tube (or the equivalent), a standard charcoal tube (with a pressure drop of about 10" H_2O across it), a H_2O manometer (optional), liquid detergent (or other soap), and a 200 ml. beaker (see Figure 1). After the equipment is set up, turn on the personal sampling pump and determine the pressure drop across the charcoal tube from the manometer. This pressure drop should be 9-11" of H_2O. Moisten the interior surface of the buret and draw several bubbles up the buret. Measure the time it takes a bubble to move from one calibration to another, say from 0 to 500 ml. The flow is then determined. By setting the rotometer at different calibrations and measuring the flow, a graph of flow (l/min) vs. rotometer readings is obtained. Determine where the 1 ppm setting is and set the pump for this flow. Determine the exact flow and the length of time the pump must run to draw a 10 liter sample through the charcoal tube. All samples should be taken for this length of time.

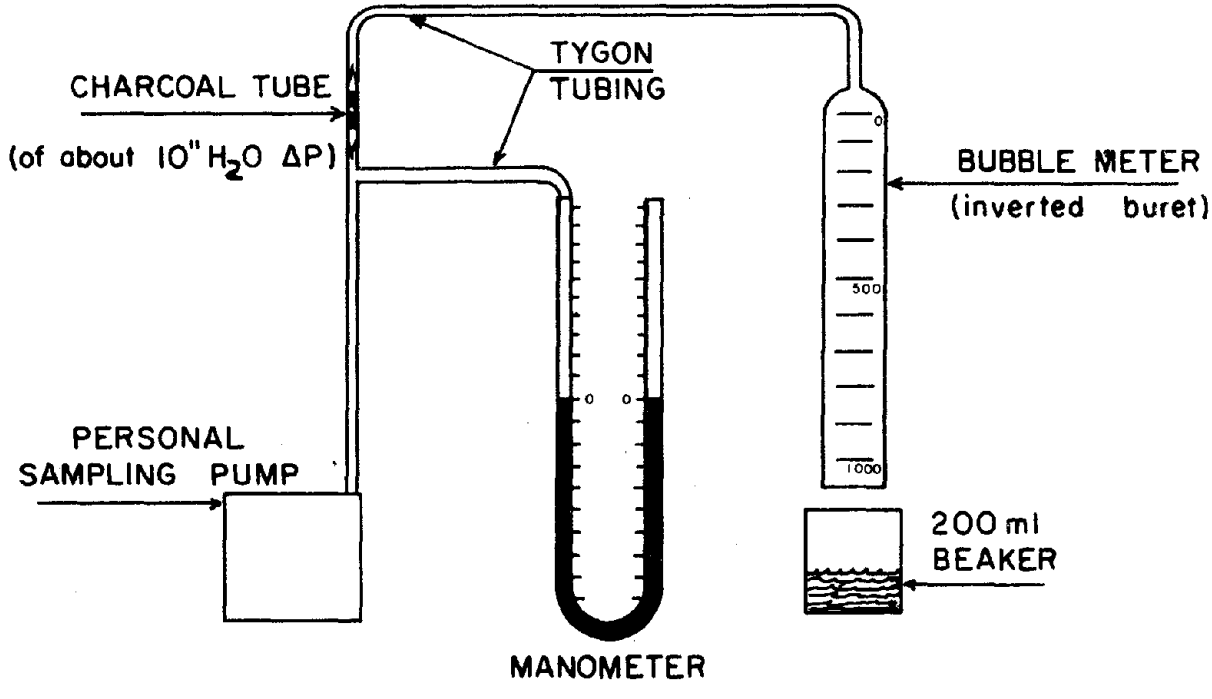

Figure 1. Personal sampling pump calibration setup.

TENTATIVE METHOD FOR THE ANALYSIS OF ORGANIC COMPOUNDS ADSORBED ON CHARCOAL TUBES

1. Scope

A method is described for analyzing various organic compounds which have been adsorbed onto charcoal in glass tubes.

2. Principle of the Method

The charcoal containing organic vapors is transferred from the collection tube to a small test tube. It is desorbed with carbon disulfide, and an aliquot is injected into a gas chromatograph. Concentrations are determined by comparing the area of the resulting chromatographic peak with areas obtained from the injection of standards.

3. Apparatus

3.1 Charcoal tubes: Glass tube with both ends flame sealed, 7 cm. long with a 6 mm. OD and 4 mm. ID, containing 2 sections of 20/40 mesh activated charcoal separated by a 2 mm. portion of urethane foam. The adsorbing section contains 100 mg. of charcoal, the back-up section 50 mg.

3.2 Gas chromatograph equipped with a flame ionization detector.

3.3 Column capable of separating the organics to be determined.

3.4 An integrator or a recorder with some method for determining peak area.

3.5 Glass-stoppered test tubes: 100 x 13 mm. (Corning 9810 or equivalent).

3.6 Syringes: 10 µl and convenient sizes for making standards, (Hamilton or equivalent).

4. Reagents

4.1 Spectroquality carbon disulfide.

4.2 Bulk samples of organics to be determined.

4.3 Grade A helium.

4.4 Prepurified hydrogen.

4.5 Oxygen or filtered compressed air.

5. Analytical Procedure

5.1 Cleaning of Equipment

All equipment used in the analysis should be detergent washed followed by tap and distilled water rinses.

5.2 Preparation of Samples

In preparation for analysis, each charcoal tube is scored with a file in front of the first section of charcoal and broken open. The glass wool is removed and discarded. The charcoal in the first (larger) section is transferred to a glass stoppered test tube. The separating section of foam is removed and discarded; the second section is transferred to another test tube. These two sections are analyzed separately.

5.3 Desorption of Samples

One-half milliliter of carbon disulfide is pipetted into each test tube and tube is stoppered immediately. (All work with carbon disulfide should be performed in a hood because of its high toxicity.) Allow samples to desorb for thirty minutes with occasional agitation.

5.4 Gas Chromatograph Conditions

A gas chromatograph with flame ionization detector is set up with appropriate column and conditions to separate the organics to be analyzed.

5.5 Injection of Samples

To eliminate difficulties arising from blowback or distillation within the syringe needle, the solvent flush injection technique is employed. The 10 µl syringe is first flushed with carbon disulfide several times to wet the barrel and plunger. Two microliters of carbon disulfide are drawn into the syringe to increase the accuracy and reproducibility of the injected sample volume. The needle is removed from the carbon disulfide and the plunger is pulled back about 0.2 µl to separate the solvent flush from the sample with a pocket of air to be used as a marker. The needle is then immersed in the sample, and a 5 µl aliquot is withdrawn, taking into consideration the volume of the needle, since the sample in the needle will be completely injected. After the needle is removed from the sample and prior to injection, the plunger is pulled back a short distance to minimize evaporation of the sample from the tip of the needle. After injection the needle is allowed to remain in the injection block for several seconds to insure complete evaporation of the solvent. Duplicate injections of each sample should be made.

5.6 Measurement of Peak Area

The area of the sample peak is measured by an electronic integrator or some other suitable form of area measurement, and preliminary sample results are read from a standard curve prepared as discussed below.

6. Calibration and Standards

6.1 Preparation of Standards

It is convenient to prepare standards in terms of mg/0.5 ml CS_2 because samples are desorbed in this amount of CS_2. To minimize error due

to the volatility of carbon disulfide, twenty times the weight can be injected into 10 ml of CS_2. For example, to prepare a 0.3 mg./0.5 ml. standard, 6.0 mg is injected into exactly 10 ml of CS_2 in a glass-stoppered flask. The density for the organic is used to convert 6.0 mg into microliters for easy measurement with a microliter syringe. A series of at least three standards is prepared varying in concentration over the range of interest and analyzed under the same G.C. conditions and during the same time period as the unknown samples. Curves are established by plotting concentration versus average peak area. New standards should be made up daily.

6.2 Determination of Desorption Efficiency

It is necessary to determine the percentage of each organic on the charcoal that is removed in the desorption process. This desorption efficiency may be determined once for a given compound for each batch of charcoal used in the tubes. It is convenient to use the charcoal from an unexposed charcoal tube for desorption efficiency determinations. The 100 mg. of charcoal from the front adsorbing section is placed in its original tube or in a 4 mm. ID x 50 mm. long glass tube with one end sealed. The open end is capped with Parafilm. The tube must contain only charcoal, and all urethane foam, glass wool, etc. must be removed.

A known volume of the organic is injected directly onto the activated charcoal with a microliter syringe, and the tube is capped with more Parafilm. The amount injected is usually equivalent to that present in a 10 liter sample at a concentration equal to the federal standard. A minimum of five tubes are prepared in this manner and allowed to stand for at least one day to assure complete adsorption of the organic onto the

the charcoal. These tubes are desorbed and analyzed in exactly the same manner as sampling tubes except that the standards are made up by injecting the same amount of organic as was injected onto the charcoal into 0.5 ml of CS_2 in a glass-stoppered tube. The same syringe is used so that any error in measuring the organic should be cancelled out. The results of each analysis are compared to the standards to determine what fraction of the original amount of organic was desorbed. The average fraction desorbed is used as a factor in all sample analyses.

7. Calculations

The first step in determining the results is to read the weight in milligrams corresponding to each peak area from the standard curve. The standard curve is based on mg/0.5 ml CS_2, and therefore, no correction need be made for the volume of the sample injected, since this is identical to the volume of the standards injected.

The weight of organic on the front section of the blank is subtracted from the weight determined for the front section of each sample; a similar procedure is followed for the back-up sections.

Amounts present on the front and back-up sections of the same tube are then added together to determine the total amount detected in the sample.

This total weight on the tube is corrected by dividing by the desorption factor to determine the total number of milligrams in the sample.

Milligrams can be converted into parts per million concentration by volume in the air sampled by the following equation:

$$\text{at } 25^{\circ}\text{C parts per million} = \frac{24{,}450 \text{ ml/mole} \times \text{mg/liter}}{\text{molecular wt.}}$$

INSTRUCTIONS FOR ANALYSIS OF CHARCOAL TUBES

PHASE I OF NIOSH COLLABORATIVE STUDY

The analytical method to be followed is attached. It has been revised to reflect decisions made at the seminar on February 15. The analyses must be performed according to the method as presented at the seminar. The calculations will be performed as described below instead of as indicated in Section 7 of the method.

You have been shipped two packages. The first contains 10 cc vials of each of the seven solvents to be determined in the program. Use these vial samples for making all standards and performing desorption efficiency determinations. Assume that the solvent purity is 100%. The second package contains 180 charcoal tubes with adsorbed organics and 70 blank, unopened tubes for desorption determinations.

The analysis schedule for your laboratory is also attached. The tubes are to be analyzed on the days specified and in the order specified. The tubes are marked by a code number. The first two digits are your laboratory code. This is denoted by xx on the schedule. The 144 tubes to be analyzed are scheduled over a ten day period. The test days need not be consecutive calander days, but must follow the specified chronological order. It is most desirable that all tubes scheduled for a particular day be analyzed within the same day. Only those back-up sections marked with an asterisk are to be analyzed. The remaining back-up sections are to be left in their tubes, capped and stored for possible future analysis. It is recommended that analyses be performed within 90 minutes of the start of desorption.

All tubes scheduled for analysis end in the digit 1, 2, 3 or 4. Tubes ending in the digit 0 are included as spares. If any of the tubes scheduled are broken in shipment or if the sample is lost in analysis, substitute the spare tube with the same code number except for the last digit. For example, tube number 17030400 should be substituted for 17030402 if the latter is lost.

The enclosed data sheets are to be used to transmit your data to us. Use one sheet for each day's schedule. A sample sheet is attached. Enter the code number of the tube in columns 3 through 10. Enter the compound analyzed for in columns 11 through 20. Use separate lines where more than one compound is analyzed. In column 21 enter F for front section and B for back-up section. Enter the average weight of organic found as read from your calibration curve in columns 22 through 27 as xx.xxxx mg The desorption factor for the compound is entered in columns 28 through 31 as x.xxx. The corrected weight (average weight divided by the desorption factor) is enterred in columns 32 through 37 as xx.xxxx mg. Columns 38 through 61 are to be used for enterring the replicate determination data (not corrected for the desorption factor). The remaining columns are to be used for pertinent remarks such as tubes analyzed on the following day, sample spilled etc.

The data sheets for desorption factor determinations and general information are self explanatory. If you have any question do not hesitate to call Louis Reckner at 215-766-8861.

INSTRUCTIONS FOR ANALYSES OF CHARCOAL TUBES
PHASE II OF NIOSH COLLABORATIVE STUDY

The analytical procedure to be followed in Phase II is identical to that used in Phase I except that it will be required that standards be prepared which are within a factor of two of all samples. Since the amounts of hydrocarbons adsorbed on the Phase II samples are in the same range as the Phase I samples, you should be able to determine what standard concentrations you will need prior to analyzing the samples. The requirement for additional standards is being added because in Phase I the precision of the data for the samples representing concentrations near the TLV was considerably better than that for samples with either greater and lesser amounts.

The analysis schedule for your laboratory is attached. The tubes are to be analyzed on the days specified and in the order specified. The tubes are marked by a code number. The first two digits are your laboratory code. This is denoted by xx on the schedule. The tubes to be analyzed are scheduled over a ten day period. The test days need not be consecutive calander days, but must follow the specified chronological order. It is most desirable that all tubes scheduled for a particular day be analyzed within the same day. Only those back-up section marked with an asterisk are to be analyzed. The remaining back-up sections are to be left in their tubes, capped and stored for possible future analysis. It is recommended that analyses be performed within 90 minutes of the start of desorption.

In Phase II tubes scheduled for analysis end in the digits 12, 13, 14 or 15. Tubes ending in the digits 11 are included as spares. If any of the tubes scheduled are broken in shipment or if the sample is lost in analysis, substitute the spare tube with the same code number except for the last digit. For example, tube number 17030411 should be substituted for 17030412 if the latter is lost.

In order to compare the Phase II data to Phase I data, a number of the unanalyzed spare tubes from Phase I have been included in your Phase II schedule. This should not require additional time on your part because the number of back-up sections to be analyzed has been reduced. In addition, you will not have to perform any desorption experiments, and you should use the desorption factors you determined in Phase I.

The data sheets are to be filled out as in Phase I. Please note that the decimal is to be positioned at the point indicated by the dotted line in the column headings. General information data sheets should also be filled out as in Phase I.

APPENDIX B
PHASE I SCHEDULE, DATA AND DATA ANALYSIS

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

Laboratory No. ____ Day No. A

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx050401	Etnylene Dichloride
2	xx050204	"
3	xx050104	"
4	xx050203*	"
5	xx050402	"
6	xx050103*	"
7	xx090104	Ethylene Dichloride & Trichloroethylene
8	xx090103*	"
9	xx060504	Trichloroethylene
10	xx060302	"
11	xx060201	"
12	xx060503	"
13	xx060202	"
14	xx060301*	"
15	xx	
16	xx	

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. B

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx050304	Ethylene Dichloride
2	xx050202	"
3	xx050201	"
4	xx050501	"
5	xx050502	"
6	xx050303*	"
7	xx090102	Ethylene Dichloride & Trichloroethylene
8	xx090101	"
9	xx060203*	Trichloroethylene
10	xx060102	"
11	xx060204	"
12	xx060401*	"
13	xx060101	"
14	xx060402	"
15	xx	
16	xx	

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. C

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx050302	Ethylene Dichloride
2	xx050503*	"
3	xx050102	"
4	xx050301	"
5	xx050101	"
6	xx050404	"
7	xx050504	"
8	xx050403*	"
9	xx040302	Dioxane
10	xx040103*	"
11	xx040203	"
12	xx040504	"
13	xx040104	"
14	xx040301*	"
15	xx040204	"
16	xx040503	"

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. D

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx010304	Benzene
2	xx010204	"
3	xx010401*	"
4	xx010303	"
5	xx010101*	"
6	xx010402	"
7	xx010203	"
8	xx010102	"
9	xx030104	Chloroform
10	xx030301*	"
11	xx030204	"
12	xx030103*	"
13	xx030402	"
14	xx030302	"
15	xx030203	"
16	xx030401	"

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES
(continued)

Laboratory No. ____ Day No. E

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx030202	Chloroform
2	xx030303	"
3	xx030404	"
4	xx030101	"
5	xx030201*	"
6	xx030102	"
7	xx030403*	"
8	xx030304	"
9	xx070501*	Xylene
10	xx070403	"
11	xx070202	"
12	xx070304	"
13	xx070502	"
14	xx070404	"
15	xx070201*	"
16	xx070303	"

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

FIGURE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. F

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx010202	Benzene
2	xx010503*	"
3	xx010201*	"
4	xx010403	"
5	xx010504	"
6	xx010404	"
7	xx080101	Benzene & Xylene
8	xx080102	"
9	xx070203	Xylene
10	xx070102	"
11	xx070402	"
12	xx070204	"
13	xx070101*	"
14	xx070401*	"
15	xx	
16	xx	

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. G

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx010301*	Benzene
2	xx010103	"
3	xx010502	"
4	xx010302	"
5	xx010501	"
6	xx010104	"
7	xx080104	Benzene & Xylene
8	xx080103*	"
9	xx070503	Xylene
10	xx070104	"
11	xx070302	"
12	xx070504	"
13	xx070301*	"
14	xx070103	"
15	xx	
16	xx	

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. H

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx060103*	Trichloroethylene
2	xx060303	"
3	xx060403	"
4	xx060502	"
5	xx060404	"
6	xx060104	"
7	xx060501*	"
8	xx060304	"
9	xx020402	Carbon Tetrachloride
10	xx020301	"
11	xx020504	"
12	xx020204	"
13	xx020302	"
14	xx020503	"
15	xx020401*	"
16	xx020203*	"

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. I

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx040202	Dioxane
2	xx040403	"
3	xx040102	"
4	xx040501*	"
5	xx040201*	"
6	xx040101	"
7	xx040404	"
8	xx040502	"
9	xx020201	Carbon Tetrachloride
10	xx020304	"
11	xx020101*	"
12	xx020403	"
13	xx020303*	"
14	xx020102	"
15	xx020404	"
16	xx020202	"

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. J

Order of Analysis	Charcoal Tubs Code Number	Compound(s) to be Determined
1	xx040303	Dioxane
2	xx040402	"
3	xx040304	"
4	xx040401*	"
5	xx020502	Carbon Tetrachloride
6	xx020103	"
7	xx020501*	"
8	xx020104	"
9	xx	
10	xx	
11	xx	
12	xx	
13	xx	
14	xx	
15	xx	
16	xx	

* The back-up charcoal is to be analyzed in these tubes.

NOTE: The "xx" in the charcoal tube code numbers refers to your laboratory number as shown above.

TABLE B-2 SCHEDULE OF SAMPLING DAYS
FOR 15 LABORATORIES IN PHASE I

Lab	A	B	C	D	E	F	G	H	I	J
1	3	4	5	7	8	9	10	2	6	1
2	10	7	5	4	1	9	8	3	2	6
3	8	5	6	9	2	4	7	1	10	3
4	8	10	6	1	2	7	5	3	4	9
5	8	2	9	6	4	10	3	7	1	5
6	7	1	10	2	9	3	4	5	6	8
7	1	2	5	10	4	6	7	9	8	3
8	1	8	2	3	5	10	6	4	7	9
9	9	4	8	7	6	2	1	10	3	5
10	4	10	5	7	6	1	2	9	8	3
11	8	2	7	10	5	4	6	1	3	9
12	3	4	8	5	7	9	6	2	10	1
13	5	6	9	8	1	2	10	4	3	7
14	10	6	4	5	3	2	7	8	9	1
15	1	5	4	9	7	8	3	10	6	2

TABLE B-3

PHASE I ANALYTICAL RESULTS

DATE 06/29/73

BENZENE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	.01	.03	.01	.02	.01	.01	.01	.02	.02	.02	.01	.01	.78	.01	.01	.02
		.00	.03	.01	.02	.01	.01	.02	.02	.02	.02	.01	.01	.17	.01	.01	.02
	2	.02	.02	.02	.03	.02	.01	.02	.02	.02	.02	.02	.02	.02	.02	.01	.02
		.02	.02	.02	.03	.03	.01	.02	.02	.02	.02	.01	.02	.57	.02	.01	.02
2	1	.17	.17	.19	.15	.15	.16	.18	.16	.18	.18	.18	.17	.28	.17	.13	.18
		.20	.20	.19	.16	.22	.19	.19	.19	.20	.26	.18	.21	.20	.19	.14	.20
	2	.22	.22	.20	.21	.20	.21	.21	.21	.21	.21	.20	.21	.17	.20	.14*	.21
		.03*	.23	.21	.16	.18	.20	.22	.21	.22	.20	.20	.21	.19	.21	.16	.22
3	1	.27	.32	.32	.32	.28	.26	.30	.29	.41*	.25	.29	.30	3.68	.29	.25	.29
		.33	.34	.37	.35	.31	.29	.33	.30	.35	.27	.32	.33	.35	.32	.25	.33
	2	.43	.40	.31	.29	.37	.34	.37	.36	.37	.34	.34	.35	.26	.35	.26	.36
		.47	.39	.35	.32	.38	.35	.37	.37	.38	.33	.37	.36	.31	.36	.25	.37
4	1	.68	.69	.56	.55	.53	.53	.56	.58	.57	.52	.53	.57	.36	.56	.40	.56
		.67	.63	.64	.64	.63	.59	.60	.64	.63	.58	.62	.64	.41	.61	.51*	.62
	2	.60	.68	.78	.71	.85	.66	.65	.67	.69	.64	.72	.68	.80	.68	.55	.67
		.82	.73	.61	.68	.74	.65	.74	.71	.71	1.55*	.71	.69	.91	.70	.54	.69
5	1	1.50	1.53	1.56	1.67	1.40	1.33	1.45	1.49	1.42	1.12	1.36	1.43	1.59	1.38	1.17	1.39
		1.70	1.73	1.57	1.88	1.98	1.42	1.62	1.69	1.61	1.27	1.53	1.57	1.59	1.55	1.44	1.54
	2	1.60	1.94	1.89	1.86	1.89	1.89	1.67	1.78	1.72	3.10*	1.54	1.68	1.51	1.71	1.42	1.69
		2.20	1.93	1.71	2.10	1.95	1.75	1.65	1.69	1.78	1.76	1.64	1.74	1.09	1.73	1.36	1.74

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CARBON TETRACHLORIDE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	.00	.05	.06	.00	.05	.05	.07	.03	.03	.13	.04	.04	.19	.02	.10	.02
		.00	.04	.03	.00	.05	.02	.07	.04	.03	.09	.04	.04	.20	.03	.09	.03
	2	.00	.05	.05	.00	.05	.04	.07	.04	.05	.01	.03	.05	.05	.03	.02	.03
		.00	.05	.05	.00	.07	.04	.07	.05	.06	.02	.03	.05	.00	.03	.02	.03
2	1	1.33*	.48	.47	.47	.51	.38	.46	.39	.41	.44	.41	.44	.92	.40	.38	.38
		.49	.53	.53	.40	.55	.44	.52	.42	.45	.52	.49	.49	.36	.45	.42	.41
	2	.23	.53	.56	.24	.47	.46	.55	.45	.48	.58	.54	.59	.51	.53	.44	.45
		.56	.54	.59	.29*	.54	.50	.53	.54	.52	.36*	.52	.57	.77	.54	.48	.47
3	1	1.10*	.66	.83	.49	.69	.63	.63	.69	.62	.50	.68	.70	.81	.69	.59	.61
		.96	.74	.76	.43	.73	.72	.83	.75	.72	.77	.77	.83	.74	.76	.49	.67
	2	.88	.91	.93	.93	.88	.78	.78	.75	.82	.99	.81	.86	.40	.80	.69	.74
		.91	.98	.92	.81	.94	.78	.77	.78	.85	.93	.80	.87	.95	.80	.70	.76
4	1	1.28	1.34	1.46	1.50	1.59	1.46	1.31	1.10	1.30	1.48	1.34	1.33	1.31	1.35	.94	1.28
		1.85	1.47	1.95	1.26	1.59	1.70	1.54	1.51	1.44	1.48	1.55	1.49	1.83	1.56	1.39	1.41
	2	2.90*	1.92	1.79	1.70	1.91	1.91	1.50	1.53	1.55	2.07	1.64	1.66	.00	1.62	1.24	1.55
		2.30	1.85	1.66	1.49	2.10	1.84	1.56	1.24	1.56	1.96	1.66	1.71	.40	1.63	1.11	1.59
5	1	14.77	14.05	13.49	15.26	14.11	17.24	10.14	13.83	12.79	2.13*	12.16	13.65	13.68	13.90	12.53	13.77
		15.52	15.22	13.70	11.69	15.45	19.96	.67*	15.08	14.13	2.78*	13.59	15.91	16.82	16.11	14.66	15.22
	2	20.70	17.49	16.27	19.48	19.84	21.59	13.18	17.35	15.38	17.08	14.89	17.50	18.04	15.92	13.83	16.65
		23.10	17.34	13.97	24.16	18.07	22.68	15.07	18.67	15.03	17.24	14.99	18.83	18.04	15.44	17.48	17.14

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CHLOROFORM (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	.10	.10	.20*	.75*	.19*	.10	.12	.10	.12	.12	.12	.11	.11	.09	.11	.10
		.09	.08	.24*	.78*	.17*	.10	.14	.09	.12	.15	.12	.12	.00	.10	.11	.12
	2	.09	.20	.11	.05	.24	.11	.16	.14	.13	.14	.14	.12	.00	.13	.20	.13
		.08	.22	.14	.10	.16	.12	.16	.17	.15	.15	.14	.13	.00	.13	.23	.13
2	1	1.30	1.39	1.56	1.61	1.36	1.29	1.62	1.49	1.37	1.50	1.29	1.38	2.08	1.44	1.36	1.37
		.12*	1.61	1.59	1.61	1.34	1.37	1.70	1.50	1.47	1.60	1.39	1.50	2.28	1.43	1.44	1.51
	2	1.90	1.63	1.66	1.10	2.09	1.55	1.90	1.69	1.66	1.77	1.47	1.68	1.61	1.67	1.75	1.66
		2.20	1.81	1.99	1.32	1.61	1.66	2.16	1.95	1.80	1.88	1.60	1.73	1.59	1.79	1.64	1.71
3	1	2.60	2.23	2.28	1.62	2.31	2.07	2.58	2.33	2.30	2.35	2.16	2.31	.00	2.34	1.98	2.32
		2.80	2.38	2.35	2.22	2.57	2.38	2.78	2.60	2.56	2.55	2.23	2.44	2.65	2.46	2.34	2.57
	2	3.10	2.40	2.73	3.06	2.41	2.70	2.83	2.75	2.61	3.05	2.52	2.73	3.86	2.74	2.52	2.81
		1.50*	2.71	2.85	2.97	2.85	3.01	3.57	3.24	2.85	3.40	2.76	2.88	4.04	2.85	3.56	2.89
4	1	4.10	4.21	4.22	3.62	4.40	4.51	4.40	4.34	4.31	4.38	4.06	4.08	.00	4.35	3.80	4.25
		4.90	4.73	4.58	4.87	4.45	4.53	5.25	5.16	4.65	4.60	4.54	4.55	4.25	4.67	4.30	4.70
	2	3.50	5.29	4.81	6.76	4.83	5.09	5.39	5.52	4.98	4.95	4.90	5.32	7.30	4.99	4.26	5.14
		3.60	5.25	5.50	7.27	5.77	5.78	6.07	5.88	5.43	5.50	5.23	4.95	7.08	5.32	4.97	5.30

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DIOXANE (MG)

LEVEL	DAY	LAB 1*	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	2.10	.19	.21	.41	.15	.16	.00	.17	.18	.20	.16	.18	.27	.17	.15	.20
		2.00	.21	.24	.43	.18	.20	.00	.20	.25	.22	.18	.21	.45	.19	.17	.22
	2	2.30	.27	.39	.42	.14	.20	.00	.23	.25	.26	.23	.23	.26	.22	.20	.24
		.78	.28	.22	.44	.07	.20	.00	.21	.30	.24	.23	.22	.19	.22	.21	.25
2	1	*	1.73	2.15	2.01	2.15	1.85	1.40	1.79	1.98	1.88	1.83	1.82	1.13	1.73	1.65	1.92
		3.29	1.98	2.31	2.27	2.29	2.14	1.87	2.05	2.23	2.17	2.17	2.06	1.82	1.99	1.80	2.12
	2	9.00	2.30	2.66	2.25	2.57	2.27	2.01	2.31	2.40	2.58	2.19	2.32	2.15	2.24	2.24	2.32
		4.30	2.26	2.43	2.32	2.61	2.34	1.94	2.28	2.54	2.66	2.21	2.30	2.75	2.21	2.36	2.39
3	1	7.10	3.06	3.53	3.17	4.14	3.12	2.81	3.29	3.68	3.45	3.10	3.38	3.38	3.23	3.30	3.41
		16.00	3.64	4.35	4.02	4.57	3.64	3.65	3.83	4.03	4.98	3.43	3.42	4.57	3.82	3.85	3.77
	2	4.42	3.87	4.88	4.09	4.56	3.93	3.96	4.15	4.44	4.42	4.01	4.27	4.76	4.03	3.92	4.13
		4.45	3.92	6.29*	4.29	4.71	4.32	3.99	4.26	4.74	4.36	4.16	4.45	3.62	4.01	4.06	4.25
4	1	5.16	4.59	5.82	2.50*	5.62	5.20	4.80	4.93	5.36	4.98	4.92	5.11	5.49	4.78	4.76	5.20
		6.10	5.23	7.03*	5.54	6.37	5.94	5.80	5.93	6.07	5.80	5.61	5.90	5.52	5.36	5.44	5.75
	2	7.40	6.05	5.11	6.40	6.86	6.39	6.20	6.31	6.84	5.80	6.07	6.30	4.36	6.04	6.11	6.29
		6.45	5.94	5.23	6.66	8.42*	6.54	6.45	6.36	7.45	5.85	6.27	6.36	4.29	6.04	6.00	6.47
5	1	6.48	5.70	6.60	6.33	7.97*	6.14	6.28	5.93	6.65	5.45	5.85	5.92	2.66	5.52	5.77	6.15
		7.47	6.60	7.31	7.13	9.52*	7.04	7.23	6.58	7.25	6.00	6.84	6.70	4.47	6.35	6.63	6.80
	2	11.00	7.20	7.34	7.72	8.60	7.59	7.15	7.67	7.87	8.04	7.08	7.60	7.10	7.33	7.36	7.44
		19.00	7.30	9.35	6.44	9.20	7.39	8.02	7.70	8.35	7.28	7.29	7.80	6.81	7.39	7.33	7.66

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ETHYLENE DICHLORIDE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	.10	.08	.08	.20*	.10	.08	.09	.11	.20*	.09	.08	.09	.54	.08	.09	.08
		.11	.10	.10	.21*	.10	.10	.10	.12	.26*	.11	.09	.11	.59	.09	.10	.09
	2	.13	.13	.10	.33*	.12	.11	.10	.24*	.13	.12	.12	.12	1.04	.11	.12	.10
		.13	.11	.10	.33*	.10	.12	.10	.23*	.13	.13	.12	.13	.90	.11	.13	.11
2	1	1.70	1.26	1.21	1.60	1.32	1.36	1.18	1.31	1.31	1.29	1.27	1.33	2.20	1.21	1.24	1.21
		1.70	1.42	1.49	1.72	1.40	1.55	1.38	1.55	1.52	1.47	1.45	1.52	5.61	1.42	1.42	1.34
	2	1.70	1.60	1.64	1.80	1.58	1.73	1.70	1.67	1.57	1.76	1.61	1.69	2.84	1.49	1.54	1.47
		2.10*	1.56	1.63	1.84	1.65	1.80	1.68	1.53	1.60	1.81	1.63	1.73	3.45	1.52	1.50	1.55
3	1	2.70*	2.00	1.73	2.03	1.82	1.94	1.86	1.78	1.87	1.79	1.78	1.95	4.79	1.78	1.85	1.89
		3.50*	2.09	1.89	2.05	2.13	2.14	2.08	2.02	2.12	1.92	1.93	2.07	3.14	1.98	1.81	2.09
	2	2.30	2.24	1.94	2.89*	2.34	2.27	2.39	2.31	2.30	2.27	2.21	2.35	2.09	2.18	2.01	2.29
		2.50	2.25	1.96	3.12	2.48	2.60	2.11	2.60	2.50	2.40	2.43	2.60	6.34	2.35	2.23	2.35
4	1	4.50	3.61	3.97	4.19	3.93	3.82	3.37	2.26	1.19*	3.72	3.34	3.62	6.94	3.25	3.32	3.39
		4.10	3.59	3.58	4.20	3.98	2.10*	3.82	3.71	3.74	3.45	3.61	3.95	5.48	3.48	3.41	3.74
	2	3.90	4.31	4.36	4.30	4.19	4.18	3.83	3.95	3.91	4.39	4.00	3.81	1.19	3.97	3.74	4.16
		4.70	4.45	4.28	4.77	3.98	4.54	4.10	4.64	4.15	4.03	4.33	4.52	2.10	4.29	4.59	4.21
5	1	8.10	6.73	6.79	9.00	7.28	7.65	6.56	7.25	6.67	6.15	6.83	7.23	14.81	6.70	7.08	7.47
		8.70*	7.36	7.49	9.57*	7.86	7.82	6.99	7.32	7.43	6.97	7.33	7.92	14.60	7.43	7.38	8.26
	2	10.00	8.55	8.09	9.14	8.88	8.63	7.99	8.51	6.97	6.95	7.76	8.53	18.86	8.40	8.78	8.60
		8.00	9.33	8.75	9.58	8.62	9.02	7.61	9.81	7.45	7.33	8.60	9.09	1.27	9.17	8.94	9.30

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TRICHLOROETHYLENE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	.14	.19	.27	.39	.07	.00	.17	.31	.17	.17	.15	.15	.76	.15	.17	.16
		.19	.19	.35	.40	.17	.17	.18	.32	.18	.18	.19	.18	.04	.18	.17	.18
	2	.18	.20	.22	.00	.08	.18	.17	.25	.24	.05	.22	.19	.07	.20	.22	.19
		.20	.24	.26	.00	.14	.19	.18	.27	.27	.09	.00	.19	.04	.20	.25	.20
2	1	3.30	2.82	3.28	3.28	3.19	3.37	3.37	3.07	3.31	3.16	3.03	3.18	.96	3.14	2.43*	3.11
		3.50	3.15	4.00	2.44	3.74	3.68	3.53	3.75	3.56	3.13	3.25	3.45	3.50	3.40	2.56	3.44
	2	3.60	3.62	4.11	3.70	4.71*	3.81	3.74	3.98	3.65	3.40	3.76	3.78	4.40	3.58	3.32	3.76
		3.40	3.61	4.46	3.78	3.96	3.76	3.92	3.44	3.82	3.52	3.81	3.89	3.33	3.70	3.61	~~3.22~~ 3.27
3	1	5.50	4.93	5.88	4.01	5.48	5.60	5.36	5.42	5.27	5.07	5.01	5.00	.00	5.01	3.99	5.14
		5.90	5.62	6.01	5.88	5.89	5.04	6.05	5.52	6.03	5.80	5.44	5.69	4.91	6.23	3.59*	5.69
	2	5.91	6.49	6.01	5.33	6.91	5.76	6.02	6.83	6.39	6.00	5.90	6.12	5.58	6.14	7.50	6.22 ~~6.53~~
		5.50	6.33	6.26	*	6.43	6.01	6.39	6.63	6.43	7.20	6.10	6.30	6.29	6.15	6.89	6.40
4	1	8.90	9.04	10.10	9.32	9.50	8.94	9.24	11.06	9.37	7.80	8.95	9.71	7.23	8.77	9.09	8.92
		9.10	9.87	10.90	11.26	9.09	10.52	8.76	12.03	10.51	8.15	9.79	10.85	10.75	9.63	9.59	9.86
	2	12.50	11.62	9.76	11.93	11.08	10.72	11.26	11.18	11.18	9.50	10.75	11.28	.00	11.05	13.27	10.79
		12.60	11.71	10.73	9.22	12.03	11.33	11.33	11.38	11.46	9.87	10.78	11.08	9.82	11.00	11.44	11.11
5	1	15.20	13.95	14.24	9.21*	12.33	13.84	13.58	14.98	13.06	13.80	13.47	14.25	9.68	13.59	15.33	13.12
		15.60	15.80	17.75	11.87	15.20	14.91	14.92	16.39	14.63	12.50	14.71	15.98	14.09	14.56	17.87	14.51
	2	16.60	16.21	19.62	14.94	17.29	17.77	17.91	17.09	14.94	17.25	16.77	17.31	18.06	16.37	13.60	15.87
		17.00	16.85	15.74	17.71	15.74	18.02	16.38	18.73	14.92	17.17	16.34	16.89	5.71	16.18	14.36	16.34

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XYLENE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	.23	.17	.13	.24	.10	.18	.19	.17	.18	.19	.18	.15	.00	.16	.17	.18
		.22	.21	.14	.27	.10	.20	.21	.20	.22	.22	.18	.19	.13	.19	.19	.20
	2	.26	.24	.24	.43	.10	.22	.21	.24	.20	.23	.24	.23	.23	.19	.18	.22
		.27	.25	.23	.43	.09	.22	.27	.25	.26	.24	.24	.24	2.23	.23	.28	.23
2	1	2.30	2.03	1.93	2.14	2.23	2.20	2.33	2.25	2.14	2.14	2.16	2.03	2.79	1.94	1.98	2.22
		2.90	2.44	2.57	2.56	2.42	2.48	2.17	2.57	2.42	2.36	2.46	2.56	3.00	2.24	2.26	2.46
	2	2.90	2.59	2.34	2.98	2.73	2.71	2.84	3.02	2.56	3.34	2.51	2.62	3.12	2.58	2.31	2.69
		2.90	2.65	2.93	2.94	2.76	2.83	2.68	2.94	2.60	3.40	2.73	2.62	2.72	2.58	2.41	2.77
3	1	4.40	3.89	3.23	3.83	4.20	4.29	4.15	3.91	4.02	5.10*	3.80	4.11	4.22	3.72	3.83	4.20
		5.10	4.38	4.11	4.31	4.81	4.66	4.27	3.82	4.43	5.81	4.42	4.61	4.66	4.23	4.45	4.64
	2	5.50	4.91	4.71	5.12	5.07	5.18	5.16	5.72	5.02	5.32	4.83	4.96	5.85	4.90	5.07	5.07
		5.60	5.06	6.09	5.01	5.26	5.35	5.62	4.65	5.15	5.04	5.19	5.17	5.06	5.08	5.09	5.22
4	1	6.50	5.54	6.72	6.88	5.93	6.08	5.49	6.01	5.26	11.38*	5.58	5.52	6.99	5.88	6.13	6.07
		7.50	6.71	7.36	7.64	7.40	6.67	6.51	7.12	5.78	8.83	6.47	6.41	5.34	6.60	7.12	6.71
	2	8.30	7.32	6.71	7.53	7.51	7.48	7.08	8.39	8.03	7.95	6.91	7.40	7.36	7.19	7.81	7.34
		8.30	7.38	8.18	7.20	7.44	7.74	7.18	7.96	7.91	8.06	7.20	7.68	9.94	7.47	7.90	7.56
5	1	7.90	7.06	7.43	7.94	7.51	7.01	7.21	7.15	7.97	8.33	6.66	7.32	9.50	7.13	6.86	7.29
		8.90	8.14	7.70	8.31	8.60	8.40	8.02	9.23	8.63	8.22	7.95	8.10	9.06	8.07	8.24	8.06
	2	9.60	8.98	9.75	9.09	9.72	9.02	7.88	9.25	8.89	10.96	8.68	9.04	10.62	8.41	8.51	8.81
		9.80	8.78	9.88	9.07	8.87	9.42	8.21	9.61	9.01	10.96	8.97	9.11	11.68	8.36	9.12	9.07

DATE 06/29/73

BENZENE (MIXTURE) (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	.33	.33	.31	.31	.32	.28	.30	.29	.30	.29	.31	.31	.26	.29	.22*	.30
		.35	.35	.33	.33	.33	.32	.31	.31	.33	.32	.36	.33	.29	.33	.23*	.33
	2	.37	.39	.39	.38	.35	.34	.36	.33	.37	.36	.34	.34	.37	.35	.31	.37
		.35	.41	.38	.40	.41	.36	.38	.38	.40	.40	.37	.38	.37	.38	.33	.38

DATE 06/29/73

XYLENE (MIXTURE) (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	3.80	3.49	3.70	3.75	3.99	3.69	3.87	3.84	3.92	4.44	3.65	3.46	3.82	3.49	3.48	3.89
		4.45	3.77	3.83	4.03	4.10	3.93	3.85	4.02	4.17	5.02	4.08	3.91	4.49	3.90	3.66	4.30
	2	4.80	4.25	4.33	4.55	4.52	4.49	4.18	4.31	4.49	5.04	4.53	4.54	4.59	4.15	4.18	4.70
		4.90	4.58	4.60	4.80	5.18	4.70	4.65	4.91	4.64	5.65	4.58	4.81	4.13	4.44	4.47	4.84

DATE 06/29/73

ETHYLENE DICHLORIDE (MIXTURE) (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	1.50	1.93	1.75	2.78	1.93	1.94	1.87	1.94	1.84	1.96	1.94	2.01	4.05	1.84	1.87	1.92
		2.00	2.10	1.46	2.72	1.91	2.11	1.98	2.12	1.97	2.08	2.07	2.19	4.13	1.97	1.99	2.13
	2	2.20	2.19	10.50	2.73	2.22	2.56	2.10	2.22	2.19	2.43	2.17	2.38	4.57	2.09	1.77	2.32
		2.00	2.41	12.69	2.68	2.46	2.73	2.42	2.43	2.38	2.63	2.39	2.45	5.49	2.29	1.74	2.39

DATE 06/29/73

TRICHLOROETHYLENE (MIXTURE) (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13*	LAB14	LAB15	SCOTT
1	1	4.90	4.34	5.18	4.44	4.96	4.69	4.96	4.94	4.87	4.25	4.86	4.60	5.16	4.73	4.42	4.91
		5.50	4.73	5.37	4.35	5.00	4.98	5.04	5.07	5.19	4.25	5.08	5.18	6.16	5.09	4.69	5.43
	2	6.30	5.64	4.35	4.79	6.24	6.19	5.64	5.82	5.79	5.90	5.46	5.69	2.75	5.60	4.19	5.93
		5.80	5.82	4.67	4.72	6.83	6.60	6.68	6.34	6.31	6.42	6.11	5.84	3.50	5.94	3.75	6.11

TABLE B-4

YOUDEN ANALYSIS ON DATA FOR EACH

PAIR AT EACH LEVEL (PHASE I)

DATE 06/08/73

BENZENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	PRECISION S_r^2	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.0152	.0162	.0014	.539-05	.309-04	12.45	13	15.263	-.63
	2	.0176	.0189	.0013	.414-05	.161-04	8.86	13	11.540	-1.18
2	1	.1847	.1874	.0067	.239-03	.251-03	3.10	13	8.546	-1.30
	2	.2475	.2132	.0147	.153-03	.384-04	1.50	11	5.966	-3.46
3	1	.3038	.3049	.0062	.858-04	.761-03	18.74	12	3.050	-.78
	2	.3557	.3613	.0056	.177-03	.191-02	22.59	13	3.737	-.47
4	1	.5971	.5861	-.0110	.947-03	.772-03	2.63	12	5.154	1.12
	2	.6912	.6532	-.0380	.420-02	.840-03	1.40	12	9.379	.53
5	1	1.5134	1.4685	-.0449	.801-02	.190-01	5.76	13	5.912	1.11
	2	1.7626	1.7118	-.0508	.197-01	.161-01	2.63	12	7.969	1.14

DATE 06/29/73

CARBON TETRACHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	s_r^2 PRECISION	SYSTEMATIC ERROR s_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.0447	.0256	-.0191	.131-03	.875-03	14.34	13	25.624	2.33
	2	.0365	.0291	-.0073	.181-04	.449-03	58.57	13	11.668	1.28
2	1	.4562	.3950	-.0611	.680-03	.141-02	5.13	12	5.717	5.28
	2	.5109	.4606	-.0503	.438-02	.416-03	1.19	11	12.961	3.41
3	1	.6816	.6427	-.0389	.546-02	.568-02	3.08	12	10.845	1.53
	2	.8453	.7510	-.0942	.113-02	.542-02	10.59	13	3.979	4.55
4	1	1.4486	1.3456	-.1030	.236-01	.617-02	1.52	13	10.606	2.87
	2	1.6699	1.5687	-.1012	.798-02	.534-01	14.38	12	5.351	1.52
5	1	14.5341	14.4951	-.0390	.133+01	.152+01	3.30	11	7.927	.09
	2	17.5929	16.8982	-.6946	.187+01	.668+01	8.08	13	7.763	.95

DATE 06/08/73

CHLOROFORM

ANALYSIS SUMMARY (MG)

LEVEL	PATH	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.1409	.1399	.0010	.786-04	.194-03	6.45	10	8.063	-.01
	2	.1444	.1281	-.0163	.479-03	.152-02	7.35	13	15.149	1.45
2	1	1.4742	1.4421	-.0321	.218-02	.997-02	10.50	12	3.116	.96
	2	1.7383	1.6807	-.0575	.211-01	.324-01	4.08	13	8.347	1.04
3	1	2.3611	2.4444	.0833	.940-02	.364-01	8.73	13	4.107	-1.53
	2	2.8678	2.8489	-.0189	.428-01	.240-01	2.12	12	7.214	.32
4	1	4.4476	4.4781	.0305	.565-01	.937-02	1.33	13	5.346	-.59
	2	5.2543	5.2200	-.0343	.597-01	.505+00	17.92	13	4.649	.18

DATE 06/08/73

DIOXANE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.1928	.2109	.0181	.128-03	.768-02	120.58	12	5.880	-.74
	2	.2260	.2461	.0202	.147-02	.924-02	13.60	12	16.948	-.73
2	1	1.9732	2.0171	.0439	.374-02	.299-01	16.97	12	3.101	-.89
	2	2.3379	2.3512	.0134	.473-02	.290-01	13.25	12	2.942	-.27
3	1	3.6349	3.5931	-.0418	.633-01	.955-01	4.02	12	6.919	.42
	2	4.2047	4.1883	-.0164	.827-02	.539-01	14.04	11	2.162	.24
4	1	5.3877	5.4727	.0851	.897-02	.922-01	21.56	10	1.758	-.91
	2	6.1989	6.3793	.1804	.190-01	.210+00	23.09	11	2.225	-1.33
5	1	6.4090	6.4774	.0684	.982-02	.148+00	31.24	11	1.546	-.60
	2	7.6953	7.5504	-.1449	.236+00	.187+00	2.58	12	6.308	.95

DATE 06/08/73

ETHYLENE DICHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.4966	.4995	-.0071	.154-04	.683-04	9.63	11	4.119	2.82
	2	.1146	.1443	-.0122	.485-04	.816-04	4.36	11	5.975	4.13
2	1	1.4147	1.2787	-.1359	.242-02	.138-01	12.41	13	3.475	4.15
	2	1.6486	1.5119	-.1367	.167-02	.879-02	11.54	12	2.478	5.02
3	1	1.9397	1.9449	.0512	.468-02	.522-02	3.23	12	3.527	-2.12
	2	2.3139	2.3207	.0068	.122-01	.159-01	3.61	12	4.769	-.17
4	1	3.6648	3.5538	-.1349	.120+00	.751-01	2.25	11	9.456	.95
	2	4.2223	4.1873	-.0349	.686-01	-.111-01	.68	13	6.203	.86
5	1	7.1763	7.0917	.6854	.302-01	.974-01	7.44	11	2.423	-7.08
	2	8.5174	8.9488	.4310	.312+00	.320+00	3.05	13	6.555	-2.34

DATE 06/08/73

TRICHLOROETHYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.1990	.1681	-.0309	.114-02	.642-02	12.25	13	16.982	1.38
	2	.1751	.1959	.0209	.233-02	.463-02	4.98	13	27.550	-1.03
2	1	3.3142	3.2715	-.0387	.746-01	.122-01	1.33	12	8.251	.63
	2	3.7228	3.4885	-.2343	.255-01	.331-01	3.60	12	4.288	3.95
3	1	5.5258	5.4147	-.1111	.109+00	.207-01	1.38	12	5.979	1.46
	2	6.3309	6.4672	.1363	.108+00	.105+00	2.96	12	5.179	-1.23
4	1	9.6374	9.3921	-.2452	.225+00	.611+00	6.44	13	4.918	1.08
	2	11.1796	10.9480	-.2316	.506+00	.313+00	2.24	13	6.361	1.15
5	1	14.7102	13.8142	-.8960	.701+00	.651+00	2.86	12	5.692	3.23
	2	16.6329	16.1027	-.5301	.121+01	.575+00	1.95	13	6.626	1.82

DATE 06/08/73

XYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.1853	.1919	.0067	.125-03	.129-02	21.63	13	6.036	-.68
	2	.2402	.2237	-.0164	.549-03	.419-02	16.26	13	9.758	.92
2	1	2.2933	2.3395	.0463	.193-01	.554-02	1.57	13	6.054	-1.41
	2	2.7495	2.7271	-.0223	.149-01	.524-01	8.02	13	4.444	.34
3	1	4.1320	4.4176	.2856	.304-01	.661-01	5.35	12	4.159	-2.85
	2	5.1732	5.1494	-.0237	.132+00	-.364-01	.45	13	7.014	.52
4	1	6.4161	6.3901	-.0260	.349-01	.235+00	14.43	12	2.912	.19
	2	7.6130	7.4499	-.1631	.973-01	.100+00	3.07	13	4.097	1.58
5	1	7.8579	7.6744	-.1835	.149+00	.495-01	1.67	13	4.910	1.99
	2	9.1773	8.9411	-.2362	.603-01	.454+00	16.07	13	2.675	1.27

DATE 06/08/73

BENZENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.3183	.3187	.0004	.723-04	.153-03	5.23	12	2.671	-.09
	2	.3688	.3715	.0028	.163-03	.335-03	4.66	13	3.668	-.51

DATE 06/08/73

XYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	3.9030	4.0935	.1905	.173-01	.745-01	9.60	13	3.373	-2.47
	2	4.6165	4.7717	.1552	.182-01	.637-01	7.99	13	2.923	-2.15

DATE 06/08/73

ETHYLENE DICHLORIDE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	1.9348	2.0242	.0895	.145-01	.102-01	2.41	12	6.230	-2.44
	2	2.3181	2.3595	.0415	.108-01	.528-01	10.81	12	4.477	-.62

DATE 06/08/73

TRICHLOROETHYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	4.8454	5.1655	.3211	.207-01	.796-01	8.69	13	2.971	-4.01
	2	5.6934	6.0224	.3245	.871-01	.519+00	12.91	13	5.183	-1.64

TABLE B-5

YOUDEN ANALYSIS ON DATA AT EACH

LEVEL (REPLICATES AVERAGED) (PHASE I)

DATE 06/18/73

BENZENE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.0164	.0175	.0012	.148-04	.111-04	2.50	13	23.487	-1.01
2	.1957	.2028	.0071	.120-03	.573-04	1.95	11	5.607	-2.27
3	.3289	.3356	.0068	.107-02	.391-03	1.73	12	9.943	-.80
4	.6521	.6347	-.0174	.106-02	.696-04	1.13	11	4.992	2.46
5	1.6501	1.5902	-.0599	.427-02	.168-01	8.86	12	3.960	1.57

DATE 06/29/73

CARBON TETRACHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.0406	.0274	-.0132	.544-03	.155-03	1.57	13	57.463	2.39
2	.4887	.4278	-.0609	.715-03	.986-03	3.76	10	5.471	5.51
3	.7615	.6969	-.0646	.610-02	.123-02	1.40	12	10.259	3.56
4	1.5548	1.4571	-.0976	.137-01	.242-01	4.54	12	7.519	2.00
5	16.2259	15.6967	-.5293	.245+01	.248+01	3.02	11	9.648	.95

DATE 06/18/73

CHLOROFORM

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.1286	.1190	-.0096	.814-03	.355-04	1.09	10	22.171	1.51
2	1.5922	1.5614	-.0308	.215-01	.290-02	1.27	12	9.211	.95
3	2.6814	2.6465	.0451	.331-01	.666-02	1.40	12	6.993	-1.07
4	4.8510	4.8491	-.0019	.273+00	.135-01	1.10	13	10.764	.02

DATE 06/18/73

DIOXANE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.2094	.2285	.0191	.669-03	.819-02	25.50	12	12.350	-.75
2	2.1556	2.1842	.0286	.757-02	.240-01	7.34	12	4.036	-.62
3	3.9070	3.8907	-.0163	.261-01	.675-01	6.17	11	4.139	.20
4	5.7978	5.9260	.1283	.302-01	.673-01	5.46	9	2.997	-1.41
5	7.0002	7.0139	.0137	.897-01	.763-01	2.70	11	4.279	-.14

DATE 06/18/73

ETHYLENE DICHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.1050	.0969	-.0080	.374-04	.274-04	2.47	10	5.825	3.93
2	1.5207	1.3953	-.1253	.339-02	.590-02	4.48	12	3.828	5.19
3	2.1190	2.1558	.0369	.734-02	.791-02	3.15	11	4.044	-1.19
4	3.9456	3.8756	-.0700	.612-01	.175-01	1.57	11	6.267	1.11
5	7.7918	8.4053	.6135	.132+00	.156+00	3.36	11	4.664	-4.51

DATE 06/18/73

TRICHLOROETHYLENE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.1870	.1820	-.0050	.751-02	-.112-02	.70	13	46.340	.36
2	3.5208	3.3800	-.1408	.239-01	.237-01	2.99	11	4.390	2.58
3	5.9165	5.9410	.0244	.928-01	-.184-01	.60	11	5.148	-.51
4	10.4085	10.1701	-.2384	.632+00	.126-01	1.04	13	7.639	1.56
5	15.6834	14.9585	-.7249	.129+01	-.153+00	.76	12	7.243	3.73

DATE 06/18/73

XYLENE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.2127	.2078	-.0049	.857-03	.205-02	5.79	13	13.761	.37
2	2.5214	2.5333	.0120	.243-01	.132-01	2.09	13	6.180	-.28
3	4.6823	4.7835	.1012	.419-01	.147-01	1.70	12	4.371	-1.93
4	6.9995	6.9200	-.0795	.163+00	.369-01	1.45	12	5.767	.83
5	8.5176	8.3058	-.2118	.174+00	.130+00	2.50	13	4.893	1.70

DATE 06/18/73

BENZENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	s_r^2 PRECISION	SYSTEMATIC ERROR s_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.3455	.3451	-.0003	.162-03	.530-04	1.65	12	3.684	.10

DATE 06/18/73

XYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	4.2547	4.4326	.1729	.711-02	.708-01	20.91	13	1.980	-2.37

DATE 06/18/73

ETHYLENE DICHLORIDE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	2.1240	2.1919	.0679	.176-01	.109-01	2.24	11	6.254	-1.67

TRICHLOROETHYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	5.2696	5.5944	.3248	.282+00	.446-01	1.32	13	10.069	-2.82

TABLE B-6

YOUDEN ANALYSIS ON LOG-TRANSFORMED

DATA FOR EACH PAIR AT EACH LEVEL (PHASE I)

DATE 06/15/73

BENZENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-4.2994	-4.1240	.1754	.236+00	.151+00	2.28	13	-11.299	-1.27
	2	-4.0698	-3.9687	.1011	.127-01	.442-01	7.93	13	-2.774	-1.68
2	1	-1.7203	-1.6758	.0446	.568-02	.900-02	4.17	13	-4.382	-1.53
	2	-1.5747	-1.5222	.0525	.417-02	.799-03	1.38	11	-4.103	-3.39
3	1	-1.1969	-1.1726	.0243	.861-03	.878-02	21.38	12	-2.452	-.91
	2	-1.0418	-1.0180	.0238	.151-02	.165-01	22.85	13	-3.729	-.68
4	1	-.5190	-.5355	-.0165	.258-02	.208-02	2.61	12	-9.793	1.03
	2	-.3743	-.3810	-.0067	.836-02	.258-02	1.62	12	-24.423	.29
5	1	.4068	.3830	-.0238	.289-02	.907-02	7.28	13	13.212	.87
	2	.5613	.5375	-.0239	.569-02	.605-02	3.13	12	13.438	.91

DATE 06/29/73

CARBON TETRACHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-3.9477	-3.6665	.2812	.553-01	.512+01	186.31	13	-5.955	-.46
	2	-4.0233	-3.5361	.4872	.148+00	.409+01	56.12	13	-9.578	-.89
2	1	-.7906	-.9300	-.1394	.338-02	.660-02	4.90	12	-7.354	5.52
	2	-.6852	-.7753	-.0901	.313-01	.570-03	1.04	11	-25.838	2.45
3	1	-.3971	-.4433	-.0461	.135-01	.160-01	3.36	12	-29.305	1.10
	2	-.1724	-.2864	-.1140	.143-02	.787-02	11.97	13	-21.967	4.60
4	1	.3609	.2956	-.0654	.120-01	.325-02	1.54	13	30.361	2.54
	2	.5018	.4501	-.0516	.331-02	.211-01	13.73	12	11.465	1.24
5	1	2.6700	2.6726	.0026	.664-02	.575-02	2.73	11	3.052	-.09
	2	2.8550	2.8271	-.0279	.586-02	.200-01	7.84	13	2.682	.69

DATE 06/15/73

CHLOROFORM

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-2.2184	-2.2090	.0094	.651-02	.163-01	6.01	10	-3.636	-.22
	2	-1.9825	-2.0547	-.0721	.249-01	.817-01	7.55	13	-7.963	.88
2	1	.3825	.3648	-.0177	.972-03	.454-02	10.34	12	8.152	.90
	2	.5435	.5191	-.0244	.657-02	.133-01	5.04	13	14.918	.71
3	1	.8537	.8924	.0387	.266-02	.675-02	6.08	13	6.043	-1.61
	2	1.0482	1.0468	-.0014	.455-02	.296-02	2.30	12	6.434	.07
4	1	1.4892	1.4980	.0087	.293-02	.472-03	1.32	13	3.634	-.74
	2	1.6484	1.6524	.0040	.218-02	.197-01	19.09	13	2.834	-.10

DATE 06/15/73

DIOXANE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-2.1899	-1.5577	.6322	.357-02	.451+01	2527.61	12	-2.728	-1.07
	2	-2.0595	-1.4019	.6576	.368-01	.470+01	256.67	12	-9.315	-1.09
2	1	.6732	.7004	.0272	.156-02	.801-02	11.28	12	5.864	-1.05
	2	.8464	.8548	.0085	.802-03	.541-02	14.50	12	3.345	-.40
3	1	1.2819	1.2778	-.0042	.394-02	.682-02	4.46	12	4.899	.16
	2	1.4345	1.4322	-.0023	.454-03	.296-02	14.05	11	1.485	.14
4	1	1.6800	1.6985	.0185	.292-03	.314-02	22.50	10	1.018	-1.07
	2	1.8216	1.8530	.0314	.409-03	.569-02	28.88	11	1.110	-1.42
5	1	1.8546	1.8671	.0131	.253-03	.366-02	29.98	11	.858	-.74
	2	2.0374	2.0215	-.0159	.390-02	.282-02	2.45	12	3.866	.83

DATE 06/15/73

ETHYLENE DICHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-2.3429	-2.4148	-.0719	.284-02	.691-02	7.77	11	-1.929	2.80
	2	-2.1535	-2.2601	-.1066	.373-02	.610-02	4.28	11	-2.835	4.14
2	1	.3414	.2446	-.0968	.136-02	.621-02	10.16	13	10.783	4.36
	2	.4981	.4130	-.0852	.628-03	.319-02	11.14	12	5.032	5.19
3	1	.6685	.6874	.0269	.124-02	.140-02	3.26	12	5.340	-2.15
	2	.8360	.8418	.0058	.223-02	.330-02	3.96	12	5.651	-.32
4	1	1.2910	1.2696	-.0214	.123-01	.514-02	1.83	11	8.601	.70
	2	1.4381	1.4320	-.0061	.385-02	-.640-03	.67	13	4.316	.64
5	1	1.9689	2.0608	.0918	.642-03	.191-02	6.94	11	1.287	6.74
	2	2.1378	2.1908	.0529	.481-02	.510-02	3.55	13	2.961	4.35

DATE 06/15/73

TRICHLOROETHYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-1.9127	-1.7844	.1283	.190+01	.222+00	1.23	13	-72.140	-.44
	2	-2.4944	-1.6300	.8644	.222+01	.370+01	4.33	13	-59.788	-1.47
2	1	1.1925	1.1840	-.0086	.771-02	.104-02	1.27	12	7.363	.44
	2	1.3126	1.2465	-.0661	.182-02	.221-02	3.43	12	3.252	4.26
3	1	1.7054	1.6879	-.0175	.447-02	.654-03	1.29	12	3.920	1.17
	2	1.8431	1.8667	.0236	.251-02	.258-02	3.05	12	2.720	-1.38
4	1	2.2689	2.2386	-.0222	.227-02	.648-02	6.71	13	2.107	.95
	2	2.4110	2.3930	-.0179	.422-02	.252-02	2.19	13	2.694	.99
5	1	2.6845	2.6244	-.0600	.322-02	.289-02	2.79	12	2.114	3.23
	2	2.8383	2.7789	-.0294	.419-02	.249-02	2.19	13	2.304	1.63

DATE 06/15/73

XYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-1.7095	-1.6518	.0578	.359-02	.478-01	27.64	13	-3.506	-.97
	2	-1.4671	-1.4973	-.0302	.113-01	.816-01	15.40	13	-7.255	.38
2	1	.8253	.8487	.0234	.352-02	.988-03	1.56	13	7.190	-1.67
	2	1.0073	1.0032	-.0042	.213-02	.638-02	6.98	13	4.587	.18
3	1	1.4290	1.4843	.0554	.198-02	.373-02	4.77	12	3.112	-2.91
	2	1.6418	1.6388	-.0030	.474-02	-.135-02	.43	13	4.193	.36
4	1	1.8533	1.8535	.0002	.833-03	.575-02	14.80	12	1.557	-.01
	2	2.0282	2.0081	-.0201	.174-02	.168-02	2.94	13	2.055	1.49
5	1	2.0583	2.0361	-.0221	.241-02	.817-03	1.68	13	2.387	1.84
	2	2.2140	2.1906	-.0234	.730-03	.509-02	14.94	13	1.220	1.19

DATE 06/15/73

BENZENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-1.1464	-1.1448	.0016	.721-03	.150-02	5.17	12	-2.343	-.14
	2	-1.0000	-.9902	.0098	.136-02	.262-02	4.83	13	-3.694	-.64

DATE 06/15/73

XYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	1.3584	1.4081	.0497	.995-03	.440-02	9.83	13	2.323	-2.66
	2	1.5273	1.5626	.0353	.762-03	.278-02	8.31	13	1.808	-2.34

DATE 06/15/73

ETHYLENE DICHLORIDE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.6561	.7039	.0479	.496-02	.293-02	2.18	12	10.735	-2.35
	2	.8346	.8584	.0238	.215-02	.107-01	10.99	12	5.552	-.79

DATE 06/15/73

TRICHLOROETHYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	1.5757	1.6409	.0652	.861-03	.356-02	9.27	13	1.862	-3.86
	2	1.7293	1.7954	.0661	.278-02	.196-01	15.06	13	3.051	-1.71

TABLE B-7

YOUDEN ANALYSIS ON LOG-TRANSFORMED DATA

AT EACH LEVEL (REPLICATES AVERAGED) (PHASE I)

DATE 06/25/73

BENZENE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	-4.1846	-4.0463	.1382	.130+00	.301-01	1.46	13	-8.602	-1.68
2	-1.6377	-1.5990	.0387	.281-02	.190-02	2.35	11	-3.237	-2.33
3	-1.1220	-1.0953	.0267	.899-02	.474-02	2.05	12	-8.451	-1.00
4	-.4338	-.4583	-.0245	.250-02	.689-04	1.06	11	-11.525	2.34
5	.4928	.4602	-.0325	.145-02	.639-02	9.81	12	7.731	1.39

DATE 06/29/73

CARBON TETRACHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	-3.9855	-3.6013	.3842	.283+00	.437+01	31.96	13	-13.338	-.68
2	-.7227	-.8527	-.1299	.301-02	.438-02	3.91	10	-7.591	5.62
3	-.2871	-.3648	-.0777	.135-01	.237-02	1.35	12	-48.432	2.94
4	.4287	.3729	-.0558	.410-02	.121-01	6.89	12	14.931	1.69
5	2.7717	2.7498	-.0219	.719-02	.874-02	3.43	11	3.059	.68

DATE 06/25/73

CHLOROFORM

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	-2.0826	-2.1318	-.0493	.390-01	.490-02	1.25	10	-9.477	1.05
2	.4564	.4420	-.0144	.954-02	.621-03	1.13	12	21.404	.71
3	.9456	.9696	.0240	.547-02	.662-03	1.24	12	7.820	-1.48
4	1.5688	1.5752	.0064	.109-01	.510-03	1.09	13	6.647	-.31

DATE 06/25/73

DIOXANE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	-2.1247	-1.4798	.6449	.238-01	.459+01	387.23	12	-7.258	-1.00
2	.7598	.7776	.0178	.177-02	.553-02	7.27	12	5.530	-.80
3	1.3547	1.3550	.0003	.183-02	.421-02	5.60	11	3.156	-.01
4	1.7515	1.7757	.0242	.741-03	.193-02	6.20	9	1.554	-1.60
5	1.9393	1.9443	.0049	.192-02	.142-02	2.48	11	2.262	-.35

DATE 06/25/73

ETHYLENE DICHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	-2.2630	-2.3374	-.0745	.340-02	.243-02	2.43	10	-2.575	3.85
2	.4125	.3288	-.0837	.147-02	.240-02	4.28	12	9.286	5.39
3	.7444	.7646	.0202	.146-02	.184-02	3.53	11	5.128	-1.38
4	1.3650	1.3508	-.0142	.528-02	.990-03	1.38	11	5.323	.82
5	2.0471	2.1258	.0787	.188-02	.277-02	3.94	11	2.120	-4.47

DATE 06/25/73

TRICHLOROETHYLENE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	-2.2035	-1.7072	.4963	.357+01	-.572+00	.68	13	-85.691	-1.69
2	1.2535	1.2152	-.0383	.238-02	.173-02	2.51	11	3.823	2.47
3	1.7740	1.7773	.0032	.259-02	-.518-03	.60	11	2.868	-.40
4	2.3359	2.3158	-.0201	.570-02	.420-03	1.15	13	3.233	1.31
5	2.7471	2.7017	-.0455	.544-02	-.729-03	.73	12	2.685	3.67

DATE 06/25/73

XYLENE

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	-1.5883	-1.5746	.0138	.113-01	.572-01	11.16	13	-6.678	-.21
2	.9163	.9259	.0096	.287-02	.223-02	2.56	13	5.842	-.59
3	1.5353	1.5616	.0263	.221-02	.698-03	1.63	12	3.062	-2.23
4	1.9388	1.9308	-.0080	.365-02	.695-03	1.38	12	3.116	.57
5	2.1361	2.1133	-.0228	.196-02	.178-02	2.82	13	2.070	1.62

DATE 06/25/73

BENZENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	-1.0677	-1.0675	.0002	.133-02	.477-03	1.72	12	-3.417	-.03

DATE 06/25/73

XYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1.4429	1.4854	.0425	.494-03	.353-02	15.30	13	1.541	-2.58

DATE 06/25/73

ETHYLENE DICHLORIDE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	.7464	.7812	.0347	.384-02	.256-02	2.33	11	8.302	-1.88

DATE 06/25/73

TRICHLOROETHYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1.6525	1.7182	.0657	.106-01	.187-02	1.35	13	6.233	-2.90

APPENDIX C

PHASE II SCHEDULE, DATA AND DATA ANALYSIS

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

Laboratory No. ____ Day No. _A__

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx050414	Ethylene Dichloride
2	xx050113	"
3	xx050100**	"
4	xx050112	"
5	xx050313	"
6	xx050415	"
7	xx050500**	"
8	xx050312	"
9	xx090112	Ethylene Dichloride & Trichloroethylene
10	xx090113*	"
11	xx060413	Trichloroethylene
12	xx060115	"
13	xx060512	"
14	xx060412	"
15	xx060513*	"
16	xx060100**	"
17	xx060114	

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. B

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx050513	Ethylene Dichloride
2	xx050115	"
3	xx050114	"
4	xx050214	"
5	xx050215	"
6	xx050512*	"
7	xx090100**	Ethylene Dichloride & Trichloroethylene
8	xx090115	"
9	xx090114*	"
10	xx060500**	Trichloroethylene
11	xx060214	"
12	xx060515	"
13	xx060215	"
14	xx060312	"
15	xx060514*	"
16	xx060313	

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. C

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx050315	Ethylene Dichloride
2	xx050514	"
3	xx050300**	"
4	xx050213	"
5	xx050314	"
6	xx050212	"
7	xx050413	"
8	xx050515*	"
9	xx050412	"
10	xx030313	Chloroform
11	xx030114	"
12	xx030212	"
13	xx030415*	"
14	xx030115	"
15	xx030312	"
16	xx030213	"
17	xx030414	"
18	xx030400**	"

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES
(continued)

Laboratory No. ____ Day No. D

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx010300**	Benzene
2	xx010315	"
3	xx010413	"
4	xx010114	"
5	xx010314	"
6	xx010212	"
7	xx010115	"
8	xx010412	"
9	xx010213	"
10	xx040215	Dioxane
11	xx040512	"
12	xx040100**	"
13	xx040315	"
14	xx040214	"
15	xx040513*	"
16	xx040415	"
17	xx040414	"
18	xx040314	"

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES
(continued)

Laboratory No. ____ Day No. E

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx070215	Xylene
2	xx070412	"
3	xx070513	"
4	xx070112	"
5	xx070214	"
6	xx070113	"
7	xx070512*	"
8	xx070413	"
9	xx070500**	"
10	xx020100**	Carbon Tetrachloride
11	xx020414	"
12	xx020312	"
13	xx020115	"
14	xx020513	"
15	xx020415	"
16	xx020313	"
17	xx020114	"
18	xx020512*	"

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES
(continued)

Laboratory No. ____ Day No. F

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx010215	Benzene
2	xx010414	"
3	xx010214	"
4	xx010512	"
5	xx010415	"
6	xx010513*	"
7	xx010500**	"
8	xx080114	Benzene & Xylene
9	xx080100**	"
10	xx080115*	"
11	xx070114	Xylene
12	xx070515*	"
13	xx070315	"
14	xx070115	"
15	xx070514	"
16	xx070314	"

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. G

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx010312	Benzene
2	xx010112	"
3	xx010515	"
4	xx010100**	"
5	xx010313	"
6	xx010514*	"
7	xx010113	"
8	xx080113	Benzene & Xylene
9	xx080112*	"
10	xx070212	Xylene
11	xx070313	"
12	xx070100**	"
13	xx070415	"
14	xx070300**	"
15	xx070213	"
16	xx070414	"
17	xx070312	"

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES
(continued)

Laboratory No. ____ Day No. H

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx060112	Trichloroethylene
2	xx060212	"
3	xx060314	"
4	xx060415	"
5	xx060315	"
6	xx060113	"
7	xx060414	"
8	xx060300**	"
9	xx060213	"
10	xx040413	Dioxane
11	xx040500**	"
12	xx040312	"
13	xx040515*	"
14	xx040115	"
15	xx040313	"
16	xx040514	"
17	xx040412	"
18	xx040114	"

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ____ Day No. I

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx020300**	Carbon Tetrachloride
2	xx020413	"
3	xx020112	"
4	xx020213	"
5	xx020514*	"
6	xx020412	"
7	xx020212	"
8	xx020113	"
9	xx020515	"
10	xx030200**	Chloroform
11	xx030214	"
12	xx030315	"
13	xx030112	"
14	xx030412*	"
15	xx030314	"
16	xx030113	"
17	xx030413	"
18	xx030215	"

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-1

SCHEDULE FOR ANALYSIS OF CHARCOAL TUBES

(continued)

Laboratory No. ___ Day No. J

Order of Analysis	Charcoal Tubes Code Number	Compound(s) to be Determined
1	xx040112	Dioxane
2	xx040213	"
3	xx040300**	"
4	xx040113	"
5	xx040212	"
6	xx020315	Carbon Tetrachloride
7	xx020214	"
8	xx020314	"
9	xx020215	"
10	xx020500**	"
11	xx	
12	xx	
13	xx	
14	xx	
15	xx	
16	xx	

* The back-up charcoal is to be analyzed in these tubes.

** Spare charcoal from Phase I.

NOTE: The 'xx' on the charcoal tube code numbers refers to your laboratory code number shown above

TABLE C-2 SCHEDULE OF SAMPLING DAYS FOR 15 LABORATORIES IN PHASE II

Lab	A	B	C	D	E	F	G	H	I	J
1	5	8	9	2	1	3	6	10	4	7
2	5	1	9	3	6	10	2	8	7	4
3	6	2	4	1	3	8	10	7	5	9
4	6	2	7	3	9	8	4	5	10	1
5	9	4	10	7	5	8	1	3	2	6
6	10	9	3	5	8	7	6	4	1	2
7	5	4	6	9	3	1	8	7	2	10
8	2	5	10	4	9	1	7	6	8	3
9	8	6	2	10	5	9	3	1	4	7
10	5	6	1	9	3	4	8	2	10	7
11	7	5	4	1	9	8	3	6	2	10
12	8	7	9	2	1	3	10	6	4	5
13	9	1	2	4	7	5	3	10	6	8
14	4	3	2	8	1	10	9	7	6	5
15	4	7	8	10	2	1	6	3	5	9

TABLE C-3

PHASE II ANALYTICAL RESULTS

DATE 07/09/73

BENZENE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	.02	.02	.01	.00	.02	.01	.01	.01	.02	.04	.02	.02	3.60*	.01	.01	.02
		.02	.02	.02	.00	.01	.01	.00	.01	.02	.05*	.01	.02	3.35*	.01	.01	.02
	2	.02	.02	.02	.02	.01	.01	.01	.01	.02	.05*	.02	.02	.03	.02	.02	.02
		.02	.02	.02	.02	.01	.01	.00*	.01	.02	.06*	.02	.02	.02	.02	.01	.02
2	1	.24	.20	.19	.20	.18	.19	.18	.21	.20	.23	.20	.22	.24	.19	.21	.20
		.20	.18	.20	.18	.14	.19	.19	.20	.22	.25	.20	.21	.11	.20	.11	.20
	2	.18	.20	.21	.18	.20	.19	.15	.19	.21	.13	.21	.20	.24	.18	.16	.20
		.16	.21	.17	.18	.19	.17	.15	.20	.20	.13	.21	.21	.25	.18	.16	.20
3	1	.40	.40	.34	.34	.39	.29	.00*	.32	.36	.40	.35	.32	.45	.34	.25	.33
		.42	.35	.35	.37	.32	.30	.26	.32	.38	.39	.36	.34	.51*	.36	.29	.33
	2	.36	.36	.36	.37	.37	.33	.31	.33	.38	.45	.33	.32	.45	.36	.37	.33
		.35	.36	.33	.36	.36	.32	.28	.32	.35	.48	.34	.35	.25	.36	.30	.33
4	1	.92	.64	.58	.67	.65	.63	.70	.60	.65	.92	.61	.65	.62	.64	.56	.63
		.81	.69	.54	.68	.65	.61	.67	.60	.63	.84	.64	.61	.66	.65	.54	.63
	2	.61	.62	.64	.64	.67	.54	.60	.60	.65	.51	.63	.60	.62	.61	.45	.63
		.55	.69	.60	.66	.65	.59	.64	.58	.50	.44	.61	.60	.67	.65	.49	.63
5	1	1.50	1.71	1.69	1.81	1.65	1.51	1.64	1.40	1.75	1.40	1.54	1.68	1.30	1.53	1.78	1.57
		1.20	1.77	1.48	1.82	1.67	1.59	1.43	1.56	1.75	1.30	1.56	1.56	1.23	1.58	1.64	1.57
	2	1.71	1.59	1.70	1.77	1.67	1.45	1.71	1.58	1.79	1.60	1.47	1.60	2.03	1.60	1.73	1.57
		1.84	1.65	1.40	2.06	1.81	1.48	1.77	1.61	1.77	1.60	1.49	1.62	1.65	1.52	1.60	1.57

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CARBON TETRACHLORIDE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	.11*	.04	.11*	.02	.06	.04	.04	.03	.04	.84*	.03	.04	.00	.03	.03	.03
		.14*	.09	.05	.02	.07	.03	.04	.02	.29*	.82*	.04	.04	.00	.03	.02	.03
	2	2.03*	.03	.03	.03	.06	.04	.04	.02	.03	1.23*	.04	.04	.00	.03	.02	.03
		2.33*	.08	.04	.00	.06	.04	.04	.03	.04	.90*	.03	.04	.00	.03	.02	.03
2	1	.43	.42	.50	.49	.43	.42	.54	.48	.50	.36	.50	.52	.49	.48	.41	.43
		.31	.44	.47	.52	.48	.39	.45	.50	.54	.95*	.45	.58	.45	.47	.37	.43
	2	.47	.50	.60	.52	.47	.45	.46	.43	.50	.55	.46	.52	.47	.34	.40	.43
		.47	.50	.51	.46	.47	.47	.46	.46	.49	.50	.48	.51	.41	.47	.43	.43
3	1	6.55*	.91	.79	.72	.70	.68	.77	.78	.82	1.23*	.75	.78	1.51*	.79	.53	.71
		10.02*	.92	.91	.85	.72	.68	.72	.79	.81	.95	.75	.82	1.67*	.77	.57	.71
	2	.94	.78	.77	.57	.73	.74	.78	.71	.80	.82	.77	.85	.63	.79	.59	.71
		.94	.81	.76	.80	.72	.66	.77	.72	.79	.94	.81	.88	.71	.78	.55	.71
4	1	1.98	1.81	1.14	1.35	1.61	1.62	1.59	1.63	1.61	.65*	1.54	1.64	1.05	1.42	2.58*	1.47
		.98	1.48	1.77	1.58	1.58	1.77	.05*	1.50	1.59	1.15	1.29	1.62	.97	1.46	1.34	1.47
	2	3.79*	1.68	1.69	1.92	1.39	1.61	1.61	1.49	1.64	1.07	1.22	1.59	1.84	1.52	1.08	1.47
		4.88*	1.57	1.73	1.79	1.44	1.57	1.52	1.51	1.75	1.18	1.29	1.57	3.99*	1.53	1.01	1.47
5	1	52.44*	18.38	15.69	19.02	13.69	17.56	13.68	16.87	16.15	25.80*	14.11	16.77	37.21*	14.60	12.49	15.65
		55.44*	18.20	14.24	17.94	14.21	18.40	13.12	15.62	15.54	26.90*	13.61	16.64	29.11*	15.07	15.05	15.65
	2	18.98	19.60	15.83	14.23	13.17	19.38	12.37	16.38	15.13	21.84	14.35	16.55	.78*	15.12	16.13	15.65
		14.19	19.80	17.69	14.71	11.59	18.93	10.71	16.12	16.79	19.60	13.58	16.56	13.72	14.71	14.73	15.65

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CHLOROFORM (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	.25 *	.15	.10	.10	.13	.11	.07	.13	.12	.16	.13	.14	.13	.13	.11	.12
		1.33 *	.14	.11	.10	.20	.12	.09	.13	.14	.16	.14	.12	.00 *	.12	.16	.12
	2	.19	.09	.10	.21	.20	.12	.12	.13	.13	.14	.13	.13	5.29 *	.12	.11	.12
		1.90 *	.13	.11	.20	.05	.12	.12	.12	.12	.12	.13	.14	15.08 *	.13	.11	.12
2	1	1.40	1.84	1.76	1.90	1.47	1.51	1.49	1.47	1.78	1.20	1.53	1.70	14.28 *	1.60	1.27	1.58
		1.50	1.37	1.79	2.04	1.45	1.51	1.60	1.61	1.80	1.80	1.60	1.64	31.21 *	.67 *	1.27	1.58
	2	2.20 *	1.89	1.69	1.92	1.62	1.44	1.49	1.64	1.60	1.72	1.51	1.69	1.33	1.66	1.84	1.58
		2.19 *	1.84	1.70	1.68	1.66	1.47	1.22	1.56	1.76	1.80	1.68	1.67	1.50	1.58	1.17	1.58
3	1	2.40	3.22	2.88	3.84 *	2.48	2.68	2.18	2.82	2.88	3.00	2.61	2.93	2.42	2.54	2.54	2.51
		2.80	3.11	2.87	3.37	2.21	2.57	2.26	2.14	2.68	3.10	2.51	2.97	7.16 *	2.58	2.27	2.51
	2	3.15	3.06	2.77	3.22	2.37	2.57	2.48	2.80	2.86	3.09	2.50	2.81	2.75	2.67	3.13	2.51
		4.83 *	3.03	2.80	2.81	2.51	2.51	2.35	2.52	2.92	3.09	2.52	2.82	2.34	2.74	2.57	2.51
4	1	8.85 *	5.16	5.26	5.48	4.78	5.17	5.29	4.34	4.77	5.29	5.13	5.22	4.63	4.91	6.64 *	4.91
		9.35 *	5.32	2.72	5.34	4.72	5.22	5.39	3.71	4.82	5.64	4.98	5.06	5.20	4.71	6.78 *	4.91
	2	3.70	5.31	5.91	6.17	4.63	5.32	5.84	3.84	5.12	5.10	4.96	5.50	4.05	4.34	4.11	4.91
		4.70	5.44	5.03	5.37	4.40	5.44	6.28	3.90	5.31	5.30	5.00	5.54	21.66 *	4.29	4.44	4.91

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DIOXANE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	.23	.19	.22	.34 *	.11	.19	.15	.20	.23	.29	.20	.19	.00 *	.16	.18	.23
		.23	.20	.22	.36 *	.20	.19	.15	.19	.23	.31	.21	.19	.00 *	.16	.17	.23
	2	.24	.19	.21	.54 *	.15	.19	.15	.19	.24	.21	.22	.21	.70 *	.19	.18	.23
		.24	.19	.18	.49 *	.05*	.19	.17	.21	.22	.23	.20	.19	.71 *	.18	.18	.23
2	1	2.40	1.97	2.24	1.94	2.40	1.74	1.51	2.06	2.67	2.38	1.92	2.07	1.76	1.75	1.65	2.18
		2.37	1.90	2.08	1.90	2.18	1.83	1.48	2.06	2.37	2.26	1.51	2.01	1.13	2.03	1.87	2.18
	2	1.90	1.90	2.32	2.39	1.95	2.10	1.61	2.03	2.47	2.83	1.84	1.88	3.39	1.90	1.21	2.18
		2.30	1.90	2.41	1.73	1.87	1.86	1.51	2.08	2.50	2.83	1.79	1.70	2.60	1.87	1.49	2.18
3	1	4.30	3.32	4.27	4.36	3.36	3.91	2.22*	3.86	4.70	4.76	3.43	3.61	3.74	3.61	3.27	3.91
		4.20	3.33	4.71	4.58	3.37	3.81	1.56*	3.80	4.44	3.96	3.60	3.96	3.29	3.46	3.03	3.91
	2	2.90	3.32	4.02	4.17	3.72	3.94	6.16*	3.68	4.28	4.19	3.91	3.62	7.50 *	3.02	2.90	3.91
		3.40	2.74	4.24	4.13	3.67	3.81	2.92	3.70	4.47	5.10	3.56	3.46	6.75 *	3.50	3.12	3.91
4	1	6.00	5.06	6.33	6.97	5.49	6.08	5.23	5.36	7.00	5.66	5.14	5.30	5.03 *	4.73	5.24	5.94
		6.50	5.21	5.78	6.06	5.35	6.00	5.62	5.47	6.58	6.12	5.29	5.43	4.00 *	4.70	5.63	5.94
	2	4.20	4.81	5.73	6.59	5.35	6.27	4.25	5.67	6.87	6.00	5.22	5.47	11.09*	4.63	4.88	5.94
		4.40	4.68	6.10	6.51	5.59	6.05	4.22	5.65	7.20	5.89	5.24	5.62	10.74*	5.00	4.83	5.94
5	1	6.40	6.00	6.93	6.20	6.42	6.37	6.07	6.11	7.57	7.81	6.73	6.83	8.91 *	5.51	6.72	7.04
		6.30	6.07	7.78	5.93	6.98	7.01	3.05*	6.00	7.27	8.15	6.68	7.33	11.15 *	5.41	6.59	7.04
	2	8.70	5.47	7.37	8.52	.00*	6.22	5.23	5.70	7.33	6.23	6.83	7.09	5.56	5.80	7.02	7.04
		8.10	6.29	7.48	7.18	6.38	7.46	5.18	6.10	7.43	8.15	6.70	7.44	5.56	5.91	6.32	7.04

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ETHYLENE DICHLORIDE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	.10	.14	.15	.58 *	.12	.09	.09	.10	.12	.09	.11	.11	.10	.09	.12	.10
		.10	.14	.16	.57 *	.13	.10	.09	.09	.12	.10	.12	.11	.09	.09	.10	.10
	2	.10	.14	.13	.14	.12	.09	.08	.10	.12	.12	.10	.10	13.97*	.10	.08	.10
		.11	.14	.12	.15	.12	.09	.10	.10	.11	.13	.11	.11	13.97*	.10	.09	.10
2	1	2.10*	1.50	1.59	1.34	1.29	1.53	.17 *	1.38	1.52	1.14	1.50	1.41	1.23	1.36	1.43	1.41
		1.90*	1.48	1.51	1.36	1.52	1.53	1.42	1.35	1.53	1.14	1.47	1.49	1.34	1.42	1.50	1.41
	2	2.00*	1.38	1.40	1.55	1.32	1.38	1.38	1.34	1.63	1.24	1.88	1.57	1.79	1.37	1.19	1.41
		2.00*	1.36	1.50	1.58	1.35	1.42	1.50	1.39	1.56	1.24	1.15	1.52	1.73	1.32	1.36	1.41
3	1	2.37	1.99	1.90	3.04*	2.08	2.36	1.88	2.15	2.25	1.86	2.29	2.19	1.68	2.13	2.18	2.19
		2.60	2.12	2.23	2.16	2.13	2.24	1.89	1.99	2.20	1.76	2.19	2.08	1.54	2.16	2.19	2.19
	2	2.50	2.05	2.10	1.93	2.21	2.30	1.94	2.13	2.47	1.34 *	2.20	2.26	1.62	2.09	2.18	2.19
		3.10	2.30	2.18	1.63	1.89	2.28	2.08	2.23	2.27	2.38	2.16	2.50	2.60	2.09	2.06	2.19
4	1	5.20	3.81	4.06	4.11	3.47	4.16	3.48	3.75	4.43	3.00	4.23	4.19	3.23	3.92	3.56	3.89
		5.10	3.89	4.22	4.16	3.94	4.25	3.57	3.71	4.62	2.79	4.11	4.47	3.30	4.08	3.83	3.89
	2	4.84	3.90	3.87	5.15	3.81	4.08	6.04 *	4.19	4.22	3.51	4.36	4.35	4.12	4.06	3.63	3.89
		4.49	3.89	3.82	5.23	3.36	4.15	2.28	3.95	4.37	3.00	4.09	4.46	3.58	4.06	3.68	3.89
5	1	9.20	7.86	7.09	10.68*	7.48	8.02	7.49	7.90	8.47	6.92	8.24	9.47	7.42	7.95	7.02	7.55
		8.80	8.30	8.30	10.47*	7.71	8.13	7.65	7.87	8.25	7.02	8.16	9.97	7.88	7.90	6.37	7.55
	2	23.00*	8.05	7.24	9.85	9.73	8.65	6.64	7.91	8.97	8.37	8.20	8.65	6.35	8.24	6.92	7.55
		11.60*	7.64	8.30	9.69	7.74	8.52	6.78	7.36	9.05	7.23	8.21	7.99	.63*	8.30	7.35	7.55

DATE 07/12/73

TRICHLOROETHYLENE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	.20	.23	.20	.49*	.11	.18	.17	.20	.19	.18	.20	.19	.08	.17	.12	.18
		.13	.20	.18	.51*	.15	.20	1.14*	.19	.17	.00*	.20	.16	.07*	.19	.19	.18
	2	.17	.29	.20	1.24*	.13	.15	.20	.18	.19	.18	.21	.17	.87*	.20	.33	.18
		.17	.30	.20	1.17*	.10	.15	.20	.19	.20	.18	.20	.18	.29	.20	.17	.18
2	1	5.10*	3.98	3.42	3.31	3.20	3.22	4.34	3.13	3.53	2.90	3.68	3.33	2.95	3.59	3.61	3.55
		2.70	3.50	3.46	3.26	3.46	3.34	4.23	2.98	3.71	2.70	3.61	3.44	3.54	3.57	3.77	3.55
	2	3.90	3.56	3.82	3.37	3.50	3.13	5.30*	3.11	3.55	2.90	3.76	3.02	1.18*	3.89	3.25	3.55
		3.70	3.32	4.52	3.43	4.28	3.37	.02*	3.03	3.38	3.30	3.39	2.30	.17*	3.56	3.63	3.55
3	1	6.40	6.41	7.02	5.69	5.69	5.48	5.82	6.04	5.98	5.30	5.77	5.61	.38*	5.48	6.33	5.84
		6.00	4.98	5.89	5.40	5.43	5.50	5.82	5.95	5.92	4.70	6.12	5.69	.46*	5.76	5.02	5.84
	2	9.00*	5.04	5.64	4.53	5.04	5.04	6.06	5.84	6.21	4.40	5.81	5.53	5.56	5.72	5.50	5.84
		7.10	5.39	5.80	4.19	4.54	5.36	7.25	5.54	6.71	4.50	5.81	5.61	5.73	5.65	5.61	5.84
4	1	11.15	9.08	12.20	11.73	9.50	10.61	16.80*	10.44	11.03	8.60	11.10	11.09	6.52*	10.14	8.49	10.11
		7.20*	8.82	10.70	10.53	10.34	10.30	16.67*	9.84	9.75	8.40	10.87	10.82	8.74*	9.82	9.13	10.11
	2	9.30	10.30	9.50	8.88	8.40	10.69	9.56	10.80	10.22	7.50	10.63	10.38	9.11	9.82	10.05	10.11
		15.40*	9.87	9.50	10.02	9.21	10.12	9.34	10.73	10.21	5.90*	10.64	10.72	12.88	9.67	10.27	10.11
5	1	16.24	13.73	13.12	17.84	16.35	15.39	18.64*	16.35	16.83	12.00	16.33	16.96	13.00	15.35	13.44*	14.95
		16.19	13.40	16.37	14.82	14.79	14.63	19.76*	16.74	15.71	11.00	16.28	16.69	11.66	15.51	30.34*	14.95
	2	16.40	14.50	18.31	15.31	17.87	15.09	20.51*	17.03	13.73	9.70*	16.52	15.94	13.03	15.27	17.83	14.95
		17.00	14.70	16.79	2.10*	14.82	14.63	20.32*	16.74	14.02	9.50*	16.67	15.61	15.92	15.86	11.59	14.95

DATE 07/09/73

XYLENE (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	.21	.22	.20	.24	.20	.19	.14	.21	.19	.24	.19	.21	.00 *	.19	.00*	.21
		.23	.21	.17	.25	.19	.19	.15	.21	.18	.27	.19	.19	.00 *	.18	.16	.21
	2	.23	.22	.17	.21	.15	.18	.21	.20	.22	.20	.19	.17	.33 *	.19	.18	.21
		.22	.21	.21	.23	.18	.20	.01*	.19	.22	.21	.19	.17	.29 *	.18	.18	.21
2	1	2.75	2.39	2.52	2.40	2.30	2.20	2.33	2.40	2.35	2.30	2.17	2.57	5.52 *	2.26	2.25	2.56
		2.86	2.31	2.50	2.37	2.61	2.55	2.55	2.34	2.52	2.41	2.42	2.41	3.01 *	2.20	2.38	2.56
	2	2.90	2.37	2.59	2.80	2.42	2.34	1.90	2.42	2.40	2.52	2.21	2.67	2.28	2.32	2.48	2.56
		2.70	2.36	2.56	2.71	2.36	2.18	1.92	2.46	2.32	2.30	2.36	2.31	5.69 *	2.24	2.37	2.56
3	1	4.81	4.67	5.24	*	4.33	4.77	4.70	4.66	4.87	.87*	4.30	4.45	8.38 *	4.07	5.00	4.90
		4.50	4.62	4.77	4.95	4.19	4.47	4.59	4.96	4.86	.98*	4.43	4.47	7.41 *	3.69	4.58	4.90
	2	.20*	4.85	5.70	4.66	4.58	4.60	4.04	4.57	4.85	4.50	4.38	5.02	4.76	3.63	5.40	4.90
		4.80	4.95	5.26	4.81	4.49	4.53	4.00	4.58	4.78	4.39	4.51	4.80	4.34	3.84	4.94	4.90
4	1	8.70	6.76	6.99	7.26	6.98	6.40	5.53	7.06	8.35	5.59	6.46	7.94	5.85	6.31	7.16	6.80
		8.50	6.92	7.98	6.89	6.83	5.98	.08*	7.12	7.81	6.14	6.48	7.42	4.57 *	6.38	.19*	6.80
	2	6.24	6.76	8.03	7.18	6.68	6.32	.04*	7.47	8.13	1.42*	6.93	7.08	8.78	6.70	7.79	6.80
		6.02	6.95	7.42	7.45	6.73	6.71	6.62	7.20	7.89	1.64*	6.83	8.11	9.79 *	6.77	7.72	6.80
5	1	9.10	8.11	8.98	9.86	8.55	9.40	7.74	8.16	9.31	10.47	8.07	8.94	6.97	8.17	7.92	8.52
		9.90	8.01	8.68	9.85	8.56	8.47	7.30	7.70	9.25	6.03	8.07	9.97	8.64	7.91	8.35	8.52
	2	7.90	8.21	9.37	9.45	8.30	9.60	7.63	8.34	7.85	7.02	8.27	8.82	8.75	7.87	8.43	8.52
		8.60	8.75	8.26	10.32	7.90	8.20	7.56	8.14	7.90	6.69	8.16	9.52	9.36	8.20	8.19	8.52

DATE 07/09/73

BENZENE (MIXTURE) (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	.39	.38	.33	.40	.32	.34	.32	.31	.40	.38	.36	.37	1.44 *	.35	.29	.35
		.44	.36	.33	.42	.34	.33	.32	.33	.40	.39	.37	.37	1.81 *	.33	.29	.35
	2	.21	.41	.34	.42	.33	.34	.38	.33	.42	.26	.34	.39	.37	.34	.38	.35
		.28 *	.40	.38	.38	.34	.34	.37	.31	.39	.33	.36	.40	.41	.31	.38	.35

DATE 07/09/73

XYLENE (MIXTURE) (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	4.78	4.19	4.11	5.08	4.06	4.48	3.79	4.14	4.73	1.10*	4.04	4.75	7.25*	4.16	3.57	4.55
		4.36	4.10	4.09	5.49	3.92	4.32	3.60	4.35	4.88	1.01*	4.31	4.62	7.30*	4.00	3.72	4.55
	2	3.60	4.67	4.47	5.19	3.73	4.35	4.91	4.23	4.66	3.18	3.89	4.65	3.07	3.99	3.52	4.55
		3.60	4.54	4.39	4.74	4.14	4.28	4.67	4.10	4.60	4.39	4.01	4.69	2.75*	3.77	3.74	4.55

DATE 07/09/73

ETHYLENE DICHLORIDE (MIXTURE) (MG)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	2.14	2.18	2.14	2.86	1.85	2.68	1.92	2.27	1.84	2.17	2.26	2.65	.82*	2.04	2.38	2.22
		2.29	2.20	2.39	2.93	1.90	2.16	.63*	2.32	1.99	2.17	2.13	2.61	.90*	2.10	2.15	2.22
	2	2.90	1.97	2.83	3.34	2.34	2.55	1.85	2.43	2.14	1.96	2.25	2.58	2.08	1.91	2.29	2.22
		2.80	2.15	3.13	3.60	2.02	2.36	1.92	2.31	2.35	2.17	2.29	2.82	1.34	2.12	2.36	2.22

DATE 07/12/73

TRICHLOROETHYLENE (MIXTURE) (ppm)

LEVEL	DAY	LAB 1	LAB 2	LAB 3	LAB 4	LAB 5	LAB 6	LAB 7	LAB 8	LAB 9	LAB10	LAB11	LAB12	LAB13	LAB14	LAB15	SCOTT
1	1	5.42	5.51	5.27	5.62	4.93	5.31	5.55	6.15	5.84	5.68	5.84	6.12	3.14*	5.37	5.89	5.66
		5.37	5.52	5.46	5.83	5.13	5.17	2.77	5.35	5.91	5.64	5.26	6.36	2.93*	5.46	5.38	5.66
	2	5.41	4.95	5.25	5.63	5.14	5.74	5.42	5.69	5.08	4.58	5.59	6.48	4.82	4.94	5.76	5.66
		5.5	5.51	5.31	5.95	4.71	5.41	5.53	5.36	5.18	4.98	5.68	7.11	2.66*	5.45	5.88	5.66

TABLE C-4

YOUDEN ANALYSIS ON DATA FOR EACH

PAIR AT EACH LEVEL (PHASE II)

DATE 07/17/73

BENZENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.0140	.0176	.0036	.118-04	.281-04	5.76	12	24.546	-2.22
	2	.0178	.0176	-.0002	.495-05	.122-04	5.93	12	12.521	.15
2	1	.1960	.2031	.0071	.101-02	-.714-04	.86	14	16.183	-1.32
	2	.1871	.2031	.0160	.110-03	.674-03	13.23	14	5.611	-2.30
3	1	.3492	.3335	-.0157	.468-03	.123-02	6.24	12	6.196	1.48
	2	.3517	.3335	-.0182	.143-02	.678-03	1.95	14	10.755	1.88
4	1	.6626	.6271	-.0355	.934-03	.826-02	18.68	14	4.612	1.47
	2	.5972	.6271	.0299	.163-02	.263-02	4.23	14	6.759	-1.97
5	1	1.5686	1.5716	.0030	.822-02	.212-01	6.14	14	5.781	-.07
	2	1.6634	1.5716	-.0918	.137-01	.101-01	2.48	14	7.032	2.73

DATE 07/17/73

CARBON TETRACHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.0350	.0280	-.0070	.124-03	.274-03	5.44	10	31.781	1.26
	2	.0328	.0280	-.0048	.141-03	.179-03	3.54	12	36.243	1.09
2	1	.4646	.4338	-.0308	.141-02	.195-02	3.77	13	8.089	2.23
	2	.4745	.4338	-.0407	.132-02	.958-03	2.46	14	7.646	3.92
3	1	.7633	.7051	-.0582	.138-02	.756-02	11.92	11	4.874	2.22
	2	.7643	.7051	-.0592	.271-02	.708-02	6.22	14	6.809	2.50
4	1	1.5001	1.4692	-.0309	.726-01	- 432-02	.88	11	17.963	.60
	2	1.4985	1.4692	-.0293	.313-02	.532-01	35.06	12	3.731	.45
5	1	15.6862	15.6500	-.0362	.599+00	.312+01	11.41	11	4.934	.07
	2	15.7102	15.6500	-.0602	.121+01	.676+01	12.21	13	6.989	.08

DATE 07/17/73

CHLOROFORM

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.1271	.1212	-.0059	.312-03	.346-03	3.22	12	13.904	.94
	2	.1276	.1212	-.0064	.985-03	.126-03	1.26	12	24.593	.93
2	1	1.5890	1.5814	-.0076	.257-01	.192-01	2.49	12	10.098	.15
	2	1.6191	1.5814	-.0377	.249-01	.890-02	1.71	13	9.747	.97
3	1	2.6622	2.5128	-.1494	.320-01	.646-01	5.04	12	6.718	1.90
	2	2.7369	2.5128	-.2241	.239-01	.394-01	4.29	13	5.653	3.70
4	1	4.9334	4.9132	-.0202	.289+00	.778-01	1.54	12	10.888	.15
	2	5.0103	4.9132	-.0971	.110+00	.385+00	7.99	13	6.622	.55

DATE 07/17/73

DIOXANE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.1999	.2306	.0307	.334-03	.144-02	9.66	12	9.136	-2.76
	2	.2007	.2306	.0299	.122-03	.421-03	7.92	11	5.493	-4.72
2	1	1.9817	2.1844	.2027	.265-01	.864-01	7.51	14	8.220	-2.49
	2	2.0719	2.1844	.1125	.468-01	.170+00	8.26	14	10.446	-.99
3	1	3.8584	3.9107	.0523	.517-01	.210+00	9.11	13	5.895	-.40
	2	3.7140	3.9107	.1967	.757-01	.229+00	7.06	12	7.406	-1.37
4	1	5.6899	5.9376	.2477	.838-01	.294+00	8.01	13	5.088	-1.60
	2	5.4326	5.9376	.5050	.355-01	.712+00	41.08	13	3.469	-2.21
5	1	6.6582	7.0371	.3789	.729-01	.434+00	12.90	12	4.055	-1.99
	2	6.7267	7.0371	.3104	.328+00	.709+00	5.32	13	8.517	-1.24

DATE 07/17/73

ETHYLENE DICHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.1102	.0994	-.0108	.347-04	.372-03	22.43	13	5.349	2.04
	2	.1112	.0994	-.0118	.332-04	.296-03	18.82	13	5.183	2.50
2	1	1.4177	1.4103	-.0074	.315-02	.118-01	8.50	12	3.961	.23
	2	1.4426	1.4103	-.0323	.229-01	.849-02	1.74	13	10.472	.86
3	1	2.0940	2.1859	.0919	.104-01	.418-01	9.03	13	4.870	-1.59
	2	2.1913	2.1859	-.0054	.614-01	.227-01	1.74	13	11.308	.09
4	1	3.9554	3.8860	-.0694	.149-01	.282+00	38.86	14	3.085	.50
	2	4.0806	3.8860	-.1946	.302-01	.215+00	15.26	13	4.258	1.52
5	1	7.9589	7.5497	-.4092	.101+00	.522+00	11.36	13	3.990	2.02
	2	8.1374	7.5497	-.5877	.280+00	.498+00	4.56	12	6.504	2.65

DATE 07/17/73

TRICHLOROETHYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.1788	.1842	.0054	.719-03	.175-03	1.49	10	14.994	-.77
	2	.1944	.1842	-.0102	.108-02	.134-02	3.49	12	16.875	.85
2	1	3.4559	3.5525	.0966	.309-01	.109+00	8.05	13	5.088	-1.02
	2	3.4603	3.5525	.0922	.984-01	.106+00	3.15	12	9.063	-.84
3	1	5.7576	5.8372	.0796	.164+00	.396-01	1.48	13	7.038	-.85
	2	5.4870	5.8372	.3502	.851-01	.386+00	10.07	13	5.318	-2.00
4	1	10.1380	10.1112	-.0268	.245+00	.803+00	7.56	11	4.880	.10
	2	10.0586	10.1112	.0526	.633+00	.857-01	1.27	12	7.907	-.30
5	1	15.1249	14.9482	-.1767	.103+01	.233+01	5.54	12	6.696	.38
	2	15.6608	14.9482	-.7126	.254+01	.748-01	1.06	11	10.176	2.13

DATE 07/17/73

XYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.2415	.2082	.0067	.132-03	.748-03	12.37	12	5.691	-.85
	2	.1962	.2082	.0120	.135-03	.267-03	4.96	12	5.913	-2.36
2	1	2.4191	2.5569	.1378	.112-01	.130-01	3.32	13	4.375	-3.78
	2	2.4106	2.5569	.1463	.778-02	.435-01	12.17	13	3.659	-2.52
3	1	4.5838	4.9418	.3580	.275-01	.792-01	6.76	11	3.617	-3.61
	2	4.6341	4.9018	.2677	.236-01	.179+00	16.18	13	3.315	-2.29
4	1	7.0518	6.8006	-.2512	.102+00	.594+00	12.65	11	4.527	1.08
	2	7.1297	6.8006	-.3291	.850-01	.292+00	7.86	11	4.089	1.97
5	1	8.5484	8.5229	-.0255	.900+00	.617-01	1.14	14	11.099	.14
	2	8.3862	8.5229	.1367	.217+00	.412+00	4.79	14	5.558	-.73

DATE 07/17/73

BENZENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.3557	.3542	-.0015	.138-03	.137-02	28.89	13	3.301	.14
	2	.3566	.3542	-.0024	.467-03	.123-02	6.28	13	6.061	.23

DATE 07/17/73

XYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	PRECISION S_r^2	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	4.2942	4.5485	.2543	.263-01	.193+00	15.64	12	3.778	-2.02
	2	4.2397	4.5485	.3088	.773-01	.165+00	5.26	13	6.559	-2.56

DATE 07/17/73

ETHYLENE DICHLORIDE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	2.2617	2.2157	-.0460	.194-01	.644-01	7.63	12	6.163	.61
	2	2.3725	2.2157	-.1568	.396-01	.193+00	18.73	14	8.391	1.32

DATE 07/17/73

TRICHLOROETHYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	5.7493	5.6552	-.0941	.374+00	.316+00	2.69	13	10.636	.50
	2	5.8590	5.6552	-.2038	.140+00	.378+00	6.41	13	6.376	1.14

TABLE C-5

YOUDEN ANALYSIS ON DATA AT EACH

LEVEL (REPLICATES AVERAGED) (PHASE II)

DATE 07/18/73

BENZENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.0158	.0176	.0018	.234-04	-.327-05	.72	11	30.544	-2.11
2		.1916	.2431	.0115	.728-03	-.148-03	.59	14	14.088	-3.04
3		.3528	.3335	-.0193	.560-03	.826-03	3.95	12	6.705	2.09
4		.6299	.6271	-.0028	.738-02	-.130-02	.65	14	13.638	.22
5		1.6160	1.5716	-.0444	.193-01	.186-02	1.19	14	8.588	1.61

DATE 07/18/73

CARBON TETRACHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.0336	.0280	-.0056	.100-04	.306-03	62.07	10	9.437	1.04
2		.4677	.4338	-.0339	.136-02	.728-03	2.07	13	7.888	3.38
3		.7555	.7051	-.0504	.186-02	.506-02	6.43	11	5.715	2.26
4		1.5628	1.4692	-.0936	.168-01	.181-02	1.22	9	8.295	2.93
5		15.6197	15.6500	.0303	.139+01	.307+01	5.41	11	7.554	-.05

DATE 07/18/73

CHLOROFORM

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.1273	.1212	-.0061	.697-03	-.136-03	.61	12	20.728	1.52
2		1.6184	1.5814	-.0370	.138-01	.133-01	2.94	11	7.252	.90
3		2.6986	2.5128	-.1858	.145-01	.544-01	8.52	11	4.459	2.59
4		5.0372	4.9132	-.1240	.141+00	.176+00	3.50	11	7.456	.86

DATE 07/18/73

DIOXANE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.2021	.2306	.0285	.376-03	.655-03	4.49	11	9.593	-3.41
2		2.0268	2.1844	.1576	.111+00	.358-01	1.65	14	16.421	-2.02
3		3.7994	3.9107	.1113	.666-01	.189+00	6.68	12	6.793	-.85
4		5.5613	5.9376	.3763	.215+00	.317+00	3.95	13	8.344	-2.16
5		6.8038	7.0371	.2341	.329+00	.248+00	2.51	11	8.427	-1.26

DATE 07/18/73

ETHYLENE DICHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.1142	.0994	-.0108	.106-03	.219-03	5.14	12	9.343	2.36
2		1.4344	1.4103	-.0201	.156-01	.184-02	1.24	12	8.742	.74
3		2.1695	2.1859	.0164	.146-01	.285-01	4.91	12	5.567	-.31
4		4.0333	3.8864	-.1473	.765-01	.187+00	5.89	13	6.858	1.16
5		7.9496	7.5497	-.3999	.258+00	.242+00	2.88	11	6.386	2.28

DATE 07/18/73

TRICHLOROETHYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.1869	.1842	-.0027	.767-03	.615-03	2.60	10	14.814	.29
2		3.4181	3.5525	.1344	.759-01	.417-01	2.10	11	8.060	-1.65
3		5.5991	5.8372	.2381	.143+00	.145+00	3.03	12	6.757	-1.84
4		10.1579	10.1112	-.0467	.551+00	-.929-02	.97	10	7.305	.30
5		15.5461	14.9482	-.5979	.985+00	.653+00	2.33	10	6.384	1.85

DATE 07/18/73

XYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.2417	.2082	.0065	.235-03	.231-03	2.96	11	7.604	-1.21
2		2.4149	2.5569	.1420	.185-01	.145-01	2.56	13	5.632	-3.45
3		4.6139	4.9018	.2879	.568-01	.115+00	5.06	10	5.166	-2.52
4		7.1162	6.8006	-.3156	.334+00	.107+00	1.64	10	8.118	2.00
5		8.4673	8.5229	.0556	.259+00	.257+00	2.98	14	6.016	-.35

DATE 07/18/73

BENZENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.3527	.3542	.0015	.600-03	.779-03	3.60	12	6.944	-.16

DATE 07/18/73

XYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		4.2845	4.5485	.2640	.112+00	.914-01	2.63	12	7.824	-2.48

DATE 07/18/73

ETHYLENE DICHLORIDE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		2.3614	2.2157	-.1457	.409-01	.878-01	5.29	12	8.561	1.60

DATE 07/18/73

TRICHLOROETHYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		5.8442	5.6552	-.1490	.167+00	.308+00	4.68	13	7.049	.89

TABLE C-6

YOUDEN ANALYSIS FOR LOG-TRANSFORMED

DATA FOR EACH PAIR AT EACH LEVEL (PHASE II)

DATE 07/17/73

BENZENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-4.7528	-4.0399	.7129	.960+00	.187+01	4.90	12	-20.613	-1.68
	2	-4.0527	-4.0399	.0128	.852-02	.360-01	9.45	12	-2.278	-.23
2	1	-1.6447	-1.5941	.0507	.363-01	-.341-02	.81	14	-11.579	-1.62
	2	-1.6872	-1.5941	.0932	.316-02	.210-01	14.32	14	-3.331	-2.40
3	1	-1.0588	-1.0981	-.0393	.406-02	.110-01	6.42	12	-6.019	1.24
	2	-1.0533	-1.0981	-.0448	.123-01	.392-02	1.64	14	-10.512	1.73
4	1	-.4244	-.4666	-.0462	.170-02	.162-01	19.98	14	-9.817	1.37
	2	-.5215	-.4666	.0549	.496-02	.866-02	4.49	14	-13.503	-2.01
5	1	.4442	.4521	.0079	.380-02	.909-02	5.79	14	13.874	-.29
	2	.5050	.4521	-.0529	.449-02	.371-02	2.65	14	13.263	2.65

DATE 07/17/73

CARBON TETRACHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-3.8624	-3.5756	.2868	.373-01	.323+01	174.24	10	-5.002	-.53
	2	-4.0287	-3.5756	.4532	.132+01	.256+01	4.87	12	-28.537	-.91
2	1	-.7745	-.8352	-.0607	.755-02	.981-02	3.60	13	-11.221	1.95
	2	-.7505	-.8352	-.0847	.671-02	.404-02	2.20	14	-10.914	3.81
3	1	-.2778	-.3494	-.0717	.224-02	.150-01	14.38	11	-17.034	1.96
	2	-.2771	-.3494	-.0723	.544-02	.126-01	5.63	14	-26.614	2.26
4	1	.3896	.3847	-.0049	.349-01	.125-02	1.07	11	47.955	.12
	2	.3917	.3847	-.0070	.150-02	.275-01	37.58	12	9.900	.15
5	1	2.7459	2.7505	.0046	.279-02	.123-01	9.82	11	1.925	-.14
	2	2.7391	2.7505	.0114	.598-02	.273-01	10.11	13	2.824	-.25

DATE 07/17/73

CHLOROFORM

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-2.0825	-2.1103	-.0278	.144-01	.268-01	4.72	12	-5.761	.54
	2	-2.0932	-2.1103	-.0171	.904-01	-.120-01	.74	12	-14.362	.34
2	1	.4547	.4583	.0036	.110-01	.709-02	2.29	12	23.050	-.12
	2	.4753	.4583	-.0170	.111-01	.331-02	1.60	13	22.130	.68
3	1	.9726	.9214	-.0512	.511-02	.884-02	4.46	12	7.351	1.73
	2	1.0027	.9214	-.0813	.317-02	.526-02	4.32	13	5.612	3.68
4	1	1.5869	1.5919	.0050	.185-01	.309-02	1.33	12	8.568	-.16
	2	1.6020	1.5919	-.0100	.456-02	.164-01	8.18	13	4.214	.28

DATE 07/17/73

DIOXANE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-1.6306	-1.4671	.1635	.140-01	.308-01	5.41	12	-7.250	-3.03
	2	-1.6122	-1.4671	.1451	.330-02	.103-01	7.24	11	-3.563	-4.60
2	1	.6691	.7813	.1123	.101-01	.227-01	5.48	14	15.036	-2.61
	2	.7057	.7813	.0756	.976-02	.382-01	8.84	14	13.996	-1.41
3	1	1.3422	1.3637	.0215	.312-02	.141-01	10.03	13	4.163	-.64
	2	1.3018	1.3637	.0619	.573-02	.167-01	6.82	12	5.815	-1.60
4	1	1.7334	1.7813	.0479	.223-02	.900-02	9.07	13	2.724	-1.78
	2	1.6805	1.7813	.1008	.149-02	.243-01	33.64	13	2.295	-2.39
5	1	1.8906	1.9512	.0606	.151-02	.979-02	13.95	12	2.057	-2.13
	2	1.8954	1.9512	.0558	.648-02	.164-01	6.08	13	4.246	-1.49

DATE 07/17/73

ETHYLENE DICHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-2.2199	-2.3286	-.0887	.269-02	.263-01	20.54	13	-2.335	2.00
	2	-2.2084	-2.3086	-.1002	.305-02	.231-01	16.17	13	-2.501	2.39
2	1	.3453	.3438	-.0015	.159-02	.660-02	9.30	12	11.544	.06
	2	.3596	.3438	-.0158	.106-01	.372-02	1.70	13	28.642	.62
3	1	.7333	.7820	.0487	.239-02	.105-01	9.76	13	6.672	-1.69
	2	.7763	.7820	.0057	.132-01	.385-02	1.58	13	14.817	-.21
4	1	1.3660	1.3574	-.0086	.109-02	.185-01	35.05	14	2.414	.24
	2	1.3993	1.3574	-.0419	.216-02	.124-01	12.53	13	3.320	1.35
5	1	2.0698	2.0215	-.0483	.171-02	.789-02	10.21	13	2.000	1.93
	2	2.0910	2.0215	-.0695	.419-02	.753-02	4.60	12	3.094	2.55

DATE 07/17/73

TRICHLOROETHYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-1.7361	-1.6917	.0444	.287-01	.627-02	1.44	10	-9.751	-1.03
	2	-1.6656	-1.6917	-.0262	.208-01	.374-01	4.60	12	-8.664	.43
2	1	1.2347	1.2677	.0330	.271-02	.893-02	7.58	13	4.219	-1.22
	2	1.2332	1.2677	.0345	.870-02	.946-02	3.17	12	7.564	-1.06
3	1	1.7472	1.7643	.0170	.493-02	.125-02	1.51	13	4.018	-1.05
	2	1.6950	1.7643	.0693	.249-02	.135-01	11.83	13	2.943	-2.14
4	1	2.3113	2.3136	.0023	.224-02	.830-02	8.41	11	2.047	-.08
	2	2.3051	2.3136	.0085	.557-02	.113-02	1.41	12	3.238	-.49
5	1	2.7089	2.7046	-.0043	.458-02	.120-01	6.25	12	2.498	.13
	2	2.7458	2.7046	-.0412	.116-01	-.134-04	1.00	11	3.928	1.87

DATE 07/17/73

XYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-1.6119	-1.5693	.0426	.284-02	.188-01	14.20	12	-3.307	-1.08
	2	-1.6335	-1.5693	.0642	.375-02	.683-02	4.64	12	-3.751	-2.48
2	1	.8814	.9388	.0574	.193-02	.199-02	3.46	13	4.983	-3.95
	2	.8756	.9388	.0632	.131-02	.784-02	12.99	13	4.131	-2.57
3	1	1.5200	1.5896	.0696	.130-02	.410-02	7.32	11	2.369	-3.50
	2	1.5289	1.5896	.0607	.102-02	.870-02	18.00	13	2.093	-2.37
4	1	1.9470	1.9170	-.0300	.202-02	.116-01	12.54	11	2.307	.92
	2	1.9609	1.9170	-.0439	.154-02	.587-02	8.64	11	2.000	1.86
5	1	2.1393	2.1428	.0035	.136-01	.314-03	1.05	14	5.459	-.16
	2	2.1224	2.1428	.0203	.279-02	.607-02	5.35	14	2.490	-.91

DATE 07/17/73

BENZENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_E^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	-1.0394	-1.0379	.0015	.946-03	.110-01	24.20	13	-2.959	-.05
	2	-1.0376	-1.0379	-.0003	.417-02	.100-01	5.81	13	-6.222	.01

DATE 07/17/73

XYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	1.4524	1.5148	.0624	.130-02	.101-01	16.45	12	2.485	-2.19
	2	1.4388	1.5148	.1768	.506-02	.909-02	4.60	13	4.945	-2.67

DATE 07/17/73

ETHYLENE DICHLORIDE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	.8089	.7956	-.0133	.362-02	.117-01	7.46	12	7.441	.41
	2	.8450	.7956	-.0494	.105-01	.303-01	6.80	14	12.099	1.02

DATE 07/17/73

TRICHLOROETHYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PATH	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1	1	1.7367	1.7326	-.0041	.198-01	.963-02	1.97	13	8.105	.11
	2	1.7609	1.7326	-.0283	.421-02	.114-01	5.23	13	3.685	.92

TABLE C-7

YOUDEN ANALYSIS ON LOG-TRANSFORMED DATA

AT EACH LEVEL (REPLICATES AVERAGED) (PHASE II)

DATE 07/18/73

BENZENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		-4.3353	-4.0399	.2955	.122+01	-.125+00	.79	11	-25.480	-1.47
2		-1.6660	-1.5941	.0719	.235-01	-.481-02	.59	14	-9.198	-3.35
3		-1.0477	-1.0981	-.0504	.431-02	.656-02	4.04	12	-6.268	1.95
4		-.4710	-.4666	.0043	.166-01	-.255-02	.69	14	-27.377	-.22
5		.4746	.4521	-.0225	.809-02	.382-03	1.09	14	18.954	1.31

DATE 07/18/73

CARBON TETRACHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		-4.0100	-3.5756	.4344	.305+00	.319+01	21.94	10	-13.769	-.79
2		-.7663	-.8352	-.0688	.703-02	.326-02	1.93	13	-10.943	3.13
3		-.2874	-.3494	-.0620	.309-02	.107-01	7.91	11	-19.342	1.94
4		.4410	.3847	-.0562	.718-02	.894-03	1.25	9	19.216	2.66
5		2.7393	2.7505	.0112	.592-02	.129-01	5.35	11	2.808	-.31

DATE 07/18/73

CHLOROFORM

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		-2.0879	-2.1103	-.0224	.449-01	-.113-01	.50	12	-10.144	.77
2		.4736	.4583	-.0153	.564-02	.528-02	2.87	11	15.850	.59
3		.9874	.9214	-.0660	.207-02	.749-02	8.22	11	4.612	2.48
4		1.6078	1.5919	-.0159	.744-02	.749-02	3.02	11	5.363	.52

DATE 07/18/73

DIOXANE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		-1.6105	-1.4671	.1435	.696-02	.163-01	5.68	11	-5.181	-3.53
2		.6874	.7813	.0940	.264-01	.905-02	1.69	14	23.631	-2.44
3		1.3253	1.3637	.0384	.489-02	.130-01	6.32	12	5.275	-1.11
4		1.7069	1.7813	.0744	.822-02	.935-02	3.28	13	5.310	-2.40
5		1.9103	1.9512	.0409	.649-02	.596-02	2.84	11	4.217	-1.48

DATE 07/18/73

ETHYLENE DICHLORIDE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		-2.2177	-2.3086	-.0909	.778-02	.165-01	5.25	12	-3.976	2.29
2		.3524	.3438	-.0086	.735-02	.124-02	1.34	12	24.321	.44
3		.7681	.7820	.0140	.318-02	.614-02	4.97	12	7.246	-.57
4		1.3864	1.3574	-.0290	.423-02	.123-01	6.83	13	4.692	.90
5		2.0687	2.0215	-.0472	.381-02	.403-02	3.11	11	2.985	2.12

DATE 07/18/73

TRICHLOROETHYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		-1.7015	-1.6917	.0098	.173-01	.214-01	3.47	10	-7.734	-.19
2		1.2231	1.2677	.0446	.673-02	.389-02	2.16	11	6.709	-1.81
3		1.7168	1.7643	.0475	.470-02	.497-02	3.11	12	3.994	-2.00
4		2.3152	2.3136	-.0016	.554-02	-.826-04	.97	10	3.215	.10
5		2.7395	2.7046	-.0349	.437-02	.300-02	2.37	10	2.412	1.61

DATE 07/18/73

XYLENE

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		-1.6471	-1.5693	.0378	.524-02	.551-02	3.10	11	-4.504	-1.45
2		.8785	.9388	.0603	.358-02	.223-02	2.27	13	6.731	-3.58
3		1.5249	1.5896	.0647	.268-02	.578-02	5.31	10	3.396	-2.54
4		1.9581	1.9170	-.0411	.640-02	.205-02	1.64	10	4.086	1.88
5		2.1349	2.1428	.0119	.346-02	.384-02	3.22	14	2.761	-.62

DATE 07/18/73

ETHYLENE DICHLORIDE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		.8479	.7956	-.0523	.627-02	.138-01	5.40	12	9.340	1.45

DATE 07/18/73

TRICHLOROETHYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		1.7488	1.7326	-.0162	.719-02	.914-02	3.54	13	4.847	.54

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DATE 07/18/73

BENZENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		-1.0478	-1.0379	.0099	.523-02	.609-02	3.33	12	-6.981	-.38

DATE 07/18/73

XYLENE (MIXTURE)

ANALYSIS SUMMARY (MG)

LEVEL	PAIR	AVERAGE AMOUNT FOUND	AVERAGE AMOUNT PRESENT	BIAS OF PROCEDURE	S_r^2 PRECISION	SYSTEMATIC ERROR S_b^2	F RATIO	DEGREES OF FREEDOM	COEFF OF VARIATION	T-TEST FOR SYSTEMATIC ERROR
1		1.4496	1.5148	.0652	.635-02	.465-02	2.46	12	5.497	-2.66