Task 11 - Evaluation of TCE Contamination Before and After the Field Experiment

Topical Report

September 26, 1994 - May 25, 1996

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A. Executive Summary

Development of an Integrated *in-situ* Remediation Technology

DOE Contract Number: DE-AR21-94MC31185

Topical Report for Task #11 entitled "Evaluation of TCE Contamination Before and After the Field Experiment" (September 26, 1994 - February 25, 1996)

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Abstract: Contamination in low permeability soils poses a significant technical challenge to *in-situ* remediation efforts. Poor accessibility to the contaminants and difficulty in delivery of treatment reagents have rendered existing *in-situ* treatments such as bioremediation, vapor extraction, pump and treat rather ineffective when applied to low permeability soils present at many contaminated sites. The technology is an integrated *in-situ* treatment in which established geotechnical methods are used to install degradation zones directly in the contaminated soil and electro-osmosis is utilized to move the contaminants back and forth through those zones until the treatment is completed. The present Topical Report for Task #11 summarizes the results of TCE analysis in soil and carbon before and after conducting the field experiment. In addition, a discussion of the TCE material balance demonstrates that the Lasagna™ process is effective in moving TCE from the contaminated soil into carbon treatment zones in the field experiment at DOE's Gaseous Diffusion Plant in Paducah, Kentucky.

B. Acronyms and Abbreviations

bgs	below ground surface
DOE	Department of Energy
DuPont	E. I. du Pont de Nemours & Co., Inc.
ECD	electron capture detector
EPA	Environmental Protection Agency
GAC	granular activated carbon
GC	gas chromatography
GE	General Electric Company
MMEC	Martin Marietta Energy Corporation
NA	not available
TCE	trichloroethylene
VOC	volatile organic compound

C. Units

BTU	British thermal unit
C, °C	Celsius, degrees Celsius
cc	cubic centimeters
cm	centimeters
d, D	days
deg	degrees
F, °F	Fahrenheit, degrees Fahrenheit
f, ft	feet
g	grams
gal, GAL	gallons
h, hr	hours
in	inches
k, K	thousand
kg	kilograms
l, L	liters
lb, lbs	pound(s)
m	meter
mg	milligrams
min	minutes
ml, mL	milliliters
mm	millimeters
ppb	parts per billion
ppm, ppmw	parts per million (by weight)
psi	pounds per square inch
µg	micrograms
µl, µL	microliters
"	inches
'	feet
#	pounds

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E. Background

Statement of the Problem

Contamination in low permeability soils poses a significant technical challenge to *in-situ* remediation efforts. Poor accessibility to the contaminants and difficulty in delivery of treatment reagents have rendered existing *in-situ* treatments such as bioremediation, vapor extraction, and pump and treat, rather ineffective when applied to low permeability soils present at many contaminated sites.

The Solution

The proposed technology combines electro-osmosis with treatment zones that are installed directly in the contaminated soils to form an integrated *in-situ* remedial process. Electro-osmosis is an old civil engineering technique and is well known for its effectiveness in moving water uniformly through low-permeability soils with very low power consumption.

Conceptually, the integrated technology could treat organic and inorganic contamination, as well as mixed wastes. Once developed, the technology will have tremendous benefits over existing ones in many aspects including environmental impacts, cost effectiveness, waste generation, treatment flexibility, and breadth of applications.

Consortium Description

A Consortium has been formed consisting of Monsanto, E. I. du Pont de Nemours & Co., Inc. (DuPont) and General Electric (GE), with participation from the Environmental Protection Agency (EPA) Office of Research and Development and the Department of Energy (DOE) Environmental Management Office of Science and Technology. The five members of this group are leaders in their represented technologies and hold significant patents and

intellectual property which, in concert, may form an integrated solution for soil treatment. The Consortium's activities are being facilitated by Clean Sites, Inc., under a Cooperative Agreement with EPA's Technology Innovation Office. The figure on the cover page shows a schematic diagram of the various technologies which the government/industry consortium has integrated for the development of an *in-situ* remediation technology.

Management Plan

A Management Plan for this project was prepared by Monsanto and submitted on November 30, 1994. That plan summarized the work plan which was developed in conjunction with DuPont, GE, EPA's Risk Reduction Engineering Laboratory (RREL), Martin Marietta Energy Systems (MMES), and the Department of Energy. The DOE Gaseous Diffusion Plant in Paducah, Kentucky, was chosen as the site for the initial field tests.

CDM Federal Programs Corporation was chosen to provide the on-site support of the field tests which were installed at the DOE site in November 1994. This experiment tested the combination of electro-osmosis and *in-situ* sorption in the treatment zones. In 1994 and 1995, technology development was carried out under the present contract by Monsanto, DuPont, and GE. These studies evaluated various degradation processes and their integration into the overall treatment scheme at bench and pilot scales.

Technical Deliverables

Tables 1 and 2 summarize the 13 technical tasks and the 8 topical reports which will be written describing the results obtained in the technical tasks. These two tables show which organization is primarily responsible for the tasks and for preparing the topical reports. The present topical report summarizes Task #11.

Table E-1. List of Tasks and Responsible Company

Task	Company
Task #1 - Evaluation of Treatment Zone Formation Options	DuPont
Task #2 - Electrokinetic Model Validation and Improvement	GE
Task #3 - Design Guidance for Field Experiments	GE/DuPont
Task #4 - Analysis of Electrode Geometry and Soil Heterogeneity	GE/DuPont
Task #5 - Cost Analysis	Monsanto/DuPont
Task #6 - Lab-Scale Development of Microbial Degradation Process	DuPont
Task #7 - Lab-Scale Electrokinetic and Microbial Degradation	Monsanto
Task #8 - Lab-Scale Tests of Lasagna Process Using DOE Paducah Soil	Monsanto
Task #9 - TCE Degradation Using Non-Biological Methods	GE/Monsanto
Task #10 - Bench- and Pilot-Scale Tests	Monsanto
Task #11 - Establish Contamination Conditions Before and After Tests	DuPont/MMES
Task #12 - Design and Fabrication of Large-Scale Lasagna Process	Monsanto/DuPont/Nilex
Task #13 - Large-Scale Field Test of Lasagna Process	Monsanto/CDM

Table E-2. List of Topical Reports and Responsible Company

Topical Report	Company
Task #1 - Evaluation of Treatment Zone Formation Options	DuPont
Tasks #2 - 4 Electrokinetic Modeling	GE
Task #5 - Cost Analysis	DuPont
Task #6 - Laboratory-Scale Development of Microbial Degradation Process	DuPont
Task #7 - Development of Degradation Processes	Monsanto
Tasks #8 and 10 - Laboratory and Pilot-Scale Experiments of the <i>Lasagna</i> TM Processes	Monsanto
Task #9 Part I - TCE Degradation Using Non-Biological Methods	GE
Task #9 Part II- TCE Degradation Using Non-Biological Methods	Monsanto
Task #11 - Contamination Analysis, Before and After Treatment	Monsanto
Tasks #12 and 13 - Large-Scale Field Test of <i>Lasagna</i> TM Process	Monsanto

F. Topical Report for Task #11

In January 1995, the Phase I *Lasagna*TM field experiment was begun at DOE's Gaseous Diffusion Plant at Paducah, Kentucky. Figure F-1 shows a three-dimensional view of this experiment.

The road and cylinders shown in Figure F-1 are north of the field experiment which is indicated by the black rectangular verticle electrodes and carbon-filled treatment zones

which were installed through a 5-ft. thick gravel layer and into a 10-ft. thick clay layer. This latter clay layer is contaminated with TCE at concentration greater than 100 ppmw at several locations. The groundwater shown in Figure F-1 is approximately 15 ft. below ground surface (bgs).

Figure F-1. Three-Dimension View of the Phase I *Lasagna*TM Field Experiment.

A detailed over-view of this site is shown in Figure F-2. In this figure, approximately ½ of the *Lasagna*TM grid is shown to be in the TCE concentration region greater than 50 ppm. The southeast corner of the grid contains the maximum amount of TCE at this site. Table F-1 shows preliminary TCE concentration data for several core samples which was available to the Consortium before beginning the Phase I study.

(All core data was obtained by MMES and ORNL-Grand Junction.) More details of the field experiments can be found in the topical report for Tasks 12 and 13 of the present contract.¹

¹ Topical Report for Tasks #12 and 13 entitled "Large Scale Field Test of the *Lasagna*TM Process" by Athmer, et. al., in preparation (1996).

Figure F-2. Overview of the Phase I *Lasagna*™ Field Experiment.

Table F-1. Summary of TCE Concentrations in Core Samples Before Phase I.

Core Hole #	Depth (ft)	Concentration of TCE (mg/kg)	Mean TCE in Core Hole (mg/kg)
I-7	11 - 12	84.8	46.1
	15 - 16	7.3	
I-8	15 - 16	65.4	65.4
I-12	11 - 12	319.6	202.0
	15 - 16	84.4	
I-14	15 - 16	71.2	71.2
I-16	9 - 10	5.3	41.5
	15 - 16	77.7	
I-18	15 - 16	121.8	79.4
	18 - 19	36.9	

Mean of 10 samples = 87.4 mg/kg

Mean of six core holes = 84.3 mg/kg

Standard deviation of core hole means = 59.5 mg/kg

Variance of core hole means = 3.54×10^3

Standard error of core hole means = 24.3 mg/kg

Table F-1 shows that there is a wide range of TCE concentrations in this test region, and that there is a large amount of variability between samples taken from the same location, but at different depths. Therefore, as will be discussed in later sections, a number of different strategies were required in order to visualize these data, and to calculate average concentrations which could be used to show the soil TCE concentrations before and after the field experiment, and the TCE concentrations in the carbon.

Strategy

The primary goal of the Phase I field experiment was to determine whether electro-osmosis (EO) can be used to move TCE from the contaminated clay region into the carbon-filled treatment zones. The demonstration of TCE movement will show that EO can be used as an effective "pump", which, to date, has not

been available for low-permeability clay such as that present at Paducah. The lack of an effective "pump" is one of the reasons that there has been no cost effective *in-situ* methods available for remediating contaminated clay.

Once it has been shown that EO can move contamination, then the final development phase of the *Lasagna*TM process, scheduled to begin in 1996, will involve developing treatment zones into which the TCE can be moved and subsequently degraded.

Three important pieces of information was used to show effective TCE movement. The first two pieces were the TCE concentrations present in the clay before and after conducting the field experiments. The third piece was the concentration of TCE in the carbon-filled wickdrains which served as treatment zones in the Phase I field test. The following sections describe how this data was collected, shows the TCE analysis results, and demonstrates the consistency of the TCE concentration data in soil before and after the experiment, and the total amount of TCE in the carbon after the experiment, by calculating a TCE material balance for all of the regions of the field experiment.

Pre- and Post-Experiment Soil Analyses

In May 1994, ORNL-Grand Junction took pre-experiment core samples and in May 1995, they took post-experiment core samples. These samples were analyzed at 1-ft. intervals from 5 to 15 ft. below ground surface. Figure F-3 shows where the individual cores were drilled. In that figure, the core samples beginning with an "L" are the Pre-Experiment samples, and those beginning with an "LP" are Post-Experiment samples. The wick drains which are shown cross-hatched in Figure F-3 are those which were removed after the field experiment for TCE analysis and subsequent material balance calculations.

Figure F-3. Locations of Core Samples from the Phase I *Lasagna*™ Field Experiment.

Appendix A shows SOPs that the ORNL-Grand Junction used for sampling and analysis. Table F-2 summarizes the information that is located in Appendix A.

Table F-2. Summary of ORNL-Grand Junction SOPs in Appendix A.

No.	Title
TE-086	Soil Sampling for Field Screening using the Geoprobe and U2CRT
TE-087	Soil Sampling with the Microcorer
TE-105	U2CRT Operation
TE-160	Field Operation of the Hewlett Packard 5890 Series II Gas Chromatograph
TE-164	Preparation of Calibration Standards for GC Analysis of VOCs
TE-165	GC Analysis of VOCs using Solvent Extraction
TE-169	Using the Chrom Perfect Gas Chromatograph Software

Appendix B summarizes the data that they obtained from the pre- and post-experiment sampling. This appendix includes reports written by ORNL-Grand Junction personnel entitled: "Task 1: Pre-test Soil Characterization for Lasagna Pilot Test Site Paducah Gaseous Diffusion Plant" dated June 1994 and "Task 2 and 3: Carbon Sampling and Post-test Soil Characterization for Lasagna Pilot Test Site Paducah Gaseous Diffusion Plant" dated July 1995. Figures F-4 and F-5 summarize this data which clearly shows that in the *Lasagna*TM

experimental region, the TCE was effectively removed from the clay and, presumably transferred into the carbon treatment zones. Furthermore, this figure shows that there was little reduction of TCE concentrations in the control samples. In Figure F-4, the soil concentrations before the field experiment are labeled "pre-Lasagna". The concentrations, in ppm, are shown above each dark bar. The concentrations shown in this figure are the mean TCE concentrations measured in the clay from 4 feet bgs through 15 ft. bgs. The soil concentrations after the field experiment are labeled "post-Lasagna". The concentrations, in ppm, are shown above each light bar. The percent TCE reduction is shown as points on the line graph in this figure.

The three soil samples which are labeled as controls were taken from regions of the field experiment which were not between the electrodes. LP-14 and LP-15 were located within an electrically shielded region adjacent to the anode, and LP-16 was located within 1 ft. of the anode. The sample located to the right of Figure F-4 labeled "Control-Average" is the average of these three control points, and the sample labeled "Lasagna-Average" is the average of the remaining soil borings located between the anode and cathode. Figure F-5 depicts the pre-Lasagna and post-Lasagna concentrations in alternate rows of a 3-dimensional bar chart.

In order to prove that the TCE did not evaporate, or leave the site by some unexplained phenomenon, the TCE material balance was considered important to understand the decrease of TCE concentrations in the clay *Lasagna*TM field experiment.

Figure F-4. Average TCE Concentrations Pre- and Post-*Lasagna*™ Field Experiment.

Figure F-5. TCE Concentrations Pre- and Post-*Lasagna*™ Field Experiment.

Carbon Analyses

Figure F-3 shows the locations of the carbon wickdrains of the field experiment. All shaded wickdrains also contained carbon sampling cassettes which were approximately 12" wide and 1" thick. They were enclosed in outer stainless steel frames which permitted the original inner cassettes to be easily removed. As mentioned later, one of the cassettes (C4-7) was removed after two months of operation of the field experiment and a fresh cassette was inserted into the holder. At the completion of the test, C4-7, along with all of the remaining cassettes, were removed for TCE analysis.

The naming convention for the cassettes combined the names shown at the top of Figure F-3 (Anode, C1, C2, etc.) with the cassette number which is shown to the right or left of each cassette. These numbers ranged from 1 to 9 for the anode and cathode cassettes, and from 1 to 12 for the wickdrain cassettes. The SOP which describes the procedures used for cassette sampling is shown in Appendix C.

Cassette C4-7

After the field experiment had been operated for over 2 months (after movement of

approximately 2 pore volumes of water), the sampling cassette in wickdrain C4-7 was removed and analyzed for TCE. This intermediate analysis was performed so that the Consortium could determine whether TCE was being absorbed by the carbon, and to estimate how long the experiment should be conducted before the Phase I site would be cleaned up. On March 20, 1995 the cassette was removed and sampled according to the protocol shown in Appendix C. The samples were sent to General Electric to perform transuranic isotope analysis of the samples before they were sent to Monsanto for analysis.

Appendix D summarizes the methods that were used to determine the amount of moisture in the carbon, and the extraction efficiencies for removing TCE from wet carbon using hexane, and Appendix E contains the protocol used for measuring total TCE in carbon cassette C4-7. Figure F-6 summarizes the concentrations measured. In this figure, the error bars are shown which indicate the range of concentrations measured in duplicate analyses. Rough, order-of-magnitude estimates, yielded the conclusion that almost all of the TCE in the contaminated clay had been removed and trapped in the carbon-filled wick drains.

Figure F-6. Concentrations of TCE Measured in Carbon Cassette C4-7 After 2 Pore Volumes.

All Cassettes

On March 20, after the first carbon-filled wick drain was removed for analysis, a fresh carbon-filled wick drain was inserted in the C4-7 cassette holder. This was done in order to determine whether additional TCE would be absorbed in the final months of operation of the field experiment. Figure F-7 shows the original TCE concentration data shown in Figure F-6, and the additional TCE data which was obtained from sampling on May 19 and 20 at the end of the experiment. This latter data is represented by the dark bars in Figure F-7 and are identified as "1 pore". As can be seen, very little additional TCE was absorbed by the freshly filled wick drain at depths less than 14 ft. during the additional 2 months of operation. This indicates that after 2 pore volumes of water movement, over 90% of the TCE had been moved into the carbon-filled wick drains.

The remaining 12 wick-drain cassettes were also removed from the field experiment on May

19 and 20. The results of the TCE concentrations measured in these samples, as a function of depth, are shown in Figures F-8 and F-9. Table F-3 also shows the concentrations of TCE measured, assuming 50% moisture and 33.3% extraction efficiency.

QA/QC for Carbon Analyses

Since the TCE concentration data in carbon was considered to be critical for the material balance calculations, a number of QA/QC procedures were implemented to insure that the Consortium would know the data quality of the TCE concentrations measured in the carbon cassettes. Appendix D outlines the procedures which were used to show that approximately 50% of the total wet carbon weight was due to water, and that approximately 33.3% of TCE is extracted from wet carbon, using the extraction protocol outlined in Appendix E. In addition to these studies, and in addition to all of the initial C4-7 cassette carbon samples in duplicate, additional measures shown in the next paragraph were also followed.

Blank solvent analyses were performed each day to insure that there was no low level TCE contamination that would cause our results to be biased high. Carbon was also taken from the bags of carbon at the beginning of the experiment before filling all of the wick drains. These blank carbon samples were used as

method blanks that were analyzed during carbon analysis to ensure that there were no unknown sources of contamination in the laboratory. Finally, the shallowest and deepest carbon samples in all 13 cassettes were analyzed in duplicate. The results of these QA/QC sample analyses are summarized in Appendix F.

Figure F-7. Concentrations of TCE Measured in Carbon Cassette C4-7 After 2 and 1 Pore Volumes.

Table F-3. Carbon TCE Concentrations ($\mu\text{g/g}$) (Corrected for Moisture and Recovery).

Depth	Control	Anode	C1	C2	C3	C4	Cathode
3			1,198	2,144	3,283	1,618	6,418
4	0	4,617	450	918	1,881	1,833	3,379
5	0	999	523	1,466	3,524	1,999	10,374
6	0	672	587	2,208	3,102	2,346	5,434
7	0	940	716	1,386	2,231	2,062	4,771
8	0	436	341	1,009	2,403	1,169	5,458
9	0	458	483	1,362	1,607	1,079	2,375
10	0	1,405	1,534	1,798	1,463	2,224	4,715
11	0	2,352	1,477	2,672	4,754	1,366	4,058
12	0	3,018	1,925	4,658	2,637	2,029	4,856
13	0	5,092	3,259	9,813	9,989	2,952	2,428
14		13,511	4,495	7,412	9,352	3,313	7,793
15		9,972	5,038	18,707	15,619	5,114	11,412
3	4,653	304	894	3,560	3,285	63	14,309
4	800	361	755	3,174	2,817	4,430	9,489
5	1,935	235	1,142	2,458	3,498	11,756	6,538
6	2,608	857	1,858	3,084	6,174	3,863	5,533
7	3,154	1,312	2,359	2,542	4,335	2,814	5,859
8	2,411	690	1,903	3,057	4,251	1,262	5,747
9	3,616	590	1,380	3,210	4,488	2,617	5,210
10	3,807	1,002	1,869	6,729	7,491	2,660	5,099
11	4,093	1,032	1,824	2,957	9,159	6,233	7,514
12	6,534	1,514	1,989	3,675	11,511	11,182	4,169
13	4,645	1,870	3,284	5,765	11,037	11,763	2,541
14	6,077	1,718	3,108	4,615	12,299	6,884	3,327
15	6,321	1,312	1,833	10,283	7,905	10,677	2,733

South Cartridges

North Cartridges

TCE Concentrations in Carbon Cassettes

Figure F-8. Concentrations of TCE Measured in all Carbon Cassettes after 3 Pore Volumes.

Figure F-9. Spatial Concentrations of TCE Measured in all Carbon Cassettes after 3 Pore Volumes.

The last line at the end of Appendix F shows average values measured for the TCE concentration, the RSD and the %RSD. (The RSD and %RSD were calculated from each duplicate pair. Normally these statistical functions should not be used quantitatively unless there are more than 5 points in the calculation. However, these values were calculated for the duplicate analyses for comparative purposes. They are meant to show the order of magnitude of the variability of the duplicate analyses.) Also note that the RSD and %RSD listed at the end of Appendix F are average values for these 25 pairs of duplicate analyses. The value of approximately 13% for %RSD indicates that analytical precision of the carbon analyses was probably an insignificant source of variability in the overall TCE material balance calculation.

Comparison with ORNL-Grand Junction Extract Results

A final set of analyses was performed in order to determine whether the ORNL-Grand Junction field analyses provided the same TCE concentrations as Monsanto's Environmental Sciences Center (ESC) laboratory-based analyses. During the post-*Lasagna*TM soil analyses, ORNL-Grand Junction also analyzed selected carbon samples using the analysis protocol shown in Appendix E. They also provided ESC with most of their set of extracts from their post-*Lasagna*TM soil samples. Figures F-10 and F-11 and Appendix H summarize the comparison of ORNL-Grand Junction and ESC analyses.

As can be seen from the figures and from Appendix H, for the 138 extracts compared, an RSD of approximately 18% was measured when comparing the ORNL-Grand Junction or ESC analyses to the average concentration from the

two laboratories. Twice that amount was observed between ORNL-Grand Junction and ESC results. Although this RSD of 18% corresponds to a %RSD of over 200%, Figure F-10 shows that most of this variability is due to uncertainty in the analysis of soils that have very low TCE concentrations. An RSD of 6% was measured for soil concentrations greater than 1 ppm, and an RSD of 20.6% was measured for soil concentrations less than 1 ppm.

The major data which was used to conclude that the *Lasagna*TM process produced final soil concentration < 1 ppm was based upon the low level analysis results. Although this is the concentration range of highest relative uncertainty, the absolute magnitude of uncertainty is such that analytical variability can be considered unimportant in arriving at the conclusions concerning cleanup in the field experiment.

It can also be assumed that, since the magnitude of TCE concentrations in carbon corresponds to the high TCE in soil

concentration data, that uncertainties on the order of 10% in the absolute magnitude of the TCE carbon concentrations would cause similar magnitude of error in the material balance calculations. The source of this variability is most likely due to differences in calibration solutions, since there appears to be a systematic constant of ~10% difference measured between ESC's values and the average values for TCE soil concentrations greater than 1 ppm.

Figure F-10 shows that this difference is constant over almost the entire range of TCE concentrations greater than 2 ppm. Figure F-11 shows a frequency histogram which shows that over 90% of ESC's TCE concentrations were between -6.4 and +10.3% of the average TCE concentrations. Therefore the material balance calculations which are based upon TCE concentrations measured in the field by ORNL-Grand Junction, and in the laboratory by ESC, should include errors no larger than approximately 20% attributable to analytical errors.

Figure F-10. Deviations of ESC TCE Concentrations from the Average.

Figure F-11. Histogram of the Frequency Distribution of Deviations in ESC TCE Concentrations from the Average.

Material Balance Calculations

In order to obtain a TCE material balance, calculations were performed to see whether the TCE which is removed from the soil is transferred into the carbon cassette which is located immediately behind the cleaned up soil, in the direction of the electro-osmotic flow. However, since all soil samples were not taken near all of the sampling cassettes and, as can be seen from Figure F-4, the soil TCE concentrations were highly variable, some method of soil TCE concentration estimation was needed.

The present section describes how the material balance calculations were carried out and shows these calculations, first assuming that the initial soil concentrations in front of the cassettes can be estimated from the soil boring nearest the cassette, and second assuming that the initial and final TCE concentrations in soil and carbon can be estimated using standard kriging techniques described in Appendix G.

Calculation Procedures

Figure F-12 describes how the various soil and carbon TCE concentrations were used in conjunction with the soil and carbon densities and dimensions of the field experiment, to arrive at a TCE mass balance. In all calculations, the soil weights were obtained from the field measurements of the wet soil. A density of the wet soil from the Paducah site was measured in our laboratory to be 2.0 g/cm^3 . All calculations involving the TCE concentrations in carbon are based upon a dry carbon density measured in our laboratory to be 0.5 g/cm^3 . TCE concentrations in carbon were calculated by correcting the carbon weights for 50% moisture as is shown in Appendix B. The amount of TCE which is present in a given weight of soil or carbon can be calculated from Equation 1.

$$\text{TCE}_{\text{total}} = (\text{soil or carbon weight}) \times \text{TCE}_{\text{concn}}$$

$$\text{TCE}_{\text{total}} = \text{volume} \times \text{density} \times \text{TCE}_{\text{concn}} \quad (1)$$

In order to compare the amounts of TCE in soil and carbon, a rectangular solid (shown in Figure F-12) was constructed which had a 1

cm² face and a length which was the width of the clay (21" or 53.3 cm) or the width of carbon (1" or 2.54 cm). The total amount of TCE in the soil, was calculated by multiplying the total volume of the rectangular soil solid (1 cm² x 53.3 cm) by the density of soil (2.0 g/cm³) by the TCE concentration in the wet soil. Similarly, the total amount of TCE in the carbon, was calculated by multiplying the total volume of the rectangular carbon solid (1 cm² x 2.54 cm) by the density of dry carbon (0.5 g/cm³) by the TCE concentration in dry carbon. The percent of TCE which is present in this volume of soil and which moves into this volume of carbon, can be used as a measure of the TCE material balance. Furthermore, if this calculation is performed at each depth and then averaged, the material balance for each cassette, and from this, the overall material balance for the entire experiment, can be calculated. For the present report, the material balance is reported as the percent of TCE in the soil ($TCE_{total,soil}$) which is measured in the carbon ($TCE_{total,carbon}$). For the data shown in this section and in Appendix G, the percent TCE absorbed by the carbon cassettes is used to measure the TCE material balance of the field experiment. A 100% material balance occurs if $TCE_{total,soil} = TCE_{total,carbon}$.

Discrete Soil Borings

Figure F-13 summarizes the material balance calculations which were carried out using equation 1 and soil boring concentrations of TCE in borings nearest to the carbon cassettes up-gradient from the electro-osmosis flow. Appendix I shows a table of total TCE amounts calculated using Equation 1 with discrete soil TCE concentrations measured in the nearest boring to the cassette, prior to the *Lasagna*TM field experiment. (All soil boring concentration data was taken from studies conducted by ORNL-Grand Junction and shown in Appendix B.) These calculations also assume that 100% of the TCE is removed from the soil and that the final soil concentrations were 0.0

ppm. This is very nearly that which was observed and is shown in Figure F-3.

The table in Appendix I is arranged so that each column labeled "Soil" immediately before each column labeled "Carbon" identifies which soil boring was used with each carbon cassette. For example, the Anode-5 carbon cassette TCE concentrations were compared to the LP16 soil boring TCE concentrations, the C1-7 cassette was compared to the L5 boring, etc.

The values shown in the columns labeled "Soil" give the total amount of TCE calculated to be present in a 21" x 1 cm² soil volume of soil at various depths. The values shown in the columns labeled "Carbon" give the total amount of TCE calculated to be present in a 1" x 1 cm² volume of carbon at various depths. The last line of the table shows the average TCE amounts in the soil and carbon volumes. Figure F-13 shows the results of calculating the percent TCE Absorbed by the carbon, using the TCE soil and carbon data in Appendix I.

Kriged Data

Figure F-14 shows a similar plot using data from Appendix G. In this figure, rather than averaging the amount of TCE in each soil boring and in each cassette, the kriged values of TCE amounts in the soil and carbon at different depths are calculated and the percent TCE Absorbed by the carbon as a function of depth is shown. More details of these kriging calculations and the table of data which is used to create Figure F-14 can be seen in Appendix G. Note that the last value shown in Figures F-13 and F-14 is the Average % TCE Absorbed for the total field experiment. The average percent TCE Absorbed using the discrete soil boring data and the kriged data are very similar (i. e. 62% versus 51%), so that the overall material balance for the field experiment of approximately 50% is irrespective of the method of estimating the concentrations.

Conclusions

The good agreement of the amount of TCE in the soil and that absorbed by the carbon, indicates that the *Lasagna*TM can be used to transfer TCE from contaminated soil into reaction zones. This fact will be exploited in the next phase of *Lasagna*TM process development for the Department of Energy.

Figure F-12. Diagram of Material Balance Calculation.

Figure F-13. Results of Material Balance Calculations using Discrete Soil Borings.

Figure F-14. Results of Material Balance Calculations using Kriged Data From Appendix G.

in Appendix L. Appendix J summarizes these results.

Analysis of Chlorinated Compounds in Soil and Water

During the post-experiment soil sampling and the final carbon wick-drain sampling, various soil and water samples were collected for analysis of chlorinated compounds other than TCE. This was done since there is a possibility that the original TCE that was released into the environment may have contained impurities, and also there are biological and non-biological TCE degradation processes that produce *cis*-1,2-dichloroethylene and vinyl chloride.

The water samples were split between ESC and the Paducah Gaseous Diffusion Plant Analytical Department. The ESC analyses for only TCE were performed using the SOPs shown in Appendix E and the PGDP analyses were performed using a purge-and-trap GC/MS analytical method similar to Method 624 shown

Soil samples were analyzed from the post-experiment sampling of the field experiment. The results of those analyses are shown in Appendix K. Attempts were also made to analyze selected carbon samples for chlorinated compounds found in the water and soil samples. Thermal desorption GC/MS analysis confirmed the presence of vinyl chloride and *cis*-1,2-dichloroethylene in addition to the TCE which was also discussed in previous sections. However, no quantitative measurements of these less-chlorinated compounds, due to limited sensitivity using the analytical methods developed for TCE analysis.

Conclusions

It is clear that vinyl chloride and *cis*-1,2-dichloroethylene are present at low concentrations in water, soil, and carbon

samples. However, the source of these lower chlorinated compounds is not known. GC/MS analyses were performed on the pre-experiment soil samples by the PGDP Analytical Laboratory. However, the analytical procedures involved significant dilution of the sample with methanol. Therefore low levels of detection were not achieved for vinyl chloride and *cis*-1,2-dichloroethylene.

However, the soil analysis data shown in Appendix K includes the analysis of soil borings taken outside of, and below the present *Lasagna*TM field experiment. If these samples can be taken as representative of the pre-experiment soil analyses, then it can be seen that *cis*-1,2-dichloroethylene which was detected in the post-experiment samples was also probably present before the experiment began.

If this is the case, *cis*-1,2-dichloroethylene and possibly vinyl chloride were probably not produced as a part of the *Lasagna*TM experiment. It is still unknown at this time if the source of these compounds was from the original TCE spill, or were produced subsequent to the spill, from known biological and/or non-biological degradation processes. It is hoped that the final phase of development of the *Lasagna*TM process will include complete degradation of all chlorinated compounds, regardless of the source. It is planned to use zero valent iron metal to degrade all aliphatic chlorinated compounds, removing the possibility of introducing these compounds into the water table below the PGDP site.

Conclusions

The present topical report shows the analytical chemical basis upon which the success of the *Lasagna*TM Phase I are based. This report documents from soil TCE concentration data taken before and after the field experiment, that electro-osmosis can be used on a field-scale to "pump" TCE into treatment zones over a distance of 21". In

addition, the TCE is moved almost quantitatively, with a measured material balance on the order of 50%. Furthermore, there does not appear to be any unexpected concentrations of other chlorinated compounds, as a result of electro-osmosis.

It is hoped that the Phase IIa field experiments, scheduled to begin in the summer of 1996, will show that the treatment zones can be constructed of materials which will ultimately degrade all chlorinated organic compounds into chloride and small, harmless organic molecules. This final demonstration will be used to demonstrate the widespread usefulness of the *Lasagna*TM process for remediating clay soils which contain aliphatic chlorinated compounds such as TCE.

Acknowledgments

There are a number of people that should be acknowledged for their support, without which this project could not have been successfully completed. In the laboratory, the carbon analyses were performed by Elaine M. Holland. Further technical discussions were also held with Robert G. Orth and David E. McKenzie. In the field, Van Rudolf of Asphalt Paving Incorporated and Bob Hines, CDM Federal Programs, Inc.'s Project Manager for this project, were critical in the field installation and daily maintenance of the site. Fraser Johnstone of Lockheed Martin Energy Systems, was critical for interfacing with regulatory agencies. Finally, all of Monsanto's Environmental Sciences Center provided organizational support and encouragement, which permitted us to participate in these field experiments.

Appendix A. ORNL-Grand Junction SOPs

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SOIL SAMPLING FOR FIELD SCREENING USING THE GEOPROBE AND U2CRT

1.0 PURPOSE

This procedure describes the use of the Geoprobe in conjunction with the U2CRT truck-mounted auger rig for collecting soil samples for field screening of contaminants.

2.0 APPLICABILITY

This procedure applies to the Geoprobe, a hydraulically driven probe that enables the operator to collect undisturbed soil samples to bedrock or refusal. The Geoprobe may be used to collect samples that provide a maximum of 450 g of soil from a 2-ft (0.6 m) sampler.

3.0 OTHER DOCUMENTS

3.1 REFERENCES

- 3.1.1 Procedure TE-105, U2CRT Operation
- 3.1.2 Procedure TE-072, Sample Packaging, Preservation, and Shipping
- 3.1.3 Procedure TE-071, Sample Documentation
- 3.1.4 Procedure TE-034, Chain of Sample Custody
- 3.1.5 Procedure TE-073, Equipment Decontamination

4.0 EQUIPMENT AND MATERIALS

4.1 EQUIPMENT

- 4.1.1 U2CRT
- 4.1.2 Push rods
- 4.1.3 Soil sample tube assembly
- 4.1.4 Sample logbook

APPROVALS	
Author	Date
Supervisor	Date
Group Leader <i>Mike Kato</i>	Date 8/4/93
Section Head <i>Chris Little</i>	Date 8/6/93

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4.1.5 Extension rods

4.1.6 Extruder rack

4.2 MATERIALS

4.2.1 Two 24-in. (0.6-m) bars

4.2.2 3/4-in.-diameter (1.9-cm) steel rod or wooden dowel

4.2.3 Square sample tubes

4.2.4 Wooden blocks

5.0 RESPONSIBILITY

5.1 It is the responsibility of the U2CRT operator to follow this procedure and to ensure that the equipment is operated safely.

5.2 It is the responsibility of the U2CRT technician to provide support for the U2CRT operator and to follow this procedure and all instructions of the operator.

5.3 It is the responsibility of the site project manager to direct the operator and technician to required sample locations and intervals.

6.0 DEFINITIONS

6.1 Geoprobe: a system for hydraulically driving rods and various sampling devices into the subsurface.

6.2 Geoprobe adapter assembly (sub): a device that adapts the U2CRT drive rod to the Geoprobe system.

6.3 Latch: a device that attaches to the adapter assembly to pull the Geoprobe rods out of the ground.

6.4 Hammer anvil: a device that fits into the Geoprobe adapter assembly and is used for pushing and hammering rods into the ground.

6.5 Soil sample tube: a thin-walled, shelby-type tube with a sharpened end; used for collecting soil samples.

6.6 Piston rod and point: a point and attached rod that slides into the soil sample tube. When held in place by the piston stop pin, no soil sample is collected; after removing the piston stop pin, the point and rod slide up into the sample tube allowing soil to enter the tube.

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- 6.7 Piston stop pin: a pin that threads (reverse) into the sample tube adapter and holds the piston rod and point in place.
- 6.8 Sample tube adapter: an adapter from soil sample tube to probe rods.
- 6.9 Extension rods: rods that fit inside the probe rods, the ends of which thread into piston stop pins, allowing the pin to be removed at the proper sample depth.

7.0 SAFETY

- 7.1 Strictly adhere to the safety instructions given in Procedure TE-105, U2CRT Operation.
- 7.2 All personnel within a minimum radius of the height of the drill mast must wear hard hats, steel-toed boots or shoes, and safety glasses or goggles.
- 7.3 Wear ear plugs or muffs with a minimum noise reduction rating of 15 dBA, during operation of the hydraulic hammer.
- 7.4 Use gloves during operation of U2CRT. Gloves are required when contact with sample material is anticipated.
- 7.5 KNOW THE LOCATION OF THE KILL SWITCH.

8.0 PROCEDURE

- 8.1 Following Procedure TE-105, prepare the U2CRT for Geoprobe sampling.
 - 8.1.1 Lift the latch and insert the hammer anvil into the Geoprobe.
 - 8.1.2 Push the latch back in order to hold in the hammer anvil.
- 8.2 Assemble the soil sample tube in the following way:
 - 8.2.1 Thread the piston rod onto the point.
 - 8.2.2 Slide the piston rod and point into the sample tube.
 - 8.2.3 Thread the sample tube adapter into the soil sample tube and thread (reverse thread) the piston stop pin into the sample tube adapter.
- 8.3 Prepare the probe rods for sampling:

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- 8.3.1 Thread a section of probe rod onto the sample tube and install the drive cap.
- 8.3.2 Position the sampling rod assembly below the hammer anvil.
- 8.3.3 Lower the drive rod and sub until the hammer anvil makes contact with the drive cap.
- 8.3.4 Push the sampling rod assembly into the ground, increasing the hydraulic pressure and/or the hydraulic hammer as needed.
- 8.3.5 Raise the drive rod and sub and connect a second Geoprobe rod.
- 8.3.6 Continue with this procedure until the desired depth is reached.
- 8.4 Extract the piston stop pin:
 - 8.4.1 Slide the drill rig mast forward to allow more working room, if possible.
 - 8.4.2 Remove drive cap.
 - 8.4.3 Using the necessary sections of threaded extension rods, remove the sampler piston stop pin by rotating the extension rods until the pin is free.
- 8.5 Collect the soil sample and remove from ground.
 - 8.5.1 Slide the drill rig mast back over the probe rods.
 - 8.5.2 Replace the drive cap and advance the Geoprobe rods the length of the sampler less 3 in. (7.6 cm) to compensate for the length of the sampler adapter and the piston point.
 - 8.5.3 Raise the drive rod and remove the hammer anvil.
 - 8.5.4 Thread the probe rod puller cap onto the probe rod.
 - 8.5.5 Lower the drive rod so that it is sitting directly on top of the rod puller cap.
 - 8.5.6 Latch the drive rod to the puller cap.

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GEOPROBE AND U2CRT

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- 8.5.7 Stack wooden blocks under the derrick feet or lower the "outriggers" on the U2CRT, if needed.
- 8.5.8 Slowly raise the drive rod and pull the probe rods from the ground.
- 8.5.9 Unthread each probe rod and lay to the side to avoid tripping hazards.
- 8.5.10 Install the probe rod puller cap and repeat the steps above until the sample tube has been retrieved to the surface.
- 8.6 Extract the soil sample:
 - 8.6.1 Place two 24-in. (0.6-m) bars across the derrick feet.
 - 8.6.2 Place the extruder rack on the bars and align it directly below the drive rod.
 - 8.6.3 Remove the sample tube from the probe rods; remove the piston rod, point, and sample tube adapter from the sample tube.
 - 8.6.4 Place the sampler in the extruder rack.
 - 8.6.5 Push the sample from the sample tube using a 3/4-in.-diameter (1.9-cm) steel rod or wooden dowel and the drive rod from the U2CRT.
- CAUTION: Perform this maneuver very carefully.
- 8.7 Package and preserve the sample according to Procedure TE-072 or according to special instructions from the site project manager.
- 8.8 Label and document samples according to Procedure TE-071 and Procedure TE-034, or as instructed by the site project manager.
- 8.9 Decontaminate the sample tubes according to Procedure TE-073 or site-specific procedures before re-use.
- 8.10 Decontaminate the probe rods and other downhole equipment according to Procedure TE-073 or site-specific procedures prior to use at another location.

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SOIL SAMPLING WITH THE MICROCORER

1.0 PURPOSE

This procedure describes the use of the microcorer, a small, hand-held, soil-extraction device.

2.0 APPLICABILITY

This procedure applies to sampling soils to be analyzed for volatile organic compounds (VOCs). The microcorer is often used to obtain gram-sized samples from larger samples, such as those extracted during soil boring or groundwater-monitoring-well drilling.

3.0 OTHER DOCUMENTS

3.1 REFERENCES

- 3.1.1 Procedure TE-072, Sample Packaging, Preservation, and Shipping.
- 3.1.2 Procedure TE-073, Equipment Decontamination.

3.2 EXHIBITS

- 3.2.1 Exhibit 1: Diagram of Microcorer

4.0 EQUIPMENT AND MATERIALS

4.1 EQUIPMENT

- 4.1.1 Microcorer sampler
- 4.1.2 Electronic balance
- 4.1.3 Automatic pipette

APPROVALS	
Author	Date
Supervisor	Date
Group Leader <i>[Signature]</i>	Date 8/4/93
Section Head <i>[Signature]</i>	Date 8/6/93

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4.2 MATERIALS

- 4.2.1 Sample logbook
- 4.2.2 40-mL glass vials and caps
- 4.2.3 Methanol, pesticide grade
- 4.2.4 Deionized (DI) water
- 4.2.5 Disposable pipette tips

5.0 RESPONSIBILITY

- 5.1 It is the responsibility of the field personnel collecting soil samples to follow this procedure if so directed by the site-specific sampling plan or the site project manager (SPM).

6.0 DEFINITIONS

- 6.1 **Microcorer:** a small, hand-held sampler used to collect a soil sample that will fit inside a 40-mL glass vial.

7.0 PROCEDURE

- 7.1 Clean and decontaminate all equipment and materials that will be used during sampling according to Procedures TE-072 and TE-073.
- 7.2 Determine an appropriate sampling point for VOCs as directed by the site-specific sampling plan or the SPM.
- 7.3 Using a pipette, introduce 10 mL of methanol or DI water into a pre-cleaned 40-mL glass vial, depending on what is called for in the site-specific sampling plan.
- 7.4 Cap and weigh the vial.
 - 7.4.1 Record weight on the vial or label.
- 7.5 Set microcorer for the desired sample size.
 - 7.5.1 Each line inscribed on the barrel indicates approximately 1 g, depending on the soil type to be sampled.
 - 7.5.2 Loosen the set screw, slide the stop to the desired line, and tighten the set screw.
- 7.6 Insert the microcorer into the soil to be sampled, ensuring that soil fills the barrel to the stop.

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- 7.7 Extrude the soil from the microcorer into the pre-weighed 40-mL vial.
- 7.8 Weigh the vial, including methanol (or DI water) and soil.
 - 7.8.1 Record weight in sample logbook and/or on the vial label as directed by the sampling plan or site project manager.
- 7.9 Decontaminate the microcorer according to Procedure TE-073.

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Exhibit 1. Diagram of Microcorer

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U2CRT OPERATION

1.0 PURPOSE

This procedure describes the set up and general operation of the ORNL-developed truck-mounted auger rig, the U2CRT.

2.0 APPLICABILITY

This procedure applies to drilling and sampling methods using the U2CRT.

3.0 OTHER DOCUMENTS

3.1 REFERENCES

- 3.1.1 Procedure TE-073, Equipment Decontamination
- 3.1.2 Procedure TE-106, Solid-Stem Augering Using the U2CRT
- 3.1.3 Procedure TE-107, Split-Spoon Sampling Using the U2CRT
- 3.1.4 Procedure TE-085, Water Sampling for Field Screening Using the Geoprobe and U2CRT
- 3.1.5 Procedure TE-086, Soil Sampling for Field Screening Using the Geoprobe and U2CRT

3.2 EXHIBITS

- 3.2.1 Exhibit 1: U2CRT with Mast Raised
- 3.2.2 Exhibit 2: U2CRT Operating Control Panel

4.0 EQUIPMENT AND MATERIALS

4.1 EQUIPMENT

- 4.1.1 U2CRT drill rig
- 4.1.2 Flexible exhaust pipe

APPROVALS	
Author	Date
Supervisor	Date
Group Leader	Date 8/4/93
Section Head	Date 8/6/93

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- 4.1.3 Hydraulic jacks
- 4.1.4 Screw jacks
- 4.1.5 Tool box

5.0 RESPONSIBILITY

- 5.1 It is the U2CRT operator's responsibility to operate all equipment in a safe and efficient manner. The operator should not leave the control panel when any part of the rig is moving or engaged.
- 5.2 It is the U2CRT technician's responsibility to perform tasks such as making connections, moving equipment, and assisting the operator in all aspects of rig operations while drilling, soil sampling, and installing piezometers so that the operator can concentrate on the operation of the rig.

6.0 DEFINITIONS

- 6.1 "A" rod: 2-in. hollow coupled rods that are driven into the borehole, forming the connection rod between the split-spoon and the drive hammer.
- 6.2 Control panel: the panel on the left rear of the drill rig containing controls for operating the drill rig.
- 6.3 Drill stem: all parts in the assembly used for drilling from the swivel to the bit, including the kelly, drill pipe/rods, augers, and bit.
- 6.4 Drive shoe: an adapter for driving split spoons that is threaded onto the hydraulic hammer on one end and to the "A" rod on the other.
- 6.5 Geoprobe adapter assembly (sub): an adapter that threads onto the drive rod that attaches the Geoprobe sampling system.
- 6.6 Hammer attachment: tooling to attach the hydraulic hammer to the "A" rod and to the drive shoe.
- 6.7 Hydraulic hammer: a hydraulically-powered piston hammer located on the middle of the mast used to drive split-spoon samplers and Geoprobe rods into the ground.
- 6.8 Hydraulic splitter: a device, operated with a push/pull button, that diverts the flow of hydraulic oil from one system to another; only one system is active at a time.
- 6.9 Kelly: a hollow steel bar that is the top section of the drill stem and transmits power directly from the rotary motor to the auger flights.

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- 6.10 Kill switch: a safety switch used to turn off the drill rig in an emergency.
- 6.11 Mast: the portable derrick, capable of being erected as a unit, that is upright during drilling or sampling.
- 6.12 NFPA: National Fire Protection Association.
- 6.13 P.T.O. (Power Take Off): a mechanism that converts power from the truck engine to the hydraulic system that powers all functions on the rig.
- 6.14 RPM: revolutions per minute.
- 6.15 Table: the flat, movable surface at the back of the drilling rig.

7.0 SAFETY

- 7.1 Inspect the first aid kit for proper bandages, eye wash, and burn dressings.
- 7.2 Check the fire extinguisher for NFPA-type ABC extinguishing agents and determine that it is fully charged.
- 7.3 Look for overhead and underground utilities within the site and review utility maps to determine safe locations for soil borings.
- 7.4 Ensure a safe distance between the mast and overhead electric power lines.
 - 7.4.1 Do not operate drilling equipment closer than 10 ft to electrical transmission lines.
- 7.5 Confirm that no unnecessary personnel or support vehicles are within the zone having a minimum radius of the height of the drill mast.
- 7.6 Check the drill rig for contamination, damaged cables, loose bolts, frayed wires, and leaks.
 - 7.6.1 Wrap hydraulic lines if necessary.
 - 7.6.2 Check drill stems for contamination and decontaminate as necessary. See Procedure TE-073.
- 7.7 Watch for pinch points, cables and hoses hanging up, loose equipment lying on the mast, and other hazards as the drill mast is being raised.
- 7.8 Wear hard hats, steel-toed boots, and safety glasses or goggles within a minimum radius of the height of the drill mast.

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- 7.9 When performing any activities within the area having a minimum radius of the height of the drill mast, approach the drill rig only after confirming that the operator is aware of approach and that drill stems, augers, or split-spoons have been secured on the ground, on racks, or safely supported next to the drill mast.
 - 7.10 Avoid touching the drill rig when auguring in areas of potential electrical hazard.
 - 7.10.1 The operator should contact only the control knobs in these situations.
 - 7.11 When drilling operations are performed in areas of suspected underground pipelines, vaults, or other buried objects, use extreme caution when noting unusual auger sounds, change in speed, change in depth rate, or directional deviations.
 - 7.11.1 A change in auger soil cuttings from normal lithology to back-fill material (especially sand) will be cause to suspend auguring operations until the cause of the lithology change can be determined and safe working conditions can be assured.
 - 7.12 KNOW THE LOCATION OF THE KILL SWITCH.
- 8.0 PROCEDURE
- 8.1 Set up the drill rig. See Exhibit 1.
 - 8.1.1 Locate the rig across the fall line of the terrain, rather than nose up or nose down.
 - 8.1.2 Place the transmission of the truck in neutral.
 - 8.1.3 Set the emergency brake on the truck.
 - 8.1.4 Using screw jacks mounted on each side of the rear of the truck, level the rig from side to side.
 - 8.1.5 Attach flexible exhaust pipe to tailpipe of truck.
 - a. Position the end of this hose downwind from the rig and away from personnel and sampling equipment.
 - 8.1.6 Engage the P.T.O.

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- a. The P.T.O. is located in the cab of the truck and provides power to the hydraulics of the drill rig.
 - b. Leave the engine at idle speed.
- 8.1.7 To engage the hydraulics for the mast, press down the button located in the upper left corner of the control panel marked ACCY HYD - MAST.
 - a. See Exhibit 2 for control panel description.
- 8.1.8 Using the lever located in the lower left corner of the control panel marked MAST-RAISE/LOWER, slowly engage the hydraulics by moving the lever to raise the mast.
- CAUTION: WATCH ALL HYDRAULIC LINES SO THAT THEY DO NOT BECOME ENTANGLED IN TOOLS OR OBSTRUCTIONS IN THE TRUCK BED.
- 8.1.9 After the mast is in the full upright position, lock the mast in place by installing the pin through holes on the drill table and through the turn buckle attached to the mast.
- 8.1.10 Set the engine RPMs to 1900 using the throttle control inside of the truck on the P.T.O.
 - a. Watch the tachometer located on the left side above the dashboard to find the RPM setting.
- 8.2 To auger with U2CRT, see Procedure TE-106, Solid-Stem Auguring Using the U2CRT.
- 8.3 To soil sample with U2CRT, see Procedure TE-107, Split-Spoon Sampling Using the U2CRT.
- 8.4 See Procedures TE-085 and TE-086 for operation of the U2CRT during Geoprobe sampling.

U2CRT OPERATION

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Exhibit 1. U2CRT with Mast Raised

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Exhibit 2. U2CRT Operating Control Panel

- A. ACCY/HYD - MAST: hydraulic splitter control; when in the up position, the rotary hydraulic motor or the hydraulic hammer is activated; when in the down position, the hydraulics are diverted to the mast hydraulic ram.
- B. Power switch: emergency kill switch for the drill rig; when the switch is pushed away from the operator, power is on; when it is pulled toward the operator, power is off.
- C. HYDRAULIC SYSTEM PRESSURE: pressure dial for the hydraulic system; the operator should not exceed 2000 psi when operating the system.
- D. HAMMER FLOW CONTROL-ROTARY SPEED: controls hydraulic pressure to either the hydraulic hammer or the rotary motor, depending on which is selected by the hydraulic splitter on the mast. It also acts as a splitter between the hydraulic hammer or the rotary motor and the hydraulic rams that move the kelly up and down. The closer the operator moves the lever towards him, the less hydraulic pressure provided to the kelly.
- E. HAMMER/ROTARY: hydraulic splitter button that turns on the hydraulic hammer or the rotary motor, depending on which has been selected using the hydraulic splitter button on the mast. When in the OFF position, the hydraulics are diverted to the Table Slide.
- F. MAST: toggle lever that raises and lowers the mast when the hydraulic splitter button ACCY/HYD - MAST is engaged to MAST.
- G. FEEDER FLOW CONTROL: a throttle that controls the downward movement of the auger during drilling operations.
- H. WORK LIGHTS: toggle switch that operates the rig's overhead work lights; when the switch is pushed away from the operator, the lights will be activated; when switch is towards the operator, the lights they will be off.
- I. WINCH: switch that operates the electric winch line located on top of the mast; twisting the switch clockwise reels the winch line in; counter-clockwise lets the line out.
- J. FEEDER: toggle lever that raises and lowers the hydraulic cylinders for both the hydraulic hammer and the rotary motor.

U2CRT OPERATION

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Exhibit 2 (continued)

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FIELD OPERATION OF THE HEWLETT PACKARD 5890 SERIES II GAS CHROMATOGRAPH

1.0 PURPOSE

This procedure describes the use of the Hewlett Packard (HP) 5890 Series II gas chromatograph (GC) for analysis of environmental pollutants.

2.0 APPLICABILITY

This procedure applies to the HP 5890 Series II gas chromatograph (GC), which is equipped with a packed injector and a split/splitless injector. The GC includes two detectors: an electron capture detector (ECD) and a photoionization detector (PID). The unit is configured for two 30-m, 60-m, or 120-m megabore capillary columns (0.53-mm I.D.).

3.0 OTHER DOCUMENTS

3.1 REFERENCES

- 3.1.1 Hewlett Packard 5890 Series II Gas Chromatograph operating manual.
- 3.1.2 HP 3396 Series II Integrator operating manual.
- 3.1.3 Procedure TE-169, Using the Chrom Perfect Gas Chromatograph Software.

3.2 EXHIBITS

- 3.2.1 Exhibit 1: Example of Chromatogram and Report

4.0 EQUIPMENT AND MATERIALS

4.1 EQUIPMENT

APPROVALS	
Author <i>[Signature]</i>	Date 9/15/95
Supervisor <i>[Signature]</i>	Date 10-9-95
Group Leader <i>[Signature]</i>	Date 10-9-95
Section Head <i>[Signature]</i>	Date 10/16/95

FIELD OPERATION OF THE HEWLETT PACKARD 5890 SERIES II GAS CHROMATOGRAPH	Number: TE-160 Page: 2 of 6
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- 4.1.1 HP 5890 Series II gas chromatograph
- 4.1.2 HP 3396 Series II integrator
- 4.1.3 Carrier gas bottles
- 4.1.4 In-line oxygen trap
- 4.1.5 Moisture trap
- 4.1.6 Forceps
- 4.1.7 Set of box and open-end wrenches
- 4.1.8 Personal computer (PC) when using Chrom Perfect software

4.2 MATERIALS

- 4.2.1 Liquid leak detector
- 4.2.2 Non-liquid leak detector
- 4.2.3 Capillary column
- 4.2.4 Septums

5.0 RESPONSIBILITY

- 5.1 It is the responsibility of the operator of the HP 5890 Series II gas chromatograph to follow this procedure.

6.0 DEFINITIONS

- 6.1 Carrier gas: gas that serves to transport the standard or sample through the column.
- 6.2 Chromatogram: a graphic display, in the form of peaks, of the detector's response to a given standard or sample.
- 6.3 Electron capture detector (ECD): a GC detector that measures a decrease in electrical signal rather than an increase in electrical current. The loss of current is proportional to the amount of compound present. The ECD is virtually insensitive to hydrocarbons. It is especially valuable for the analysis of pesticides, PCBs, and chlorinated solvents.
- 6.4 Photoionization detector (PID): a GC detector in which the GC column effluent gas stream is subjected to ultraviolet radiation energetic enough to ionize many organic compounds in the gas stream. Since each organic molecule has a characteristic ionization potential (the energy required to remove an electron from a molecule), the PID can be tuned to detect certain classes of organic compounds.
- 6.5 Retention time: the elapsed time from the injection of a standard or sample (when START is activated) until detection.

FIELD OPERATION OF THE HEWLETT PACKARD 5890 SERIES II GAS CHROMATOGRAPH	Number: TE-160 Page: 3 of 6
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7.0 PROCEDURE

7.1 Start up the GC following the steps below:

7.1.1 Connect the GC to the carrier gas bottles.

Note: Nitrogen, if used with the ECD, must be at least 99.9995% hydrocarbon-free.

7.1.2 Install the moisture trap and in-line oxygen trap as close to the injector as possible to ensure carrier gas purity.

7.1.3 Install the column. See the GC operating manual, Reference 3.1.1, for installation instructions.

7.1.4 Test all connections for leaks.

a. Use a liquid leak detector such as Snoop™ on the connections to the oxygen trap.

b. Use a non-liquid leak detector on all gas-line and column connections past the oxygen trap.

7.1.5 Set the desired column flow rate and the total flow output, referring to the operating manual, Reference 3.1.1.

Note: Recommended column flow rates are between 5 and 15 mL/min but may be outside this range depending on the type of analysis performed. When using the ECD detector, total flow must be greater than 50 mL/min.

7.1.6 If using a new column, condition the column according to the manufacturer's instructions.

7.1.7 Enter the oven ramping program and the injector and detector temperatures, referring to the operating manual.

Example: for the VOCOL column with the ECD detector enter:

ECD temperature: 250°C

Injector temperature: 175°C

Program: 35°C for 4.00 min

Ramp at 4.0°C/min to 100°C

Hold at 100°C for 1.00 min

Ramp at 20°C/min to 170°C

Hold at 170°C for 12.00 min

FIELD OPERATION OF THE HEWLETT PACKARD
5890 SERIES II GAS CHROMATOGRAPH

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Note: The final temperature and hold time ensure that the column is free of residual contamination.

- 7.1.8 Turn on the detector injector and oven and allow the GC to stabilize at operating temperatures.

Note: Stabilization time will depend on how long the GC has been turned off. A recommended baseline signal of the ECD is less than 20 mV.

- a. Referring to the operating manual, troubleshoot the instrument if the baseline is unstable.

- 7.1.9 Set up the HP 3396 Integrator.

- a. Connect the two INET cables from the back of the integrator to the instrument block on top of the GC.
- b. Set the current date and time.
- c. If using the Chrom Perfect software with the integrator and PC, see Procedure TE-169.

- 7.2 Calibrate the GC. Refer to the specific method to be used for calibration procedures.

- 7.3 Analyze samples. Refer to the specific method to be used for analysis procedures.

- 7.4 When contamination of the column or detector(s) is indicated by an upward drift in the signal level output, bake out either or both of these with the following steps:

- 7.4.1 Increase the column temperature to 190°C to drive off residual contamination within the column.

- 7.4.2 Increase the temperature of the ECD to 275°C and the PID temperature to 225°C to purge contaminants from the detectors.

- 7.5 Replace the septum at the end of the day in order for the GC to stabilize overnight.

- 7.5.1 Turn off the detectors.

- 7.5.2 Lower the injector and detector temperatures to 35°C.

FIELD OPERATION OF THE HEWLETT PACKARD 5890 SERIES II GAS CHROMATOGRAPH	Number: TE-160 Page: 5 of 6
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7.5.3 Unscrew the septum retaining nut and remove the septum using forceps or a clean syringe.

7.5.4 Quickly install a new septum and replace the retaining nut. DO NOT OVERTIGHTEN THE RETAINING NUT.

7.6 Turn off the instrument following these steps:

7.6.1 Lower the oven, injector, and detector temperatures to 35°C.

7.6.2 Turn off the detectors.

7.6.3 Turn off the oven.

7.6.4 Turn off the GC.

FIELD OPERATION OF THE HEWLETT PACKARD
5890 SERIES II GAS CHROMATOGRAPH

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Exhibit 1. Example of Chromatogram and Report

File=C:\CP\DATA1\TUCSII.62R Sample name=SV129 Date printed= 12-11-1993 Time= 11:41:23
0.00 to 15.00 min. Low Y = -0.08749 mv High Y = 0.41251 mv Span = 0.50000 mv

C:\CP\DATA1\Q78EA97B.BNC
Data file = C:\CP\DATA1\TUCSII.62R
Date stamp = 12/11/93 Time = 11:41:16
Sample name = SV129
Collected on DEC 11, 1993 11:26:50 from port # 1
Operator = J.L. ZUTMAN
Reference file name = Q78EA97Bb#66
Instrument = HP5890
Method name = C:\CP\DATA1\TUCIIAIR.MET version # 3
Date method last modified = 12/08/93 Time = 16:41:38
Calibration file = C:\CP\DATA1\TUCIIAIR.CAL version # 10
Date cal file last modified = 12/08/93 Time = 17:34:38
Run time = 13.67 minutes Area reject = 100
Amount injected = 1 Dilution Factor = 1
Sample Weight = 1 Internal Standard Amount = 0
Sampling rate = 10 per second
Peak detect threshold = 0 Starting peak width = .04 minutes
Chrom-Perfect Software Serial # 12948 Version = 5.05 For Martin Marietta
Today's date = 12-11-1993 Time = 11:41:47

PK	Ret Time	Name	Amount	Amount %	Area	Area %	Type	Width	Height	Height %
1	0.888		0.0000	0.0000%	19,654.3	26.316%	BB	0.042	7,861.79	57.9427%
2	2.280		0.0000	0.0000%	701.5	0.939%	BB	0.096	122.10	0.8999%
3	5.505	1,1,1,1-TCA	11.7708	0.4479%	183.0	0.245%	BB	0.150	20.31	0.1497%
4	7.737	TCE	1,204.8097	45.8427%	12,558.5	16.815%	BB	0.154	1,356.37	9.9966%
5	12.575	TETRACHLOROETHENE	1,411.5570	53.7094%	41,589.7	55.685%	BB	0.165	4,207.65	31.0111%
Total area = 74686.94			Total amount = 2628.137		Sample units = ug/l		Total height = 13568.23			

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PREPARATION OF CALIBRATION STANDARDS FOR GC ANALYSIS OF VOCs

1.0 PURPOSE

This procedure describes the preparation of calibration standards for analysis of samples for volatile organic compounds (VOCs) using a gas chromatograph (GC).

2.0 APPLICABILITY

This procedure applies to the Hewlett Packard (HP) 5890 Series II GC and Photovac 10S50 GC for VOC analysis using the headspace method for water and the solvent extraction method for water and soil.

3.0 OTHER DOCUMENTS

3.1 REFERENCES

- 3.1.1 *Test Methods for Evaluating Solid Waste*, Methods 8010 and 8020. SW-846. U. S. Environmental Protection Agency.

4.0 EQUIPMENT AND MATERIALS

4.1 EQUIPMENT

- 4.1.1 Top-loading balance accurate to 0.01 g
4.1.2 Assorted volumetric flasks, 10 to 1000 mL
4.1.3 Graduated cylinder

4.2 MATERIALS

- 4.2.1 Syringes (10 μ L, 25 μ L, 50 μ L, 100 μ L, 250 μ L)
4.2.2 15-mL vials
4.2.3 Pipettes (10-100 μ L, 50-250 μ L, 100-1000 μ L) with disposable tips

APPROVALS	
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Supervisor <i>[Signature]</i>	Date 9-12-95
Group Leader <i>[Signature]</i>	Date 9-12-95
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PREPARATION OF CALIBRATION STANDARDS FOR GC ANALYSIS OF VOCs	Number: TE-164 Page: 2 of 4
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- 4.2.4 40-mL VOA vials
- 4.2.5 GC capillary-grade solvent, usually methanol or hexane
- 4.2.6 Solvent and water dispensers
- 4.2.7 Certified standards
- 4.2.8 Deionized (DI) water

5.0 RESPONSIBILITY

- 5.1 It is the responsibility of the field laboratory analyst to follow this procedure.

6.0 DEFINITIONS

- 6.1 Calibration Standards: known concentrations of analytes of interest used to establish a calibration curve from which unknown concentrations in a sample may be determined.
- 6.2 Solvent: a liquid used for extracting chemical contaminants from soil or water. Also used as a matrix for calibration standards and as a preservative for VOCs.

7.0 PROCEDURE

- 7.1 Prepare calibration standards in water for headspace method.

- 7.1.1 Calculate the amount of prepared standard needed to add to a volume of water to obtain the desired concentration.

Note: At least three calibration concentrations are needed in order to generate a calibration curve. For quantitative results, a five point calibration is needed. Normal concentrations for VOCs in commercially available standards are 200 µg/mL (200 ppm) but may vary depending upon the manufacturer. Check the certification sheet attached to the standard ampule for the correct concentration.

$$\frac{M_1}{V_1} = \frac{M_2}{V_2}$$

where

M_1 = mass of analyte to be added, µg

V_1 = volume of diluent, mL

$\frac{M_2}{V_2}$ = desired concentration of standard, µg/mL

PREPARATION OF CALIBRATION STANDARDS FOR GC ANALYSIS OF VOCs

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Example: A concentration of 500 ppb (500 $\mu\text{g}/1000\text{ mL}$) in 100 mL of diluent is needed:

$$\frac{M_1}{100\text{ mL}} = \frac{500\text{ }\mu\text{g}}{1000\text{ mL}}$$

$M_1 = 50\text{ }\mu\text{g}$ of analyte needed

Using prepared stock standard of 200 ppm (200 $\mu\text{g}/1\text{ mL}$),
 $50\text{ }\mu\text{g} \times 1\text{ mL}/200\text{ }\mu\text{g} = 0.25\text{ mL} = 250\text{ }\mu\text{L}$.
Therefore, 250 μL of prepared stock standard is needed.

7.1.2 Fill a 100-mL flask with DI water.

7.1.3 Using a clean syringe, remove the same amount of DI water from the flask that will be injected with the prepared standards. For example, if 25 μL of prepared standard is needed to obtain the desired concentration, remove 25 μL of water from the flask prior to adding the standards.

7.1.4 Using a syringe dedicated for calibration standards, withdraw the desired amount of prepared standards from the ampule and transfer it to the flask.

Note: The end of the needle of the syringe must be below the surface of the water. Do not contact the sides of the flask.

7.1.5 Mix by inverting the flask three times.

7.1.6 Transfer the contents of the flask to 40-mL VOA vials leaving zero headspace.

7.1.7 Cap the vials and refrigerate until needed.

Note: The aqueous standards can be stored up to 24 hours.

7.2 Prepare calibration standards in solvent for direct injection method.

7.2.1 Calculate the amount of prepared standard needed to add to a volume of solvent to obtain the desired concentration.

Note: The calculation is the same as outlined in 7.1.1, but the volume of solvent will be substantially less than that of water. Normally, 10 or 20 mL of solvent is used.

PREPARATION OF CALIBRATION STANDARDS FOR GC ANALYSIS OF VOCs

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7.2.2 Add the desired volume of solvent to a 15 or 40-mL vial using a graduated cylinder.

- a. Weigh the amount of solvent added to the vial and multiply by the density of the solvent in order to ensure that the correct amount has been added.

Example: Tare weigh an empty 15-mL vial. Add 10 mL of solvent to the vial using a graduated cylinder or dispenser. Re-weigh the vial. Assuming hexane is being used as the solvent, the density of hexane at 20° C is 0.66. Therefore, the weight of 10 mL is 6.60 g (assuming a room temperature of 20° C).

- b. In order to reduce evaporation of the solvent during the standard preparation, place about 100 mL of solvent into a volumetric flask.
- c. Cap the flask and store refrigerated until the solvent is cold, about 2 hours.
- d. Use this solvent for preparing the standards.

7.2.3 Follow instructions in 7.1.3 and 7.1.4.

7.2.4 Cap the vials. Store refrigerated until needed.

Note: Calibration standards prepared in a solvent matrix are more stable than aqueous mixtures. Tightly capped and refrigerated, the standards are usable for up to two weeks.

7.2.5 See Reference 3.1.1 for further information.

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GC ANALYSIS OF VOCs USING SOLVENT EXTRACTION

1.0 PURPOSE

This procedure describes the analysis of soil and water samples in the field using a gas chromatograph (GC) and solvent extraction.

2.0 APPLICABILITY

This procedure applies to the Hewlett Packard (HP) 5890 Series II GC, the HP 7673A autosampler, and the HP 3396 integrator. The GC is equipped with a 30-m capillary column, two injector ports (packed and split-splitless), and two detectors configured for parallel operation [the electron capture detector (ECD) and the photoionization detector (PID)]. The autosampler is mounted over injector B, the split-splitless injector, and is controlled via a sequence file on the integrator. Chromatograms are generated using Chrom Perfect software.

3.0 OTHER DOCUMENTS

3.1 REFERENCES

- 3.1.1 HP 5890 Series II GC operating manual
- 3.1.2 HP 7673A autosampler operating manual
- 3.1.3 Chrom Perfect operating manual
- 3.1.4 HP 3396 Series II integrator operating manual
- 3.1.5 Procedure TE-160, Field Operation of the Hewlett Packard 5890 Series II Gas Chromatograph
- 3.1.6 Procedure TE-164, Preparation of Calibration Standards for GC Analysis of VOCs
- 3.1.7 Procedure TE-169, Using the Chrom-Perfect Gas Chromatograph Software

APPROVALS	
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Group Leader <i>[Signature]</i>	Date 9/14/95
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GC ANALYSIS OF VOCs USING SOLVENT EXTRACTION

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3.2 EXHIBITS

3.2.1 Example of Chromatogram and Report

4.0 EQUIPMENT AND MATERIALS

4.1 EQUIPMENT

- 4.1.1 HP 5890 Series II GC
- 4.1.2 HP 3396 Series II integrator
- 4.1.3 HP 7673A autosampler
- 4.1.4 GC logbook
- 4.1.5 Top-loading balance accurate to 0.01 g

4.2 MATERIALS

- 4.2.1 Gas-tight syringes (manual liquid and autosampler liquid)
- 4.2.2 Autosampler vials
- 4.2.3 40-mL VOA sample vials
- 4.2.4 15-mL dilution vials
- 4.2.5 Pipettes (10-100 μ L, 50-250 μ L, 100-1000 μ L) with disposable tips
- 4.2.6 Capillary GC-grade solvent, usually methanol or hexane
- 4.2.7 0.53 $\times$ 30-m megabore capillary column
- 4.2.8 Solvent and water dispensers
- 4.2.9 Deionized (DI) water
- 4.2.10 Waterproof labels
- 4.2.11 5 and 10-mL disposable syringes
- 4.2.12 Certified calibration standard

5.0 RESPONSIBILITY

- 5.1 It is the responsibility of the operator of the HP 5890 Series II GC to follow this procedure.

6.0 DEFINITIONS

- 6.1 Calibration standards: known concentrations of analytes of interest used to establish a calibration curve from which unknown concentrations in a sample may be determined.
- 6.2 Calibration verification: a QC sample analyzed once per day, preferably before analyzing a set of samples.
- 6.3 Chromatogram: a graphic display, in the form of peaks, of the detector's response to a given standard or sample.

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- 6.4 Matrix spike: a QC sample analyzed once per day.
- 6.5 Quality check: a QC sample analyzed once per day.
- 6.6 Reagent blank: a quality control (QC) sample analyzed a minimum of once every 20 samples and prior to initial calibration or daily calibration verification. This blank serves to verify that the solvent used is free of contaminants. It also verifies that the GC is free of contaminants and carry-over.
- 6.7 Replicate sample: a QC sample analyzed once every 20 samples.
- 6.8 Solvent: a liquid used for extracting chemical contaminants from soil or water. Also used as a matrix for calibration standards and as a preservative for volatile organic compounds.

7.0 PROCEDURE

- 7.1 Prepare soil sample vials before going into the field.
 - 7.1.1 Calibrate the solvent dispenser before using.
 - a. Adjust the dispenser to 5.0 mL.
 - b. Tare a 40-mL VOA vial on the balance.
 - c. Dispense 20 to 30 mL of solvent into the tared vial.
 - d. Weigh the vial and calculate the volume by multiplying the weight of the solvent by the density of the solvent.
 - e. Adjust the dispenser accordingly.
 - 7.1.2 Using the water dispenser, dispense 5 mL of DI water into 40-mL VOA vials.
 - 7.1.3 Using the solvent dispenser, dispense 5 mL of capillary GC-grade solvent into the same vials.
 - 7.1.4 Immediately cap the vials.
 - 7.1.5 Attach waterproof labels to the 40-mL vials.
 - 7.1.6 Weigh each vial to the nearest 0.01 g on the top-loading balance and record the weight on the label.

GC ANALYSIS OF VOCs USING SOLVENT EXTRACTION	Number: TE-165 Page: 4 of 11
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7.1.7 Store the vials in a refrigerator or cooler containing Blue Ice until needed.

7.2 Set up the GC as outlined in Procedure TE-160.

7.3 Set up the HP 7673A autosampler.

7.3.1 Attach the autosampler to the GC following procedures outlined in the HP 7673A autosampler operating manual, Reference 3.1.2.

7.3.2 Connect the INET cables between the GC, the integrator, and the autosampler controller as outlined in the autosampler operating manual.

7.3.3 Attach a 5 or 10- μ L autosampler syringe to the tower of the autosampler as outlined in the autosampler operating manual.

7.3.4 Fill solvent wash vials A and B with solvent.

7.3.5 Place the vials in the respective holders on the autosampler turret.

7.3.6 Place empty solvent waste vials A and B in the respective holders on the turret.

7.4 Calibrate the GC.

7.4.1 Prepare calibration standards following Procedure TE-164.

- a. Prepare a minimum of three calibration standards at concentrations at the low, middle, and high range of expected concentrations of the samples.

Note: It is recommended that a 5- or 6-point calibration curve be used with concentrations of 10, 50, 100, 250, 500, and 750 ppb. This range of standards establishes a good linear calibration curve. It has been found that concentrations above 750 ppb are beyond the linear range of the instrument.

7.4.2 Transfer 1 mL of the calibration standards to the autosampler vials using a pipette.

- a. Use a new pipette tip for each transfer.

GC ANALYSIS OF VOCs USING SOLVENT EXTRACTION	Number: TE-165 Page: 5 of 11
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- b. Mark the concentrations on the autosampler vials and place them in the autosampler tray.

7.4.3 Set up the options of the autosampler using the integrator.

Note: The integrator controls the autosampler through a sequence file. Commands contained in the file are sent to the autosampler controller via INET cables connecting the integrator, autosampler, and GC.

- a. Press SHIFT and SEQ simultaneously.
- b. The integrator responds with:
PREP SEQ
ALS INFORMATION
INET SAMPLER CONTROL {Y*/N}
Press Y and return.

- c. Continue through the list of prompts, referring to the autosampler operating manual for details.

7.4.4 Set up the Chrom Perfect sequence file, referring to the Chrom Perfect operating manual (Reference 3.1.3) for sequence file directions.

Note: This file is used to control the sample information listed on the chromatograms and should not be confused with the sequence file generated on the integrator to control the autosampler.

7.4.5 Analyze the calibration standards.

- a. On the integrator, press SEQ then START. The GC, integrator, autosampler, and Chrom Perfect software will be activated together.

7.4.6 At the end of the calibration, generate a calibration file using data obtained from the standards analysis (see Reference 3.1.7).

- a. Determine that the resulting calibration curve has a linear correlation coefficient greater than 0.98 (this value will vary depending upon the response of the instrument to the chemical component).

GC ANALYSIS OF VOCs USING SOLVENT EXTRACTION	Number: TE-165 Page: 6 of 11
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- b. If the correlation coefficient is less than 0.98, prepare new standards and re-calibrate.

7.4.7 Record the results of the calibration standards analysis in the GC logbook.

- a. Include the area and concentration for each component and the amount injected into the GC.

7.5 Analyze the soil samples.

7.5.1 Ensure that the amount of sample injected is equal to that of the calibration standards, under normal circumstances 1 μ L.

Note: Samples that are suspected of being highly contaminated must be diluted in order to remain within the linear range of the GC. Hand-held PID measurements taken in the field at the time of sampling may be useful in determining a good dilution factor. If in doubt, it is better to over-dilute.

- a. For a 1:10 dilution, dispense 4.5 mL of solvent using the solvent dispenser into a 15-mL vial. Using a pipette, withdraw 500 μ L of solvent, the upper liquid layer, from the sample vial and transfer to the 15-mL vial.
- b. For a 1:100 dilution, dispense 4.5 mL of solvent into a 15-mL vial. Withdraw 500 μ L of the 1:10 diluted sample and transfer to the 15-mL vial.
- c. For a 1:1000 dilution, follow this same procedure using the 1:100 diluted sample.

7.5.2 Transfer 1 mL of the sample (diluted if necessary) into an autosampler vial using a pipette.

7.5.3 Analyze the sample by repeating steps 7.3.3 through 7.3.5 above.

Note: The amount of sample injected into the GC must be equal to the amount of calibration standard injected during calibration.

7.5.4 On the integrator, press SEQ then START.

7.5.5 Examine the chromatograms. If the area counts of the contaminant peaks do not fall within the calibrated range of the GC, reanalyze the sample at a different dilution.

GC ANALYSIS OF VOCs USING SOLVENT
EXTRACTION

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Example: The GC is calibrated for trichloroethene (TCE) using 50, 100, 250, 500, and 750 ppb concentration calibration standards. The detector response in area counts for these concentrations are 4779, 9184, 21,063, 38,997, and 56,155 respectively. The results of a sample analysis show a TCE concentration of 5000 ppb with a detector response in area counts of 340,000. The area counts exceed the area counts (56,155) of the highest calibration standard, 750 ppb. The sample is diluted 1:10 and reanalyzed. Results of this analysis show a TCE concentration of 5025 ppb, with area counts of 39,250, which falls within the calibrated range of the GC. The GC software will automatically calculate the correct concentration for the specified dilution factor.

7.5.6 Record the concentrations and area counts for the target analytes in the GC logbook.

7.5.7 Calculate the concentrations in $\mu\text{g}/\text{kg}$:

$$C_w = \frac{M_a \times 0.001 \text{ g}}{M_s \quad 1 \text{ kg}}$$

where

C_w = concentration of analyte in soil sample by weight, $\mu\text{g}/\text{kg}$

M_a = mass of analyte, $C_v \times V_d$, μg

M_s = mass of soil sample, g

C_v = concentration of analyte, $\mu\text{g}/1000 \text{ mL}$ (this is the concentration reported by the GC in ppb, 1 ppb = 1 $\mu\text{g}/1000 \text{ mL}$)

V_d = volume of solvent, mL

Example: GC analysis of a 5.75 g soil sample in 5 mL of hexane indicates a concentration of 500 ppb.

$$C_v = 500 \text{ ppb} = 500 \mu\text{g}/1000 \text{ mL}$$

$$V_d = 5 \text{ mL}$$

$$M_a = \frac{(500 \mu\text{g})(5 \text{ mL})}{1000 \text{ mL}} = 2.5 \mu\text{g}$$

$$C_w = \frac{2.5 \mu\text{g} \times 0.001 \text{ g}}{5.75 \text{ g} \quad 1 \text{ kg}} = 434.7 \mu\text{g}/\text{kg}$$

7.6 Analyze the water samples.

7.6.1 Dispense 5 mL of solvent into a 40-mL VOA vial using the solvent dispenser.

GC ANALYSIS OF VOCs USING SOLVENT
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- 7.6.2 Transfer 5 mL of sample to the 40-mL VOA vial using a 5- or 10-mL disposable syringe.
- 7.6.3 Shake the vial. Allow the solvent and water to separate.
- 7.6.4 Transfer 1 mL of solvent to an autosampler vial.
- 7.6.5 Analyze the sample by repeating steps 7.3.3 through 7.3.5 and 7.5.4 through 7.5.5.
- 7.6.6 On the integrator, press SEQ then START.
- 7.6.7 Calculate the concentrations in $\mu\text{g/L}$.

$$C_{ww} = \frac{M_a \times 1000 \text{ mL}}{V_s \times 1 \text{ L}}$$

where

- C_{ww} = concentration of analyte by weight/volume, $\mu\text{g/L}$
 M_a = mass of analyte, $C_v \times V_d$, g
 V_s = volume of sample, mL
 C_v = concentration of analyte, $\mu\text{g}/1000 \text{ mL}$ (this is the concentration reported by the GC in ppb, 1 ppb = 1 $\mu\text{g}/1000 \text{ mL}$)
 V_d = volume of solvent, mL

8.0 QUALITY CONTROL

- 8.1 Analyze a reagent blank.
- 8.1.1 Dispense 5 to 10 mL of solvent into a 40-mL VOA vial using the solvent dispenser.
- 8.1.2 Transfer 1 mL of solvent from the vial into an autosampler vial using a pipette.
- 8.1.3 Analyze the blank by repeating steps 7.3.3 through 7.3.5.
- 8.1.4 On the integrator, press SEQ then START.
- 8.1.5 Examine the resulting chromatogram and record any observations in the GC logbook.
- 8.2 Perform a calibration verification.

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8.2.1 Prepare and analyze initial calibration standards as outlined in 7.4.

8.2.2 Calculate the calibration factor:

$$\text{Calibration factor} = \frac{\text{total area of peak}}{\text{concentration of standard}}$$

8.2.3 Calculate the percent difference:

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100$$

where

R_1 = calibration factor from initial calibration

R_2 = calibration factor from succeeding analysis

8.2.4 Verify that the difference is $\pm 20\%$ of the initial calibration. If it is not, recalibrate the GC.

8.3 Perform a quality check once per day.

8.3.1 Prepare the quality check sample.

- a. Obtain certified standard. Do not use the same certified standard used in the preparation of the calibration standards.
- b. Following procedure TE-164, prepare one or more quality check samples of concentrations at or near the mid-point of the calibration curve (200 to 600 ppb).

8.3.2 Transfer 1 mL of the quality check sample to an autosampler vial using a pipette.

8.3.3 Analyze the sample.

8.3.4 Verify that the concentration reported by the analysis is within $\pm 20\%$ of the calculated concentration (see 8.2.2).

8.4 Analyze a replicate sample once every 20 samples.

8.4.1 Using a pipette, transfer 1 mL of the sample extract into an autosampler vial.

8.4.2 Repeat the above procedure. Label the second sample as the replicate.

GC ANALYSIS OF VOCs USING SOLVENT EXTRACTION	Number: TE-165 Page: 10 of 11
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8.4.3 Analyze both samples. Verify that the results are within $\pm 20\%$ (see 8.2.2).

8.5 Perform a matrix spike once per day.

8.5.1 Using a pipette and either a calibration standard or quality check sample, transfer 1 to 5 mL into a previously analyzed soil sample.

8.5.2 Analyze the sample.

8.5.3 Determine the calculated amount of analyte contained in the matrix spike.

8.5.4 Verify that the reported concentration of the matrix spike sample is within $\pm 20\%$ of the calculated concentration.

GC ANALYSIS OF VOCs USING SOLVENT
EXTRACTIONNumber: TE-165
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Exhibit 1. Example of Chromatogram and Report

File=C:\CP\DATA1\TUCSII.62R Sample name=SV129 Date printed= 12-11-1993 Time= 11:41:23
 0.00 to 15.00 min. Low Y = -0.08749 mv High Y = 0.41251 mv Span = 0.50000 mv

C:\CP\DATA1\Q78EA97B.BNC
 Data file = C:\CP\DATA1\TUCSII.62R
 Date stamp = 12/11/93 Time = 11:41:16
 Sample name = SV129
 Collected on DEC 11, 1993 11:26:50 from port # 1
 Operator = J.L. ZUTMAN
 Reference file name = Q78EA97Bb#66
 Instrument = HP5890
 Method name = C:\CP\DATA1\TUCIIAIR.MET version # 3
 Date method last modified = 12/08/93 Time = 16:41:38
 Calibration file = C:\CP\DATA1\TUCIIAIR.CAL version # 10
 Date cal file last modified = 12/08/93 Time = 17:34:38
 Run time = 13.67 minutes Area reject = 100
 Amount injected = 1 Dilution Factor = 1
 Sample Weight = 1 Internal Standard Amount = 0
 Sampling rate = 10 per second
 Peak detect threshold = 0 Starting peak width = .04 minutes
 Chrom-Perfect Software Serial # 12948 Version = 5.05 For Martin Marietta
 Today's date = 12-11-1993 Time = 11:41:47

PK	Ret Time	Name	Amount	Amount %	Area	Area %	Type	Width	Height	Height %
1	0.888		0.0000	0.0000%	19,654.3	26.316%	BB	0.042	7,861.79	57.9427%
2	2.280		0.0000	0.0000%	701.5	0.939%	BB	0.096	122.10	0.8999%
3	5.505	1,1,1-TCA	11.7708	0.4479%	183.0	0.245%	BB	0.150	20.31	0.1497%
4	7.737	TCE	1,204.8097	45.8427%	12,558.5	16.815%	BB	0.154	1,356.37	9.9966%
5	12.575	TETRACHLOROETHENE	1,411.5570	53.7094%	41,589.7	55.685%	BB	0.165	4,207.65	31.0111%
Total area = 74686.94			Total amount = 2628.137		Sample units = ug/l		Total height = 13568.23			

OAK RIDGE NATIONAL LABORATORY Health Sciences Research Division	PROCEDURE Environmental Technology Section	Number: TE-169 Page: 1 of 6 Revision: 0 Date: 08/04/95
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USING THE CHROM PERFECT GAS CHROMATOGRAPH SOFTWARE

1.0 PURPOSE

This procedure describes using the Chrom Perfect software in conjunction with the Hewlett Packard (HP) 5890 Series II gas chromatograph (GC) and the HP 3396 Series II integrator for analysis of environmental pollutants.

2.0 APPLICABILITY

This procedure applies to Chrom Perfect version 6.0, the HP 3396 II integrator, and the HP 5890 Series II GC. Accessory components include a personal computer (PC) capable of running the software and a HP LaserJet printer configured with ample memory to plot the chromatograms.

3.0 OTHER DOCUMENTS

3.1 REFERENCES

- 3.1.1 Chrom Perfect user's manual.
- 3.1.2 HP 3396 Series II Integrator operating manual.
- 3.1.3 Procedure TE-160, Field Operation of the Hewlett Packard 5890 Series II Gas Chromatograph.

3.2 EXHIBITS

- 3.2.1 Example of Chromatogram and Report

4.0 EQUIPMENT AND MATERIALS

4.1 EQUIPMENT

- 4.1.1 HP 5890 Series II gas chromatograph
- 4.1.2 HP 3396 Series II integrator

APPROVALS		
Author	<i>John Zorman</i> John Zorman	Date 8/31/95
Supervisor	<i>Frank Gordon</i>	Date 9/22/95
Group Leader	<i>Frank Gordon</i>	Date 9/22/95
Section Head	<i>Chris Little</i>	Date 9/25/95

USING THE CHROM PERFECT GAS CHROMATOGRAPH SOFTWARE

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- 4.1.3 HP LaserJet III or IV printer
- 4.1.4 Personal computer with monitor
- 4.1.5 Chrom Perfect version 6.0 software
- 4.1.6 GC logbook

4.2 MATERIALS

- 4.2.1 Calibration standard
- 4.2.2 Disposable syringes

5.0 RESPONSIBILITY

- 5.1 It is the responsibility of the operator of the HP 5890 II GC to follow this procedure.

6.0 PROCEDURE

6.1 Connect the integrator to the PC:

- 6.1.1 Connect the RS-232 cable from the COM1 port on the computer to the computer output port on the integrator.
- 6.1.2 Set the integrator baud rate to 2400.
 - a. Toggle all configuration switches to the on (up) position except switches 1 and 2, which are off (down).
- 6.1.3 Connect the activator device to the LPT1 parallel port on the computer.
- 6.1.4 Connect the printer cable from the activator device on the LPT1 parallel port on the computer to the parallel port on the printer.
- 6.1.5 Turn on the PC and integrator.

6.2 Load the Chrom Perfect software into the PC.

6.3 Configure the software.

- 6.3.1 At the c:\cp> prompt, type MENU.
- 6.3.2 Tab to the Configuration option on the Chrom Perfect main menu.

USING THE CHROM PERFECT GAS CHROMATOGRAPH
SOFTWARE

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6.3.3 Follow the software prompts and enter the appropriate responses, referring to the Chrom Perfect user's manual for details.

6.3.4 Return to the main menu.

6.4 Create a method file.

6.4.1 Edit an existing file, e.g., STARTING.MET.

6.4.2 Set the necessary parameters in the Acquire, Integration, and Events options located on the main method menu, referring to the Chrom Perfect user's manual for details.

6.4.3 Save the resulting method, using a unique file name for the project, e.g., TUCSON.MET.

6.4.4 Return to the main menu.

6.5 Prepare the integrator for data acquisition.

6.5.1 From the Chrom Perfect main menu, select Show Status of Integrators. This starts the Acquire program and brings up the Acquire menu.

6.5.2 Type D from the Acquire menu to download integrator for data acquisition.

6.5.3 Follow the software prompts and enter the appropriate responses, referring to the Chrom Perfect user's manual for details.

Note: The integrator will print a list of settings including run time, sampling rate, and synchronized time with the PC.

6.6 Create a calibration file when the screen shows READY FOR RUN TO BEGIN.

6.6.1 Using a syringe, withdraw a specific volume of calibration standard.

6.6.2 Inject the calibration standard into the GC, using a quick, consistent motion.

6.6.3 Withdraw the syringe and press START on the GC or on the integrator.

USING THE CHROM PERFECT GAS CHROMATOGRAPH SOFTWARE

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- 6.6.4 Record the resulting retention times and area counts for each peak of the calibration standard in the GC logbook.
- 6.6.5 Exit to the main menu.
- 6.6.6 Enter the Calibration File Editor on the main menu.
- 6.6.7 Edit an existing calibration file, e.g., STARTING.CAL.
- 6.6.8 Following the software prompts at the bottom of the screen, enter the appropriate responses, referring to the Chrom Perfect user's manual.
- 6.6.9 Save the resulting calibration file, using a file name unique to the project, e.g, TUCSON.CAL.
- 6.6.10 Exit the Calibration File Editor and return to the main menu.
- 6.6.11 Enter the Method File Editor.
- 6.6.12 Edit the newly created method file, e.g., TUCSON.MET.
- 6.6.13 Enter the Cal option from the horizontal menu at the top of the screen.
- 6.6.14 Specify the newly created calibration file to be used, e.g., TUCSON.CAL.
- 6.6.15 Exit the Method File Editor.
- 6.7 Analyze a sample. See Procedure TE-160.
 - 6.7.1 At the Chrom Perfect main menu, tab to Show Status of HP Integrators and return.
 - 6.7.2 If the screen does not show READY FOR RUN TO BEGIN, download the integrator as outlined in 6.5 above.
 - 6.7.3 Tab to Change Sample Data and return.
 - 6.7.4 Change the necessary information in the table, which will be printed on the report at the end of the run.
 - 6.7.5 Inject the sample and press START on the GC or on the integrator.

Note: During the run, the elapsed time from when the run began will be shown on the PC screen. At the

USING THE CHROM PERFECT GAS CHROMATOGRAPH SOFTWARE

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end of the run or at any time STOP is pressed on the GC or on the integrator, the data file will be transferred to the PC, and the chromatogram and report will be printed on the HP LaserJet printer.

USING THE CHROM PERFECT GAS CHROMATOGRAPH
SOFTWARE

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Exhibit 1. Example of Chromatogram and Report

File=C:\CP\DATA1\TUCSII.62R Sample name=SV129 Date printed= 12-11-1993 Time= 11:41:23
0.00 to 15.00 min. Low Y = -0.08749 mv High Y = 0.41251 mv Span = 0.50000 mv

C:\CP\DATA1\Q78EA97B.BNC
Data file = C:\CP\DATA1\TUCSII.62R
Date stamp = 12/11/93 Time = 11:41:16
Sample name = SV129
Collected on DEC 11, 1993 11:26:50 from port # 1
Operator = J.L. ZUTMAN
Reference file name = Q78EA97Bb#66
Instrument = HP5890
Method name = C:\CP\DATA1\TUCIIAIR.MET version # 3
Date method last modified = 12/08/93 Time = 16:41:38
Calibration file = C:\CP\DATA1\TUCIIAIR.CAL version # 10
Date cal file last modified = 12/08/93 Time = 17:34:38
Run time = 13.67 minutes Area reject = 100
Amount injected = 1 Dilution Factor = 1
Sample Weight = 1 Internal Standard Amount = 0
Sampling rate = 10 per second
Peak detect threshold = 0 Starting peak width = .04 minutes
Chrom-Perfect Software Serial # 12948 Version = 5.05 For Martin Marietta
Today's date = 12-11-1993 Time = 11:41:47

PK	Ret Time	Name	Amount	Amount %	Area	Area %	Type	Width	Height	Height %
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2	2.280		0.0000	0.0000%	701.5	0.939%	88	0.096	122.10	0.8999%
3	5.505	1,1,1-TCA	11.7708	0.4479%	183.0	0.245%	88	0.150	20.31	0.1497%
4	7.737	TCE	1,204.8097	45.8427%	12,558.5	16.815%	88	0.154	1,356.37	9.9966%
5	12.575	TETRACHLOROETHENE	1,411.5570	53.7094%	41,589.7	55.685%	88	0.165	4,207.65	31.0111%
Total area = 74686.94			Total amount = 2628.137		Sample units = ug/l		Total height = 13568.23			

Appendix B. ORNL-Grand Junction Task Reports

Informal Technical Report
Task 1: Pre-test Soil Characterization
for Lasagna Pilot Test Site
Paducah Gaseous Diffusion Plant

June 1994

Prepared by:
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for

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Lasagna Project
Task 1: Site Characterization

Introduction

This report details the soil characterization approach and methods used in support of the "Lasagna" pre-test site characterization performed in May 1994. The pre-test characterization consisted of drilling and sampling soil borings at the former Cylinder Drop Test Area at the Paducah Gaseous Diffusion Plant (PGDP), Paducah, KY. The goal of Task 1, the pre-test soil characterization, was to quantify the distribution and total mass of trichloroethylene (TCE) within the selected pilot test location as the basis for evaluating the treatment effectiveness of the Lasagna field tests. This goal required a rigorous approach to the sampling and characterization which extended beyond the typical purpose of delineating the nature and extent of contamination. It was necessary to collect sufficient data such that the total beginning mass of TCE could be calculated within a reasonably small standard error. This pre-pilot total mass and TCE distribution was needed to serve as the benchmark against which the performance of the technology will be measured on a mass-balance basis.

The Lasagna Project consists of three tasks that will be performed at PGDP by the Oak Ridge National Laboratory - Grand Junction Office (ORNL/GJ): Task 1, pre-test soil characterization; Task 2, sampling of treatment zones after the small-scale treatability test; and Task 3, post-pilot-test soil characterization and sampling of the treatment zones (Lasagna Work Plan, ORNL May, 1994). The following field work was performed in the course of completing Task 1: soil coring, sampling, sample handling and preservation, sample analysis, and equipment decontamination. Task 2 and Task 3 will be completed after the small and large scale pilot tests are completed. The focus of this report is on the soil characterization associated with Task 1.

Pre-test Soil Characterization

Task 1, characterization of the Lasagna project test plot, began on May 16 and was completed on May 27, 1994. The test plot is at the former Cylinder Drop Test Area drop testing and the site has been selected to demonstrate the "Lasagna" remedial technology for TCE cleanup. Based on the estimated area of TCE contamination as determined from a previous site investigation, PGDP personnel located a grid over the test plot to include the highest known levels of TCE contamination. The test plot covered a 15 ft by 15 ft area (Figure 1), within which 10 ft by 15 ft was divided into 30 grid blocks measuring 2 ft by 2.5 ft each. The remaining 5 ft by 15 ft area was to be used for a pilot test (4 ft by 4 ft) and a control area (5 ft by 10 ft). Nine (9) of the 30 grid blocks were selected at random for pre-test soil borings. Three (3) other soil borings were drilled, 2 in the small scale test plot and 1 in the control area.

Samples were collected from location L01 according to the methods specified in the Task 1 Sampling Plan. The rig was moved to the next location and nearly pure sand was encountered at a depth of 2.5 to 3 ft. This sand was considered very unusual for two reasons; 1) normally the first 3.5 ft of soil in the cylinder yard is compacted roadbase, and 2) the presence of this type of sand is often associated with buried utilities or backfilled pits. Drilling was suspended as the field crew looked for evidence of buried utilities not indicated on the excavation permit. It was discovered that a storm sewer line, consisting of a corrugated steel culvert ran nearly through the center of the test plot. Therefore, the test plot was abandoned and a new grid was laid out approximately 10 ft east and 5 ft north of the original plot (Figure 2).

Due to lack of room, the grid layout had to be modified somewhat. The 10 by 15 ft area was turned 90 degrees from the previous grid and the pilot and control areas were moved to the north end of the grid. The pilot test and control area were thus condensed from a 5 by 15 ft area to a 4 by 10 ft area. Locations L02, L03, and L04 were drilled in the new test plot; however, analysis of the samples indicated that the level of TCE

TCE Concentrations in Soil: Based on Interra Soil Characterization, Spring 1993 (Depth 15-16 ft)

50 to 100 ppm

>100 ppm

○ Pre-test Soil Boring Location

⊕ Obstruction and Refusal: Possible Concrete

----- Steel Plate 1/4" Thick
(Vertical or on Edge Approx. 8" Below Ground Surface)

Fig. 1. Original test plot grid.

Fig. 2. Location of Grid 2.

contamination in soil is present at the site as a result of the cylinder contamination in the soil was much lower than desired for the test. Indeed, the second test plot was at the edge of the contamination.

Boring and sampling was discontinued again and the grid was relocated between the storm sewer culvert and the Interra monitoring wells (Figure 3). Borings L05, L06, L07, and L08 were drilled and sampled. Upon starting boring L09, an obstruction was encountered at a depth of about 5 ft below ground surface. Penetrating the obstruction proved impossible with both augers and Geoprobe samplers. The obstruction appeared to be concrete or highly compacted gravel and may be part of the pit where the cylinders were dipped before drop testing.

The test plot was moved about 10 ft north (Figure 4). This move allowed locations L05, L06, L07, and L08 to remain within the new test plot so that data from these locations could be used for the site characterization. Location L09 through L16 were sampled to complete the characterization. Figure 4 shows the layout of the final test plot and the sampling locations within the grid. The figure also shows the location of the boring drilled to provide samples to Monsanto and GE for laboratory testing.

Sampling Methods

At each sample location, a 4 to 6 in. diameter hole was bored through the 3.5 ft of roadbase. This pilot hole was either augured with the rig or hand dug with posthole diggers. A 3.5 ft long piece of 3 in. diameter PVC pipe was then placed in the pilot hole to prevent cave-in. Continuous soil samples were collected using Geoprobe soil sampling tubes with acetate liners. A 4 ft sample tube with liner was driven from 3.5 ft to 7.5 ft to collect the initial 4 samples. Two (2) ft long samplers were then used to collect samples from 7.5 ft to 15.5 ft deep.

Fig. 3. Location of Grid 3.

TCE Concentrations in Soil: Based on Interra Soil Characterization, Spring 1993 (Depth 15-16 ft)

50 to 100 ppm

>100 ppm

○ Pre-test Soil Boring Location

▲ GE/Monsanto Soil Boring Location

Fig. 4. Location of Grid 4.

Once a sampler was removed from the borehole, the acetate liner was withdrawn from the sample tube, and the depths were measured and marked on the outside of the liner. The liner was then cut into separate 1 ft sections and PID measurements were immediately taken at each exposed soil surface. The 1 ft liner sections were then cut lengthwise and soil samples were collected with a microcorer. Each sample consisted of approximately 5 grams of soil placed in a 40 mL vial containing 10 mL of pesticide grade hexane. The sample vials were immediately placed in a cooler containing Blue Ice for transportation to the field lab. After collection of the samples, the remaining soil was described and logged by the field geologist.

All equipment that contacted a soil sample was cleaned between each use. Equipment that did not contact soil samples was cleaned between each borehole location. Cleaning consisted of thoroughly washing the equipment using a high-pressure hot water washer adjusted to provide 180 degree F water. Sampling equipment was also rinsed with deionized water.

Borings were abandoned by pouring Volclay grout into the hole immediately after removing the Geoprobe rods. This method was used after attempts to pump grout into the holes failed. Grout could not be thinned adequately to pump down the small polyethylene tube.

QA/QC

One of every 10 samples was collected in duplicate for the field laboratory and for PGDP laboratory. The PGDP sample was collected in the same manner as the field GC sample, i.e., 5 grams of soil in 10 mL of hexane. One of every 20 samples was submitted to the PGDP laboratory for analysis of volatile organic compounds using the RCRA method 8240. The RCRA sampling method consists of packing a 40 mL vial full of soil and cooling the sample to 4 degrees C.

A field blank was collected from the deionized water used for equipment rinses. One equipment rinse was collected at each location, but not less than one per day. A trip blank was provided by PGDP and accompanied all samples to the laboratory. The PGDP laboratory analyzed all field blanks, trip blanks, and equipment rinse samples.

Field Laboratory Procedures

Before collection of soil samples, each 40 mL vial was prepared as follows: a sample label was attached, 10 mL of hexane was placed in each vial, the vial was weighed, and this "tare" weight was written on the vial's label. These vials were then taken to the field for sample collection.

Upon return to the field laboratory, each vial was weighed, this provided an accurate weight of the soil in the vial. After weighing, the vials were placed in an ultrasonic water bath for 15 minutes to help ensure that all volatiles from the soil were released into the hexane.

Depending on the PID measurement of the soil sample in the field, either a 1:10 or 1:100 dilution of the sample extractant was prepared. Dilutions were required because the field GC was calibrated to be linear within a range of 5.0 ppb to 1000 ppb; therefore, the concentration of the injected liquid needed to be within that range. If the field PID measurement was less than 10 ppm, a 1:10 dilution was used; if the PID reading was greater than 10 ppm, a 1:100 dilution was prepared. The field GC logbook shows the dilution concentration injected into the GC. Dilutions were prepared by transferring 1 mL of hexane from the sample vial into 9 mL of hexane, creating a 1:10 dilution. Further dilution was completed by transferring 1 mL of the 1:10 dilution into 9 mL of hexane, creating a 1:100 dilution. All bottles were labelled with the sample number and the dilution. Transfers were completed with a mechanical pipette. All transfers and dilutions used new sample bottles and pipette tips. Sample and dilution vials were stored until the GC analyses were completed in case additional runs or dilutions were necessary.

One (1) mL of hexane from the required dilution was then placed in a 2 mL septa top vial and loaded into the autosampler for GC analysis. The first sample loaded in the autosampler and every tenth sample from then on were blanks, consisting of 1 mL of hexane in a sample vial. These blanks were analyzed to determine if the syringe, septa, or column had become contaminated during the samples analyses. Analysts prepared samples during the day and the GC automatically analyzed the samples overnight.

Following the GC analysis, sample reports were studied by the analyst to determine if any samples were outside the calibration range or if any of the blanks contained contamination. No blanks were found to be contaminated; however, several samples were out of the calibration range on the first run. These samples were further diluted to 1:1000 by transferring 1 mL of the 1:100 dilution into 9 mL of hexane. This 1:1000 dilution was then analyzed by the GC. No samples required dilution beyond 1:1000.

All samples were analyzed on a Hewlett Packard 5890 Series II Gas Chromatograph equipped with a split/splitless injector and an electron capture detector. The GC was calibrated for TCE and 1,1-DCE. A calibration curve was generated from standards at concentrations of 10, 50, 100, 250, 500, and 1000 ppb. Standards were prepared from custom mix standards diluted to create the range of concentrations. Following initial calibration, standards were run every two days to check retention times and concentration determination. No variation was observed during the project.

One (1) μL of extractant liquid was injected directly on the column using a HP7673 autosampler. The autosampler was controlled with a HP 3396 integrator. Chromatograms were collected, stored and reported using ChromPerfect, Version 4.3. The ChromPerfect software calculated the concentration of contaminants based on the dilution level for each sample. The lab analyst calculated the concentration of TCE per kilogram of soil and entered this into the logbook. Paper and electronic copies of individual chromatograms were stored in logbooks and on floppy disks.

Table 1 shows the results of field GC analysis for TCE. No 1,1-DCE was detected in any sample.

Table 1
Lasagna Project
Field GC Results for Task 1 Soil Samples
May 16 - 27, 1994

Table 1
Lasagna Project
Field GC Results for Task 1 Soil Samples
May 16 - 27, 1994

BOREHOLE L05

<u>TCE CONCENTRATION (mg/kg)</u>		
<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	20.3	28.7
05	49.8	47.4
06	48.5	53.3
07	32.8	33.6
07 dup	25.1	40.7
08	23.7	29.2
09	22.8	25.8
10	45.2	48.7
11	69.3	85.5
12	110.4	125.0
13	169.4	149.4
13 dup	156.4	162.5
14	121.0	112.6
15	163.2	142.2
15.5	152.5	182.0

BOREHOLE L06

<u>TCE CONCENTRATION (mg/kg)</u>		
<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	4.9	17.0
05	25.2	25.7
06	40.8	29.1
07	50.7	46.0
08	19.9	33.4
09	40.3	40.1
10	58.4	54.0
10 dup	56.3	71.7
11	138.1	145.5
12	130.9	157.4
13	167.3	139.6
14	151.2	117.9
15	156.5	164.0
15.5	100.8	149.5

BOREHOLE L07

<u>TCE CONCENTRATION (mg/kg)</u>		
<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	37.2	36.9
05	62.2	44.9
06	71.9	79.4
07	68.5	79.5
07 dup	74.4	83.6
08	67.0	74.7
09	75.9	79.6
10	74.3	67.6
11	111.1	118.6
12	176.7	158.4
13	160.6	164.1
14	138.7	135.0
15	175.4	133.5
15.5	80.7	127.5

BOREHOLE L08

<u>TCE CONCENTRATION (mg/kg)</u>		
<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	9.7	6.0
04 dup	24.2	4.6
05	12.1	9.6
06	3.0	2.0
07	3.8	3.0
08	2.3	1.6
09	6.8	7.6
10	95.7	66.2
11	136.9	118.3
12	169.5	292.0
13	278.7	402.5
14	368.6	507.3
14 dup	352.5	161.5
15	237.3	185.7
15.5	221.9	199.1

BOREHOLE L09TCE CONCENTRATION (mg/kg)

<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	13.7	10.8
05	40.4	37.3
06	64.1	71.9
07	85.4	62.9
08	79.4	62.7
09	47.3	46.6
10	42.5	38.7
11	51.3	50.5
11 dup	50.2	45.9
12	113.1	108.0
13	104.4	105.4
14	77.6	108.0
15	80.0	75.5
15.5	78.1	64.0

BOREHOLE L10TCE CONCENTRATION (mg/kg)

<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	7.6	8.6
05	1.0	0.7
06	1.0	0.5U
07	1.2	2.6
08	6.0	4.8
08 dup	5.3	5.3
09	5.7	5.9
10	7.8	6.7
11	5.1	6.9
12	4.7	4.3
13	2.6	2.5
14	1.9	2.1
15	3.2	2.9
15.5	3.5	3.9

BOREHOLE L11TCE CONCENTRATION (mg/kg)

<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	4.1	8.8
05	17.6	16.0
06	19.0	19.9
06 dup	21.8	20.0
07	22.2	26.8
08	33.2	32.9
09	63.5	77.0
10	89.3	90.6
11	130.5	125.0
12	144.3	133.5
13	144.2	145.8
14	94.1	107.8
15	104.5	97.5
15.5	71.8	86.9

BOREHOLE L12TCE CONCENTRATION (mg/kg)

<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	13.7	10.4
04 dup	11.0	8.7
05	12.0	14.6
06	20.3	21.4
07	24.8	24.8
08	39.9	33.8
09	67.5	51.3
10	76.9	75.5
11	75.5	82.5
12	60.5	63.2
13	53.9	58.7
14	52.2	46.0
15	40.8	24.1
15.5	32.4	31.5

BOREHOLE L13TCE CONCENTRATION (mg/kg)

<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	38.3	30.9
05	57.3	63.1
05 dup	62.4	60.7
06	64.8	65.2
07	91.7	114.5
08	71.7	53.7
09	93.3	88.9
10	94.8	109.4
11	137.5	129.5
12	140.2	135.3
13	136.6	129.8
14	135.9	143.9
15	127.5	128.7
15.5	147.6	120.5

BOREHOLE L14TCE CONCENTRATION (mg/kg)

<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	10.4	10.6
05	42.5	36.9
06	30.8	27.4
06 dup	25.1	28.2
07	35.7	37.7
08	35.8	43.1
09	118.9	129.0
10	78.4	138.9
11	162.6	165.1
12	181.0	172.6
13	178.2	152.2
14	164.7	167.0
15	146.7	146.7
15.5	123.0	91.9

BOREHOLE L15

<u>TCE CONCENTRATION (mg/kg)</u>		
<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	61.6	54.7
05	62.8	59.6
06	47.9	47.0
07	43.7	39.2
07 dup	43.4	40.6
08	70.5	66.4
09	103.6	100.8
10	115.7	117.1
11	135.2	147.6
12	169.8	182.4
13	155.1	144.4
14	196.8	211.9
15	163.2	144.6
15.5	158.8	159.1

BOREHOLE L16

<u>TCE CONCENTRATION (mg/kg)</u>		
<u>DEPTH</u>	<u>SAMPLE 1</u>	<u>SAMPLE 2</u>
04	10.6	11.5
05	17.3	21.9
06	21.8	22.6
07	25.8	27.2
08	38.7	32.9
08 dup	49.4	43.6
09	45.9	46.8
10	69.2	80.7
11	108.7	99.1
12	109.7	107.7
13	114.2	89.2
14	97.6	105.8
15	61.1	70.9
15.5	45.7	50.3

Draft Informal Technical Report
Task 2 and 3: Carbon Sampling and Post-test Soil Characterization
for Lasagna Pilot Test Site
Paducah Gaseous Diffusion Plant

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Lasagna Project

Task 2 and 3: Carbon Sampling and Post-test Site Characterization

Introduction

This report documents the methods used in support of the "Lasagna" carbon sampling and post-test site characterization performed during March, May, and June 1995 at the Cylinder Drop Test Area at the Paducah Gaseous Diffusion Plant (PGDP), Paducah, KY (Fig. 1)

Task 2, the carbon sampling, consisted of two phases of field work, one in March and the second in May. The goals of phase 1 were to determine the effectiveness of the carbon in adsorbing TCE and to establish a termination date for the Lasagna treatment. The goal of phase 2 was to determine the total mass of TCE adsorbed onto the carbon.

Task 3, the post-test characterization, consisted of drilling, sampling, and analyzing the soil at the Lasagna test site. The goal of Task 3 was to quantify the distribution and total mass of TCE remaining in the soil after the Lasagna treatment. It was necessary to collect sufficient data such that the total mass of TCE could be calculated within a small standard error. The difference between the pre- and post-treatment mass of TCE and the mass of TCE adsorbed by the carbon provided a basis for evaluating the treatment effectiveness of the Lasagna field tests. The pre-test total mass and TCE distribution were determined during Task 1 (ORNL Technical Memo June 1994).

The Oak Ridge National Laboratory - Grand Junction Office (ORNL/GJ) performed three tasks in support of the Lasagna Project: Task 1, pre-test soil characterization; Task 2, sampling of carbon cartridges; and Task 3, post-pilot-test soil

Figure 1. Location of Lasagna Test Area

characterization (Lasagna Work Plan, ORNL May 1994). The following field work was performed in the course of completing these tasks: soil coring, sampling, sample handling and preservation, sample analysis, and equipment decontamination. The focus of this report is on Task 2 and 3.

Task 2: Carbon Sampling

The first phase of carbon sampling was performed on March 27, 1995. Carbon sampling cartridge C4-7 was removed from the treatment zone and sampled. The cartridge was cut into 1 ft long segments and the carbon from each segment was placed into a 2 liter wide-mouthed sample jar. The carbon in each jar was subsampled and placed in 40 mL vials for extraction and analysis according to the procedures presented in Standard Operating Procedure ESC-SOP-95-UT614, March 15, 1995. All samples were forwarded to General Electric (GE) for analysis. A new carbon cartridge was installed in C4-7.

The second phase of carbon sampling was performed on May 16 and 17, 1995. During this phase, all of the carbon sampling cartridges were removed, segmented, and sampled following the same procedures used in phase one. Figure 2 shows the carbon sampling cartridges that were sampled. All samples were forwarded to GE for analysis.

Task 3: Post-test Soil Characterization

Task 3, post-test characterization of the Lasagna project test plot, began on June 5 and was completed on June 16, 1995. Twelve soil borings were drilled in close proximity to the 12 pre-test soil borings L-05 through L-16 (Fig. 3). Samples were collected from

PADLASG6

locations LP-05 through LP-16 (Fig. 3) according to the methods specified in the Task 1 Sampling Plan.

Sampling Methods

At each sample location, a 4 to 6 in. diameter hole was bored through the surface gravels. This pilot hole was either augured with the rig or hand dug with a posthole digger. A 3.5 ft long piece of 3 in. diameter PVC pipe was then placed in the pilot hole to prevent cave-ins. Continuous soil samples were collected using 4 ft Geoprobe soil sampling tubes with acetate liners. Samples were collected every foot beginning at 3.5 ft and ending at 17.5 ft.

Once a sampler was removed from the borehole, the acetate liner was withdrawn from the sample tube, and the depths were measured and marked on the outside of the liner. The liner was then cut into separate 1 ft sections and PID measurements were immediately taken at each exposed soil surface. The 1 ft long liner sections were then cut lengthwise and soil samples were collected using a disposable syringe sampler. Each sample consisted of approximately 5 grams of soil placed into a pre-weighed 40 mL vial containing 5 mL of GC Capillary Grade hexane and 5 mL of deionized (DI) water. The sample vials were immediately placed in a cooler containing Blue Ice for transportation to the field lab.

All equipment that contacted a soil sample was cleaned between each use. Equipment that did not contact soil samples was cleaned between each borehole location. Cleaning consisted of thoroughly washing the equipment using a high-pressure hot water washer adjusted to provide 180 degree F water. Sampling equipment was also rinsed

with deionized water. Borings were abandoned by pouring Volclay grout into the hole immediately after removing the Geoprobe rods.

QA/QC

The GC was calibrated daily for TCE and 1,1-DCE. A linear calibration curve was generated using standards at concentrations of 10, 50, 100, 250, 500, and 750 ppb. Standards were prepared from custom mix standards diluted to create the range of concentrations. Linearity was established using a minimum coefficient of linear correlation of 0.997. Following initial calibrations, standards were run a minimum of once per day to verify retention times and concentration determination. No variation was observed during the project.

One of every 10 samples was collected in duplicate for analysis at the field laboratory. The duplicate sample was collected in the same manner as the field GC sample, i.e., 5 grams of soil in 5 mL of hexane and 5 mL DI water. The weight of each sample was determined within ± 0.01 gram. A field blank was collected from the deionized water used for equipment rinses. An equipment rinse was collected at each location. A trip blank accompanied all samples to the field laboratory. All duplicate soil samples, field blanks, trip blanks, and equipment rinses were analyzed at the field laboratory. None of the blank samples were above detection for analytes of interest. The detection limit for the field gas chromatograph (GC) was 2 ppb for trichloroethylene.

Splits of all soil sample extracts, diluted to the linear range of the GC, were sent to Monsanto for confirmation analysis.

Figure 3. Location of soil borings.

- L10 ● Pre-Treatment Soil Boring Location
 LP10 ◆ Post-Treatment Soil Boring Location
 ▲ Monsanto Soil Boring Location

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Field Laboratory Procedures

Before collection of soil samples, each 40-ml vial was prepared as follows: a waterproof sample label was attached, 5 mL of GC grade hexane and 5 ml of DI water were placed in each vial, the vial was weighed, and this "tare" weight was written on the label. The vials were placed into a cooler containing Blue Ice and taken to the field for sample collection. Samples were collected as described above. Upon return to the field laboratory, each vial was weighed, this provided an accurate weight of the soil in the vial.

Dilutions of the sample extract were required to remain within the linear range of the GC. Dilutions of 1:10, 1:100, or 1:1000 were used depending upon the sample concentration. The field GC logbook shows the dilution concentration injected into the GC. 1:10 dilutions were prepared by transferring 500 uL of sample extract into a vial containing 4.5 mL of hexane. Further dilution was completed by transferring 500 uL of the 1:10 dilution into 4.5 mL of hexane, creating a 1:100 dilution. All bottles were labeled with the sample number and the dilution. Transfers were completed with a mechanical pipette. All transfers and dilutions used new sample bottles and pipette tips. Sample and dilution vials were stored until the GC analyses were completed in case additional runs or dilutions were necessary.

One (1) mL of diluted sample extract was then placed into a 2 mL septa top vial and loaded into the autosampler for GC analysis. Hexane blanks were analyzed before the samples to verify that the syringe, septa, and column had not become contaminated during the previous sample analyses. Analysts prepared samples during the day and the GC automatically analyzed the samples overnight.

All samples were analyzed on a Hewlett Packard 5890 Series II Gas Chromatograph equipped with a split/splitless injector and an electron capture detector.

One (1) uL of extractant liquid was injected directly on the column using an HP7673 autosampler. The autosampler was controlled via a sequence file with an HP 3396 integrator. Chromatograms were collected, reported, and stored using ChromPerfect, Version 6.3. The ChromPerfect software calculated the concentration of contaminants based on the dilution level for each sample. The lab analyst calculated the concentration of TCE per kilogram of soil and entered this into the logbook. Paper and electronic copies of individual chromatograms were stored in logbooks and on floppy disks.

Following the GC analysis, sample reports were studied by the analyst to determine if any samples were outside the calibration range or if any of the blanks contained contamination. No blanks were found to be contaminated; however, several samples were either above or below the calibration range on the first run. These samples were re-analyzed using the correct dilution.

Table 1 shows the results of field GC analysis for TCE. No 1,1-DCE was detected in any sample.

Table 1

Lasagna Project

Field GC Results for Task 3 Soil Samples

TCE Concentrations (mg/kg)

June 5 - June 16, 1995

BOREHOLE LP-05

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
318	4.5	4.44	1	0.177
320	5.5	4.30	1	0.263
321	6.5	5.18	1	0.153
322	7.5	5.17	1	0.044
324	8.5	3.85	1	0.032
325	8.5 DUP	4.69	1	0.055
328	9.5	2.08	1	0.004
329	10.5	4.15	1	0.033
330	11.5	3.31	1	0.136
332	12.5	4.69	1	0.168
333	13.5	5.29	1	0.122
334	14.5	4.67	10	0.197
336	15.5	5.18	10	0.292
337	16.5	5.22	100	53.45
338	17.5	5.46	100	73.84

BOREHOLE LP-06

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
298	4.5	4.98	10	1.219
299	5.5	4.10	10	1.423
300	6.5	5.29	1	0.456
302	7.5	3.57	1	0.134
303	8.5	4.69	1	0.187
304	9.5	4.60	1	0.211
306	10.5	4.21	1	0.205
307	11.5	5.75	1	0.317
308	12.5	4.70	1	0.446
309	12.5 DUP	4.73	1	0.434
312	13.5	4.06	1	0.525
313	14.5	4.29	1	0.345
314	15.5	4.66	10	0.514
316	16.5	5.36	100	56.79
317	17.5	4.56	10	9.043

BOREHOLE LP-07

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
276	4.5	5.33	10	0.144
277	5.5	4.57	10	0.088
279	6.5	5.26	1	0.104
280	7.5	3.39	1	0.081
281	8.5	5.91	1	0.101
283	9.5	4.66	1	0.073
284	10.5	5.00	1	0.101
285	11.5	5.57	1	0.188
287	12.5	5.30	1	0.442
288	13.5	4.67	10	0.055
289	14.5	4.52	10	2.710
290	14.5 DUP	4.94	10	2.034
293	15.5	5.31	10	8.616
294	16.5	4.97	100	41.25
295	17.5	5.13	100	19.60

BOREHOLE LP-08

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
353	4.5	4.34	10	2.031
354	5.5	3.97	10	1.135
355	6.5	4.43	1	0.323
NO SAMPLE	7.5			
358	8.5	4.74	1	0.176
359	9.5	4.86	1	0.149
360	10.5	5.15	1	0.284
363	11.5	4.71	1	0.376
364	12.5	4.61	10	0.438
365	13.5	4.72	10	0.418
366	14.5	4.63	10	0.018
367	15.5	5.53	100	17.442
368	15.5 DUP	5.53	100	11.589
369	16.5	4.59	100	35.827
372	17.5	5.33	100	48.123

BOREHOLE LP-09

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
340	4.5	6.45	1	0.002
341	5.5	5.33	1	0.403
NO SAMPLE	6.5			
NO SAMPLE	7.5			
342	8.5	5.64	1	0.113
343	9.5	4.58	1	0.233
344	10.5	5.17	1	0.263
345	10.5 DUP	5.44	1	0.251
NO SAMPLE	11.5			
346	12.5	4.24	1	0.529
347	13.5	6.35	10	3.144
348	14.5	4.68	100	13.685
349	15.5	6.03	100	15.230
350	16.5	5.10	100	29.215
351	17.5	4.48	100	32.831
352	17.5 DUP	6.69	100	37.767

BOREHOLE LP-10

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
200	5	4.99	1	0.042
201	6	5.34	1	0.203
202	7	5.33	1	0.179
NO SAMPLE	8			
NO SAMPLE	9			
NO SAMPLE	10			
NO SAMPLE	11			
204	12	5.54	10	0.700
205	13	8.73	10	0.469
206	14	5.85	10	0.257
207	15	6.54	10	0.059
208	16	5.98	10	0.501
209	17	5.78	10	2.049
210	17 DUP	5.63	10	1.930

BOREHOLE LP-11

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
220	4.5	5.94	1	0.551
221	5.5	3.81	1	0.467
222	6.5	5.03	1	0.577
223	7.5	4.67	1	0.537
224	8.5	4.74	1	0.401
225	8.5 DUP	3.51	1	0.437
NO SAMPLE	9.5			
NO SAMPLE	10.5			
NO SAMPLE	11.5			
229	12.5	4.06	10	2.220
230	13.5	5.91	10	1.838
231	14.5	5.81	10	0.398
232	15.5	4.75	10	1.042
233	16.5	4.23	100	7.392
235	17.5	5.12	100	22.652

BOREHOLE LP-12

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
236	4.5	5.08	1	0.217
237	5.5	5.52	1	0.109
238	6.5	4.91	1	0.002J
239	7.5	6.01	1	0.002J
241	8.5	5.05	1	0.003J
242	8.5 DUP	4.82	1	0.003J
245	9.5	5.11	1	0.025
246	10.5	5.54	1	0.292
247	11.5	4.88	10	0.494
248	12.5	6.15	10	1.096
249	13.5	5.59	10	1.643
251	14.5	5.01	10	1.565
252	15.5	6.90	100	5.225
253	16.5	6.24	100	22.368
254	17.5	5.77	100	22.574

BOREHOLE LP-13

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
256	3.5	4.43	10	2.289
257	3.5 DUP	4.73	10	2.562
260	4.5	4.09	10	2.924
261	5.5	4.91	10	0.666
262	6.5	4.20	1	0.038
264	7.5	4.86	1	0.212
265	8.5	5.40	10	0.247
266	9.5	5.13	10	0.247
268	10.5	5.39	10	0.274
269	11.5	5.45	10	0.439
270	12.5	4.22	10	0.059
272	12.5	5.48	10	0.107
274	13.5	5.03	1	0.226
REFUSAL AT 14'				

BOREHOLE LP-14

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
387	4.5	4.65	100	24.574
388	5.5	5.05	100	33.779
NO SAMPLE	6.5			
NO SAMPLE	7.5			
389	8.5	3.91	100	68.536
390	9.5	4.82	100	89.222
391	10.5	4.16	100	80.446
392	10.5 DUP	5.13	100	88.105
393	12.5	5.35	100	96.738
394	13.5	4.63	100	78.017
395	14.5	4.68	100	91.227
396	15.5	4.45	100	101.692
NO SAMPLE	16.5			
397	17.5	5.84	100	56.384

BOREHOLE LP-15

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
374	4.5	5.13	100	10.632
375	5.5	4.42	10	6.731
376	6.5	5.63	10	7.149
NO SAMPLE	7.5			
377	8.5	4.41	100	43.041
378	9.5	4.82	100	62.653
379	10.5	5.92	100	74.260
380	11.5	5.19	100	86.675
381	12.5	6.04	100	69.204
382	12.5 DUP	5.66	100	70.889
383	13.5	4.44	100	68.454
384	14.5	6.41	1000	73.401
385	15.5	4.52	1000	109.464
386	16.5	5.12	100	36.973

BOREHOLE LP-16

<u>SAMPLE</u>	<u>DEPTH</u>	<u>WT (g)</u>	<u>DIL</u>	<u>mg/Kg</u>
398	4.5	4.51	100	2.564
399	5.5	5.03	100	13.728
400	6.5	4.93	100	4.346
401	7.5	5.34	100	6.529
402	8.5	5.60	100	11.428
403	9.5	5.14	100	16.205
404	10.5	2.98	100	50.807
406	11.5	6.42	100	33.592
407	12.5	5.23	100	30.264
408	13.5	5.02	100	30.678
409	14.5	5.40	100	19.966
410	15.5	5.07	100	14.241
411	16.5	4.23	100	9.303
412	17.5	3.51	100	25.843

PADUCAH GASEOUS DIFFUSION PLANT

- C-700 Building Number
- x - x - Fence
- ==== Road

Carbon Wickdrain
Carbon Wickdrain with Sampling Cartridge

 0 0.5 1 2
 Feet

PADLASG6

- L0 Pre-Treatment Soil Boring Location
- ◆ LP10 Post-Treatment Soil Boring Location
- ▲ Monsanto Soil Boring Location

0 0.5 1 2
Feet

PADLASG6

Appendix C. Carbon Compositing and Sampling SOP

Monsanto Company
Environmental Sciences Center
St. Louis, MO 63167

Document No. ESC-SOP-95-UT614
Issue/Revision Date: March 15, 1995
No. of Pages: 7

STANDARD OPERATING PROCEDURE

Compositing and Sampling Carbon in Sampling Cassettes from the Lasagna™ Field
Experiment at Paducah, Kentucky for DOE Contract No. DE-AR21-94MC31185

Prepared by:	Signed	<u>B. M. Hughes</u>
	Title	<u>Senior Analytical Consultant</u>
	Date	<u>March 25, 1995</u>
Approved by:	Signed	<u>Sa V. Ho</u>
	Title	<u>Fellow & Remediation Unit Leader</u>
	Date	<u>5/8/95</u>
Accepted for use by QA Supervisor:	Signed	<u>Janet M. Lee</u>
	Title	<u>Supervisor, Quality Assurance</u>
	Date	<u>March 27, 1995</u>

Revision History:

SOP Number

Issue Date

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3/25/95 1:57 PM

Document Control

Copy One - Assigned to B.M. Hughes, 6-5-95

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STANDARD OPERATING PROCEDURE ESC-SOP-95-UT614

Compositing and Sampling Carbon in Sampling Cassettes from the Lasagna™ Field
Experiment at Paducah, Kentucky for DOE Contract No. DE-AR21-94MC31185

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1 SCOPE AND APPLICATION

- 1.1 This procedure is for removing and sampling carbon-containing sampling cassettes which are present in selected carbon adsorption zones in the *Lasagna*TM field experiment being conducted in Paducah, Kentucky. This procedure was developed for DOE Contract No. DE-AR21-94MC31185 and will be used to determine the concentration of trichloroethylene (TCE) which has been adsorbed in the carbon adsorption zones. This procedure must be approved by Martin Marietta Energy Systems (MMES) and be consistent with the Requirements to Performing Work at the Paducah Gaseous Diffusion Plant¹. Appendix shows the MMES Site Specific Health and Safety Plan for this procedure.

2 SUMMARY OF PROCEDURE

- 2.1 A carbon sampling cassette (consisting of a 1-inch x 4-inch x 16-foot fabric-covered wickdrain which contains granulated carbon) will be removed from one of 12 treatment/adsorption zones which are located between the electrodes of the *Lasagna*TM field experiment. The cassette will be placed on a clean flat surface which has been covered with clean aluminum foil.
- 2.2 The cassette will be cut into 1-ft. sections and the carbon contained in each section will be composited in 16 2-liter wide mouth glass jars with Teflon-lined lids. Known weights of representative wet carbon samples will be removed from each composited sample and placed in various sampling vials for % moisture and chemical analysis. This procedure will be used to evaluate the average concentration of TCE in each 1-ft. section which will, in turn, be used to estimate the total amount of TCE which has been adsorbed in all of the carbon adsorption zones in the field experiment. A similar procedure will also be used for the sampling cassette which is located in the control region of the experiment.

3 INTERFERENCES

- 3.1 Some chemical analyses will be performed using GC/EC instrumentation which is very sensitive to chlorinated materials. Therefore great care must be exercised to prevent contamination of the glassware and carbon with chlorinated solvents. There should be no chlorinated solvents present in the field during this sampling and compositing procedure.
- 3.2 Total volatile organic compounds will be analyzed in headspace analyses using GC/MS and GC/FID instrumentation. Since the hexane solvent which is used for preservation

¹ Part III, Section J, Attachment F of DOE Contract No. DE-AR21-94MC31185 describes the Requirements to Perform Work at the Paducah Gaseous Diffusion Plant.

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will be detected in the headspace analyses, great care should be exercised to prevent contamination of the headspace analysis vials with this solvent. If possible, the headspace analysis vials should be shipped separately from the samples to which hexane has been added.

4 APPARATUS AND MATERIALS

4.1 The following materials are needed for this procedure:

- Clean rubber gloves,
- clean exacto or utility knife (or other cutting equipment),
- aluminum foil,
- disposable clean metal stirring rods,
- 20 2-liter wide mouth jars with Teflon-line lids,
- 100 16-mL vials with Teflon-lined lids,
- 20 headspace analysis vials with caps,
- crimper for the headspace vials, and
- labels.

4.2 Automatic Pipettes: 1,000 μ L Pipetteman, or equivalent.

4.3 Drying oven capable of maintaining 120 °C. overnight.

4.4 Analytical balance capable of measuring 50 grams with 0.01 gram accuracy.

5 REAGENTS

5.1 The following high purity solvents will be required:

Hexane: Fisher Ultra brand, or equivalent.

Water: Burdick and Jackson (B&J) distilled in glass brand, or equivalent.

6 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All carbon samples will be weighed in either 16-mL glass vials with Teflon-lined septa or headspace analysis vials. Carbon which will be placed in 16-mL vials for TCE analysis will be preserved with 1.0 mLs hexane. Samples collected in 16-mL vials for moisture determination will not be preserved. Moisture will be determined on-site by weighing before and after heating overnight in a drying oven. Samples which will be collected for total volatile compounds will be weighed in headspace analysis vials. No preservatives will be added for these samples.

6.2 The preserved carbon samples and the samples in headspace analysis vials will be sent to the consortium for TCE and volatile compound analysis. A representative sample of carbon will be used by MMES to screen for radioactivity before shipping the carbon samples.

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- 6.3 MMES will be responsible for moisture determination in aliquots of carbon taken from each of the 2-liter jars. They will also provide gravimetric information for the remaining carbon samples.

7 PROCEDURE

- 7.1 The 16-ft. sampling cassette will be removed from the carbon adsorption zone and placed on a clean surface which will be covered with aluminum foil. The cassette will be cut into 16 1-ft. sections using an exacto or utility knife (or other cutting equipment) and the contents of each section will be placed in 16 2-liter wide mouth jars with Teflon-line caps. Care should be taken to place all contaminated wastes in special containers provided by CDM Federal Programs for waste disposal.
- 7.1.1 Labeling: Each of the 2-liter jars will be labeled with the adsorption zone identification, followed by the depth of the section. The first section will be called 0 feet and the deepest section will be called 15 feet. This label will also be placed on each 16-mL vial and headspace analysis vial, with the letters A, B, C, etc. following the depth information.
- 7.2 Disposable clean metal stirring rods will be used to homogenize the carbon in each of the wide-mouth jars. Approximately 2 - 3 grams of wet carbon will be weighed in 5 16-mL vials with Teflon-lined lids. Each vial will be labeled with suffixes A, B, C, D, and E. A 6th and 7th sample will be weighed in headspace analyzer vials and labeled with F and G after the zone identification and depth.
- 7.3 Moisture will be determined in two of the 16-mL vials. This measurement will be made by measuring the percent of weight loss after heating the open vial overnight at 120 degrees C.
- 7.4 Fisher Ultra hexane will be added as a preservative for the three remaining 16-mL vials. A micropipette with disposable tips will be used to add 1.0 mLs to each of the vials. Addition of the hexane should occur after the vials for headspace analysis have been crimped in order to prevent contamination of these samples with the hexane solvent used for preservation.
- 7.5 After MMES has screened a representative carbon sample from one of the 2-liter jars, the 16-mL vials containing carbon with the hexane preservative, and the headspace vials containing the carbon, will be shipped to Dr. Roy Thornton of General Electric.
- 7.6 If available, a clean, fresh sampling cassette will be placed back into the slot from which the experimental cassette was removed. A slit will be made in the top of the cassette so that fresh carbon can be taken and used for quality control in section 8.0.

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8 QUALITY CONTROL

- 8.1 Two 16-mL vials of B&J distilled in glass water (with no head space) and two 16-mL vials containing approximately 8 mLs of Fisher Ultra hexane will be shipped with the 16-mL vials containing the carbon samples. If possible, the 16-mL vial samples should not be shipped in the same container which contains the headspace samples in order to minimize contamination of these samples.
- 8.2 Clean carbon should be taken from the fresh sampling cassette and placed in a 2-liter wide mouth jar. This jar of carbon should be handled in an identical manner as all samples. (After mixing the blank carbon, known amounts of carbon should be added to 5 16-mL vials and a known amount of carbon should be added to two headspace vials. The percent moisture should be measured in two of the samples, and hexane should be added to three of the vials.) The results from analysis of these samples will be used to determine whether contamination may have occurred during sampling or transportation.

9 METHOD PERFORMANCE

- 9.1 Evaluation of the homogeneity of the carbon samples will be provided by comparing the TCE concentrations in replicate samples from each 2-liter carbon sample. The analysis of solvents and clean carbon samples which were taken at the same time the cassette carbon samples were taken, will provide information concerning contamination during sampling and transportation.

10 SUMMARY OF SAMPLES TO BE TAKEN

- 10.1 An initial cassette will be removed in March 1995 after approximately 1.5 pore volumes of field operations. The results from analysis of the TCE concentrations in this initial cassette will be used to evaluate the effectiveness of electro-osmosis in moving the TCE through the clay and whether the experiment should be extended past the time originally estimated to move contaminants 2 pore volumes. In addition, the need for replicate carbon samples for subsequent cassettes will also be evaluated from the homogeneity of the replicate carbon samples from this initial cassette.
- 10.2 Table 1 summarizes the numbers and types of samples which will be taken, sample IDs which will be used and preservatives required for all carbon samples taken from the initial sampling cassette.

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Table 1. Summary of Sample Types Taken and Preservatives

Sample ID	% Moisture (MMES responsibility)		TCE (GC/EC)			Volatiles (Headspace GC/MS or GC/FID)	
	A (16-mL vial)	B (16-mL vial)	C (16-mL vial)	D (16-mL vial)	E (16-mL vial)	F (hdspc. vial)	G (hdspc. vial)
{ID}-0	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-1	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-2	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-3	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-4	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-5	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-6	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-7	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-8	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-9	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-10	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-11	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-12	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-13	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-14	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
{ID}-15	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
blank carbon	none	none	1-mL hex.	1-mL hex.	1-mL hex.	none	none
Totals	17	17	17	17	17	17	17

The total number of 16-mL vials with Teflon-lined lids is $17 \times 5 = 85$ and the total number of headspace vials with crimp caps is $17 \times 2 = 34$. In addition, 4 16-mL vials with Teflon-line caps will be required for B&J water and Fisher Ultra brand hexane.

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- 10.3 In addition to the measurement of percent moisture, MMES will also be responsible for measuring the amount of wet carbon placed in the 16-mL and headspace analysis vials for chemical analysis by the consortium. Table 2 is an example of a data table which MMES should provide with the samples at the conclusion of sampling.

Table 2. Gravimetric Information to be Provided by MMES

Sample ID	TCE (GC/EC)			Volatiles (Headspace GC/MS or GC/FID)	
	C (16-mL vial)	D (16-mL vial)	E (16-mL vial)	F (hdspc. vial)	G (hdspc. vial)
{ID}-0					
{ID}-1					
{ID}-2					
{ID}-3					
{ID}-4					
{ID}-5					
{ID}-6					
{ID}-7					
{ID}-8					
{ID}-9					
{ID}-10					
{ID}-11					
{ID}-12					
{ID}-13					
{ID}-14					
{ID}-15					
blank carbon					
Totals	17	17	17	17	17

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11 REFERENCES

1. Bellar, T.A., and J.J. Lichtenberg, J. Amer. Water Works Assoc., 66(12), pp. 739-744, 1974.
2. Bellar, T.A., and J.J. Lichtenberg, "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds," in Van Hall, ed., Measurement of Organic Pollutants in Water and Wastewater, ASTM STP 686, pp. 108-129, 1979.
3. Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters: Category 11 - Purgeables and Category 12 - Acrolein, Acrylonitrile, and Dichlorodifluoromethane, Report for EPA Contract 68-03-2635 (in preparation).
4. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule, and Proposed Rule," October 26, 1984.
5. Provost, L.P. and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.
6. "EPA Method Validation Study 23, Method 601 (Purgeable Halocarbons)," Report for EPA Contract 68-03-2856 (in preparation).

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Replaces: N/A

Appendix A

The following Site-Specific Health and Safety Plan was developed for this SOP by CDM
Federal Programs and MIMES has reviewed this plan.

CDM FEDERAL PROGRAMS CORPORATION
a subsidiary of Camp Dresser & McKee Inc.

March 14, 1995

Dr. Mason Hughes
Monsanto Company
800 North Lindbergh Boulevard
St. Louis, Missouri 63167

Subject: Health and Safety Plan, Lasagna Project
Document Control No.: 5203-006-CO-BBCB

Dear Dr. Hughes:

Enclosed you will find a copy of the Health and Safety Plan for the carbon sampling later this month. It has been reviewed by Martin Marietta Energy Systems and should be adequate for what is outlined in your sampling procedure.

If you have any questions, please contact me at your convenience.

Sincerely,

CDM FEDERAL PROGRAMS CORPORATION

Bob Hines
Project Manager

CRH/hlw

Enclosure:

cc: Fraser Johnstone, Energy Systems
Oak Ridge Document Control Center
Paducah Document Center

Paducah Gaseous Diffusion Plant Site-Specific Health and Safety Plan		SWMU Number: 091
SWMU NO.: 091 SWMU NAME: Uf6 Cylinder Drop Test Area SITE LOCATION: Between Virginia Avenue and C-745(B) Cylinder Yard SITE CONTACT: Fraser Johnstone PHONE NO.: (502)462-2928 Health and Safety Coordinator: Steven Saunders	WORK ASSIGNMENT NO.: Lasagna PROJECT NO.: 5203 CLIENT: Monsanto CLIENT CONTACT: Dr. Mason Hughes PHONE NO.: (314)694-1466 Health and Safety Manager: Chuck Myers	
OBJECTIVES OF FIELD WORK: Collect a carbon sample from the active Lasagna System	SWMU TYPE: <div style="display: flex; justify-content: space-between;"> <div> ☐ Active ☒ Inactive ☐ Secure ☐ Unsecure ☐ Enclosed space ☐ Landfill </div> <div> ☐ Uncontrolled ☐ Industrial ☐ Recovery ☐ Well Field ☐ Unknown </div> </div> ☒ Other: Drop-Test Site	
DESCRIPTION AND FEATURES: Steel cylinders were submerged in a pit containing a trichloroethylene (TCE)/dry ice slush to freeze cylinders before conducting a drop test to determine structural integrity. No waste is expected to be generated during this task that is not already handled in the performance of other current site activities.		
SURROUNDING ENVIRONMENT: ☐ Residential ☒ Industrial ☐ Rural ☒ Other: Wildlife management area.

Paducah Gaseous Diffusion Plant Site-Specific Health and Safety Plan		SHMU Number: 091
SHMU History: Used to freeze steel cylinders		
Operational Status: Inactive Dates Operated: 1979		
WASTE TYPES: () Liquid (x) Solid () Sludge () Gas () Unknown () Other:		
WASTE CHARACTERISTICS: () Corrosive () Toxic () Inert Gas () Flammable () Other		WORK ZONES: Exclusion, Contamination Reduction, and Support Zones No zones will be set up for this activity.
PRINCIPLE DISPOSAL METHODS AND PRACTICES: All waste generated will be disposed of under current site guidelines.		

Paducah Gaseous Diffusion Plant
Site-Specific Health and Safety Plan

SWMU Number: 091

HAZARDOUS MATERIAL SUMMARY:

CHEMICALS	SOLIDS	SLUDGES	SOLVENTS	OILS	OTHER
☐ Acids	☐ Flyash	☐ Paint	☒ Halogenated (chloro, bromo) Solvents	☐ Oily Wastes	☐ Laboratory
☐ Pickling Liquors	☐ Asbestos	☐ Pigments		☐ Gasoline	☐ Pharmaceutical
☐ Caustics	☐ Milling/Mine Tailings	☐ Metal Sludges	☐ Hydrocarbons	☐ Diesel Oil	☐ Hospital
☐ Pesticides	☐ Ferrous Smelter	☐ POTW Sludges	☐ Alcohols	☐ Lubricants	☒ Radiological
☐ Dyes/Inks	☐ Non-ferrous Smelter	☐ Aluminum	☐ Ketones	☐ PCBs	☐ Municipal
☐ Cyanides	☐ Metals	☐ Distillation Bottoms	☐ Esters	☐ Polynuclear Aromatics	☐ Construction
☐ Phenols	☐ Other	☐ Other	☐ Ethers	☐ Other	☐ Munitions
☐ Halogens			☐ Other		☐ Other
☐ Dioxins					
☐ Other					

OVERALL HAZARD EVALUATION: ☐ High ☐ Medium ☒ Low ☐ Unknown

FIRE/EXPLOSION POTENTIAL: ☐ High ☐ Medium ☒ Low ☐ Unknown

RFI NECESSARY:

Paducah Gaseous Diffusion Plant Site-Specific Health and Safety Plan									
SWMU Number: 091									
KNOWN CONTAMINANTS	HIGHEST OBSERVED CONCENTRATION (units, media, type)	PEL/TLV	IDLH	STEL	WARNING CONCENTRATION	SYMPTOMS/EFFECTS OF ACUTE EXPOSURE	PHOTO- IONIZATION POTENTIAL		
79-01-6 TRICHLOROETHYLENE	NA pci/l GW (2)	50.0 ppm OSHA PEL	1000.0 ppm		82.0 ppm	Headache, vertigo; visual disturbances, tremors, somnolence; nausea, vomiting; irritated eyes; dermatitis; cardiac arrhythmias, paresthesia, carcinogen	9.45 ev		

Paducah Gaseous Diffusion Plant
Site-Specific Health and Safety Plan

SWMU Number: 091

FIELD ACTIVITIES COVERED UNDER THIS PLAN

Task Description	Type	LEVEL OF PROTECTION	
		Primary	CONTINGENCY
<p>1 Cassette Sampling</p> <p>Ensure that all controlling bodies (i.e., Health and Safety Department, Health Physics Department, and the Construction Engineer) have approved the sampling plan. Follow the sampling plan generated by Monsanto.</p> <p>Hazards of Concern: Radiological, Biological, Electrical, Organic Chemicals, Ventilation Problems, Physical Injury, Slips, Trips and Falls</p>	Non-Intrusive	D* Modified	C
<p>2 Clean up</p> <p>All tools and/or supplies that come into direct contact with potentially contaminated materials will be scanned for the presence of organics and radioactivity, then cleaned or disposed of in accordance with established procedures. All such equipment will also be cleared by the Health Physics Department Before leaving the site.</p> <p>Hazards of Concern: Radiological, Biological, Organic Chemicals, Physical Injury, Slips, Trips and Falls</p>	Non-Intrusive	D* Modified	C

* Author of this plan has changed the recommended Level of Protection.

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Paducah Gaseous Diffusion Plant Site-Specific Health and Safety Plan		SWMU Number: 091	
PERSONNEL AND RESPONSIBILITIES (Include subcontractors)			
NAME	RESPONSIBILITIES	ON-SITE TASKS	
Barry Swift	Site Health and Safety Officer	1	2
Chris Athmer (Monsanto)	Field Coordinator	1	2
Jay Clausen (Energy Systems)	Hydrogeologist		
Fraser Johnstone (Energy Systems)	ERWM Project Manager		

Paducah Gaseous Diffusion Plant Site-Specific Health and Safety Plan SWU Number: 091		PROTECTIVE EQUIPMENT	
TASKS: 1 2 -- Primary		TASKS: 1 2 -- 'CONTINGENCY	
LEVEL: D Modified		LEVEL: C	
<ul style="list-style-type: none"> * Work clothing, MMS controlled long-sleeve coveralls. * Boots/shoes, leather or chemical-resistant, steel toe and shank * Boot covers, chemical-resistant * Nitrile and/or latex gloves * Safety glasses with side shields * Hard hat 		<ul style="list-style-type: none"> * Work clothing * Boots (outer), chemical-resistant, steel toe and shank * Boots (inner), chemical-resistant, disposable * Chemical-resistant clothing (hooded, one- or two-piece chemical-resistant suit) * Gloves (inner), latex rubber * Gloves (outer), chemical-resistant * Full-face, MSHA/NIOSH-approved, air purifying respirator, equipped with chemical cartridges for organic and formaldehyde vapors, acid gases, and dusts, mists, and radionuclides (approval # TC-23C-153, or equivalent) * Hard hat 	

Paducah Gaseous Diffusion Plant
Site-Specific Health and Safety Plan

SHMU Number: 091

MONITORING EQUIPMENT

INSTRUMENT	TASKS	ACTION GUIDELINES	COMMENTS (Includes schedules of use)
Radiation Survey Meter	1 2	<p>>3X Background Notify Health and Safety Manager >2mR/hr Notify Health and Safety Manager >20mR/hr Interrupt task/evacuate Notify Construction Engineer</p> <p>>150 counts/min Interrupt task above background Notify Construction Engineer</p>	Alpha, beta, and gamma detectors will be used. Alpha and beta/gamma detectors will be on-site at all times during site activities. Screening will be done before the initiation of site activities and all samples will be scanned as they are removed from the ground.
Photoionization Detector (IHNU) () 11.7 ev () 10.2 ev (✓) 9.8 ev () ____ ev	1 2	<p>0-5 ppm Level D 5-25 ppm Level C (detector tubes when appropriate) 25-50 ppm Level B (or leave area) >50 ppm Leave the area; notify PGDP Project Manager</p>	<p>Use as necessary to characterize area or item in question. Use to screen all samples as removed from the cassette.</p>

Paducah Gaseous Diffusion Plant
Site-Specific Health and Safety Plan

SWMU Number: 091

DECONTAMINATION PROCEDURES (Summarize below and/or attach diagram; discuss use of work zones.)

ATTACH SITE MAP INDICATING EXCLUSION, DECONTAMINATION, AND SUPPORT ZONES AS PAGE TWO

Personalized Decontamination

Disposable gloves will be used. Standard doffing procedures will be followed.

Wash hands and face if necessary with soap and water upon doffing personal protective equipment.

If radiological contamination is encountered, workers will be scanned before exiting the exclusion zone.

Sampling Equipment Decontamination

Heavy Equipment Decontamination

Containment and Disposal Method

All disposable garments will be placed in a suitable drum or container and disposed of through the PGDP facility.

Containment and Disposal Method

Containment and Disposal Method

Paducah Gaseous Diffusion Plant
Site-Specific Health and Safety Plan

SWMU Number: 091

THIS PAGE RESERVED FOR HOSPITAL ROUTE MAP

Paducah Gaseous Diffusion Plant Site-Specific Health and Safety Plan		SWMU Number: 091	
EMERGENCY CONTACTS		EMERGENCY CONTACTS	
Site Telephone: EPA Release Report No.: 1-800-424-8802 Facility Management: (502)441-6211		Health and Safety Manager: Chuck Myers (703)968-0900 Project Manager: Bob Hines Health & Safety Coordinator: Steven Saunders (502)4633006 Client Contact: Dr. Mason Hughes (314)694-1466 Environmental Agency: 1-800-424-8802 State Spill Number: C. Marten 502-564-8685 Fire Department: Plant Superintendent (Bell)333(PAX)555 Police Department: Plant Superintendent (Bell)333(PAX)555 State Police: (502)444-8228 Health Department: (502)444-6431 Poison Control Center: 1-800-722-5725 Occupational Physician:	
CONTINGENCY PLANS: Follow all instructions as described in the Health and Safety Plan, and all other relevant sections of the "Project Plans for Project Management and Field Support for Field Testing the Integrated In Situ Soil Remediation Technology (Lasagna) at Department of Energy's Paducah Gaseous Diffusion Plant" All reference documents and permits will be kept posted and on file in the site office trailer.			
HEALTH AND SAFETY PLAN APPROVALS		MEDICAL EMERGENCY	
Prepared by: Steve Saunders Health and Safety Coordinator Signature Health and Safety Manager Signature Project Manager Signature		Date 03/09/95 Date 3-14-95 Date 3/14/95 Date 3/14/95	
		Hospital Name: Lourdes Hospital (502)444-2444 Address: 1530 Lone Oak Road, Paducah Kentucky 42002 Name of Contact at Hospital: Name of 24-Hour Ambulance: Lourdes Hospital (502)444-2444 Route to Hospital (Attach map with route to hospital) Drive south from PGDP on Hobbs Rd. Turn left on Rt.60 east. Travel approx. 8 or 9 miles to I-24 East. Take I-24 East to exit 7. Proceed straight ahead off the ramp crossing Rt.62. At the next stop light turn left onto Lone Oak Road. The hospital is on the right just after the I-24 underpass.	

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Appendix D. QA/QC Procedures for Evaluating TCE in Carbon SOP

Monsanto Company
Environmental Sciences Center
St. Louis, MO 63167

Document No. ESC-SOP-95-UT613
Issue/Revision Date: October 15, 1995
No. of Pages: 6

STANDARD OPERATING PROCEDURE

Quality Control Experiments Used to Evaluate a Procedure for the Analysis of Trichloroethylene (TCE) in Carbon using Capillary GC/Electron Capture Systems and ^{14}C -TCE

Prepared by:	Signed	<u>B. Mann Ziegler</u>
	Title	<u>Senior Analytical Consultant</u>
	Date	<u>October 18, 1995</u>
Approved by:	Signed	<u>Sa V. Ho</u>
	Title	<u>Fellow & Business Unit Leader</u>
	Date	<u>10/24/95</u>
Accepted for use by QA Supervisor:	Signed	<u>Janet M. Lee</u>
	Title	<u>Quality Assurance Supervisor</u>
	Date	<u>October 18, 1995</u>

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SOP Number

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STANDARD OPERATING PROCEDURE ESC-SOP-95-UT613

Quality Control Experiments Used to Evaluate a Procedure for the Analysis of
Trichloroethylene (TCE) in Carbon using Capillary GC/Electron Capture Systems and ^{14}C -
TCE

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1.0 SCOPE AND APPLICATION

- 1.1 This SOP describes experiments which can be used to determine the amount of TCE which is adsorbed to carbon which is used in the treatment zones of the *Lasagna*TM field experiments being conducted at Paducah, Kentucky. This approach can be used for the determination of TCE concentrations in wet carbon.

2.0 SUMMARY OF METHOD

- 2.1 This method was developed so that partitioning of TCE between wet carbon and hexane could be demonstrated and measured in conjunction with the analysis of TCE in carbon in *Lasagna*TM field experiments. In preliminary laboratory spiking experiments, it was determined that when wet carbon was spiked with a known amount of TCE, approximately 25 - 50% of the TCE could be extracted using a 10:1 ratio of hexane:dry carbon weight. It was found, however, that this partitioning was subject to a number of variables. Therefore, to determine the amount of TCE in carbon from actual field samples, a confirmation method should be available to determine the extraction efficiency for each field sample analyzed. This is particularly important since one of the important determinations for the field experiment is a TCE material balance which can only be calculated accurately if the partitioning of TCE between hexane and wet carbon is known for each sample.
- 2.2 The present SOP outlines laboratory experiments which were conducted to determine whether ¹⁴C-TCE can be used routinely to determine the amounts of TCE which is extracted using this procedure.

3.0 INTERFERENCES

- 3.1 Refer to SW-846 Method 8000 (see Reference 1).
- 3.2 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.
- 3.3 Calibration standards can also be contaminated from residual amounts of pure TCE which may be present in pipettes used for making stock solutions. Great care must be taken so that pipettes that have been previously used with pure

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TCE are not used for low level standards, until they have been confirmed to be clean of this compound.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 Gas Chromatograph: analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detector, analytical columns, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended. The following system, or equivalent, can be used:

<u>System Name</u>	<u>Serial Number</u>
HP 5890 Series II Gas Chromatograph	3022A29344 (shipped 6/27/90)
HP 7673 ALS Injector (18593B)	3013A22217
HP 7673 ALS Controller (18594B)	3018A24560
HP-7673 ALS Tray (18596B)	3021A21718

4.1.2 Capillary Column: Supelco's Catalog No. 2-5320 VOCOL 30 m x 0.53 mm I.D. fused silica capillary column with 3.0 μ film thickness, or equivalent.

4.1.3 Detector: Electron Capture Detector (ECD).

4.2 Microsyringe: 10- and 100- μ L Hamilton syringes (#701 and #710, respectively), or equivalent.

4.3 Automatic Pipettes: Pipetteman, 250 and 1000 μ L, or equivalent.

4.4 Volumetric and Graduated Pipettes: 5- and 10-mL volumetric and graduated pipettes, or equivalent.

4.5 14 C-Counting Equipment: Wallac 1409 liquid scintillation counter, or equivalent.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as a water in which an interferent is not observed at the method detection limit (MDL) of the parameters of interest. Burdick and Jackson distilled-in-glass or equivalent was used. Each bottle should be labeled with the date opened, and an expiration date (normally 1 year).

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5.2 Standard Solutions: Description of calibration standards are located in ESC-SOP-95-UT615 entitled: *"The Analysis of Trichloroethylene (TCE) in Solid, Water and Carbon using Capillary GC/Electron Capture Systems and ^{14}C -TCE"*.

5.3 Methanol: Pesticide quality or equivalent. Store away from other solvents.

Hexane: Pesticide quality or equivalent. Store away from other solvents.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 A more detailed sampling SOP was developed for taking and homogenizing carbon from the sampling cassettes in the field experiments at Paducah, Kentucky. This information is located in ESC-SOP-95-UT614.

7.0 PROCEDURE

7.1 TCE and ^{14}C -TCE are analyzed in a number of experiments. These experiments range from method blanks to carbon and hexane which have been spiked with various known amounts of these materials. SOPs for the GC/EC analysis is located in ESC-SOP-95-UT615 and ^{14}C counting are located in Appendix C. The following paragraphs describe specific experiments and analyses which were performed.

7.2 Method blanks for glassware are prepared by placing 10.0 mLs hexane in vials and for carbon by placing 5 mLs water with 1 g carbon in vials using representative glassware and the same procedures which will be used in the other experiments. The concentrations of TCE and the ^{14}C counts were measured in hexane and water using the procedures outlined in ESC-SOP-95-UT615.

7.3 100% Recovery Standards were prepared for TCE and ^{14}C -TCE by:

i) placing 10.0 mLs hexane in representative glassware, using the same procedures which were used in the other experiments.

ii) removing 0.1 mL hexane.

iii) placing 0.1 mLs of 10 μg TCE/mL methanol or 0.1 mLs of 1 μCi ^{14}C -TCE/mL methanol in the vials. (This amount of ^{14}C -TCE should yield between 15,000 and 30,000 disintegrations per second.)

iv) The concentrations of TCE and the counts of ^{14}C were measured using the same procedures which were used to make these measurements in all other experiments.

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- v) These concentrations and counts were used for the 100% extraction points and were compared to the concentrations and counts in the hexane extracts of the carbon. The % Recovery was calculated from:

$$\% \text{ Recovery TCE} = 100 \times [\text{TCE in Extract}] / [\text{TCE in 100\% Recovery Standards}]$$

7.4 TCE Carbon Spiking and Extraction

7.4.1 TCE Carbon Spiking:

Weight 1 gram of carbon in a 14-mL Pierce vial and add 5.0 mLs reagent water. Allow to equilibrate for 4 hours at room temperature. Insert a pipette tip of the micropipetter to the bottom of the vial and slowly inject 0.1 mLs of a 10 µg/mL methanol solution of TCE into the wet carbon. Allow the solution to equilibrate overnight.

7.4.2 TCE Extraction:

Remove ~ 4 mLs water and replace with 10.0 mLs hexane. Ultrasonically extract for 0.5 hour. Equilibrated at room temperature for 4 hours. Remove 1.0 mL of the hexane extract and place in a vial for GC/EC analysis.

7.4.2.1 Extract Dilution:

For higher TCE concentrations, appropriate dilutions should be made. One-to-ten dilutions may be made by combining 1.0 mL of the extract with 9.0 mLs of hexane. One-to-one hundred dilutions may be made by combining 0.1 mL of the extract with 9.9 mLs of hexane. Great caution should be exercised in re-using automatic pipettors which may inadvertently become contaminated with TCE from high level samples and/or standards. If possible, completely disposable pipettes should be used. Periodic method blanks should be prepared using representative pipettes and solvents which are used for extract dilution.

7.4.3 Addition of ¹⁴C-TCE:

Insert a pipette tip to the bottom of the vial and slowly inject 0.1 mLs of a ¹⁴C-TCE in methanol solution to the remaining 9.0 mLs of the hexane extract. Add an additional 0.9 mLs hexane to the extract. Ultrasonically extract for 0.5 hours. After the vial reaches room temperature, remove one mL of the hexane extract for ¹⁴C counting.

7.5 ¹⁴C-TCE Carbon Spiking and Extraction

7.5.1 ¹⁴C-TCE Carbon Spiking:

Weight 1 gram of carbon in a 14-mL Pierce vial and add 5.0 mLs reagent water. Allow to equilibrate for 4 hours at room temperature.

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Insert a pipette tip of the micropipetter to the bottom of the vial and slowly inject 0.1 mLs of a $1 \mu\text{Ci } ^{14}\text{C-TCE/mL}$ methanol into the wet carbon. Allow the solution to equilibrate overnight.

7.5.2 $^{14}\text{C-TCE}$ Extraction:

Remove ~ 4 mLs water and replace with 10.0 mLs hexane. Count an aliquot of this water for ^{14}C . Ultrasonically extract in hexane for 0.5 hour. After the vial reaches room temperature, remove 1.0 mL of the hexane extract and analyze for ^{14}C .

8.0 HANDLING RADIOACTIVE MATERIALS

- 8.1 All $^{14}\text{C-TCE}$ must be handled in accordance with Monsanto's safety requirements. These requirements include the use of specially designated hoods and equipment, and safety training for personnel handling these materials. Further information can be obtained from Monsanto's safety manual and in Appendix C of this SOP.

9.0 QUALITY CONTROL

- 9.1 Refer to Chapter One - QA/QC in SW-846 (Reference 1) for specific quality control procedures and SW-846 Method 8000 for gas chromatographic procedures.
- 9.2 Mandatory quality control to validate the GC system operation is found in Method 8000, Section 8.6.
- 9.2.1 The quality control check sample concentrate (Method 8000, Section 8.6) should contain each parameter of interest at a concentration of $1 \mu\text{g/mL}$ in hexane.

10.0 METHOD PERFORMANCE

- 10.1 This method was tested by Monsanto's Environmental Sciences Center as part of the *Lasagna*TM field study funded by the Department of Energy contract DE-AR21-94MC31185.
- 10.2 The accuracy and precision obtained will be determined by the sample matrix, sample introduction technique, and by the calibration procedure used.
- 10.3 Appendix A shows tables of results obtained for a set experiments using this protocol.
- 10.4 Appendix B shows a table of results for the determination of % moisture measured in carbon taken from the *Lasagna*TM field experiment in Paducah.

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This information is required if the TCE concentrations are calculated on a dry-weight basis rather than an "as-is" basis.

11.0 REFERENCES

1. "Test Methods for Evaluating Solid Waste", SW-846, Third Edition, November 1986.
2. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.
3. Provost, L.P. and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.
4. Monsanto Notebook Pages 5,793,917 - 5,793,923 and 5,793,930.

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APPENDIX A. DETERMINATION OF TCE EXTRACTION EFFICIENCIES USING NORMAL AND ¹⁴C-TCE

Experiments were conducted using the procedures outlined in the above SOP. Table A-1 and Figure A-1 summarizes recoveries measured for TCE and ¹⁴C-TCE using this procedure. It appears that for TCE concentrations well above 100 ppm, an extraction efficiency of 33.3% can be used. In addition, ¹⁴C-TCE can be used to give additional information to confirm what the extraction efficiency is for carbon samples obtained from the field experiments at Paducah.

Table A-1. TCE Recoveries Using Hexane as Solvent and
¹⁴C-TCE for correction.

Experiment	% Recovered	Average	Std Dev	% Rel SD
TCE Calibration	100.00%	0.95	0.04	4.34%
TCE Extraction	19.61%	0.43	0.03	6.03%
TCE Extraction- 14-C corrected	161.65%	3.47	0.88	25.47%
14C-TCE Calibration	100.00%	22,048.70	1,157.08	5.25%
14C-TCE Extraction	21.36%	4,729.71	321.99	6.81%
TCE Calibration	100.00%	91.29	2.28	2.49%
TCE Extraction	32.66%	29.82	2.89	9.69%
TCE Extraction- 14-C corrected	104.27%	95.19	12.67	10.89%
TCE Calibration	100.00%	158.20	3.31	2.09%
TCE Extraction	28.62%	45.27	4.64	10.24%
TCE Extraction- 14-C corrected	162.76%	257.50	91.84	35.67%
TCE Calibration	100.00%	1,534.20	44.01	2.87%
TCE Extraction	37.15%	570.00	66.59	11.68%
TCE Extraction- 14-C corrected	133.98%	2,055.45	776.66	37.79%

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APPENDIX B. MOISTURE DETERMINATION IN WET CARBON

On March 28 and 29, 1995, carbon samples were removed from the sampling cassettes in the Paducah DOE field experiment and % Moisture was determined using procedures outlined in ESC-SOP-95-UT614. The table below shows that 50% can be used as a good approximation for the amount of moisture in carbon.

	Gravimetric Sample A			Gravimetric Sample B		
	Wet C	Wt. Lost	%Moist.	Wet C	Wt. Lost	%Moist.
{C4-7}-4	2.68	1.30	48.51%	3.20	1.60	50.00%
{C4-7}-5	3.39	1.63	48.08%	4.52	2.24	49.56%
{C4-7}-6	4.05	1.94	47.90%	4.70	2.27	48.30%
{C4-7}-7	3.40	1.61	47.35%	3.54	1.74	49.15%
{C4-7}-8	3.78	1.97	52.12%	4.41	2.33	52.83%
{C4-7}-9	4.79	2.42	50.52%	4.06	2.11	51.97%
{C4-7}-10	4.70	2.43	51.70%	4.58	2.49	54.37%
{C4-7}-11	4.88	2.39	48.98%	5.14	2.52	49.03%
{C4-7}-12	3.91	1.81	46.29%	3.76	1.80	47.87%
{C4-7}-13	5.06	2.31	45.65%	3.86	1.84	47.67%
{C4-7}-14	3.54	1.62	45.76%	3.52	1.72	48.86%
{C4-7}-15	5.10	2.65	51.96%	4.96	2.25	45.36%
{C4-7}-Blank	3.11	0.00	0.00%	3.07	0.00	0.00%
	Average = 48.74%			Average = 49.58%		
	SD = 2.36%			SD = 2.46%		
	%RSD = 4.84%			%RSD = 4.96%		

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APPENDIX C. PROCEDURES USED FOR HANDLING RADIOACTIVE MATERIALS

The following pages summarize the Radiochemical Project Checklist used for the analysis of ^{14}C -TCE in carbon extracts.

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DATE REVIEWED: 10/20/94

RADIOCHEMICAL PROJECT CHECKLIST

APPROVAL

The attached project checklist has been reviewed and approved by the Radiation Safety Officer and the Unit Radiation Safety Committee member.

Radiation Safety Officer

Radiation Safety Committee Member

The following changes or additional information is required:

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Monsanto

From

(Name -Location-Phone) Robert G. Orth -U1F-41271

FAX-41531

The Key to Environmental Safety

Date: 10-13-94

CC. Sa Ho U4E

J.W. Litschigi S3C

C. Evans S3C

Subject: Radiochemical Project.

To: J.V. O'Reilly

The carbon-hexane partitioning study of trichloroethylene, TCE, project we will be undertaking involves the use of low levels of radioactive materials. The project objectives are to determine the partitioning coefficients and recovery of TCE from hexane/carbon mixtures. The procedure is an amended procedure for soil partitioning study already submitted. The use of the radioisotopes is required so that reliable and precise data can be obtained in a cost effective and time efficient manner.

The radioisotope used is a ^{14}C -labeled TCE having a specific activity of 14.2 mCi/mole and a total activity of 250 microcuries. This solution is already in house for another project. The original material was diluted in 1 mL of non labeled TCE yielding a specific activity of about 171 microcuries per gram. Further dilutions are made in the determination so that the total concentration in hexane of TCE will not exceed 1000 dpm/mL. The resulting radioactivity in the solutions will not exceed 2 nCi/mL of the hexane solution. Total used will not exceed 0.4 microcuries for the total experiment.

At this time it is projected that I will be the only individual involved with the actual use of the radioactive materials. My background is a Ph.D. in Physical Chemistry. I also was a radio chemist with the state of Florida for 3 years and had extensive training then in handling of radioactive materials. My latest radiochemical training course was September 1994. I have a film badge which is turned in for evaluation through Connie Evans of Analytical Sciences center.

Details of the experimental methods are outlined in the Attached protocol. the general approach will require preparation of hexane solutions spiked with radiolabeled TCE which are then exposed to the appropriate carbon matrix in sealed container. Because of the low radioactivity present in these samples, no additional specialized equipment will be used. Normal laboratory glassware and other radioactive contaminated waste will be disposed of according to current site regulations as noted in the Radiochemical operations manual. The volume of solid waste should not exceed 1 kg. The volume of liquid waste should not exceed 2 L.

* Radiochemicals will be maintained in a locked freezer in laboratory U-419 when not in use. Experimentation will be conducted in a fume hood in U112 currently setup for radiochemical work, including periodic wipe test for contamination surveys. A survey will be performed at the beginning of the day and at the end during the experiment. Access to the laboratories are not and cannot be restricted. However, access to fume hoods in which the work will be done is limited to those using the radiochemicals.

Because of the low levels of radioactivity per experiment, no emergency situations are possible. If any spills occur, they will be cleaned up in accordance with current site regulations and wipe test will be requested.

If you require further information, please call.

Robert Orth
Fellow

A handwritten signature of Robert Orth in cursive.

Appendix E. TCE Analysis in Soil, Water and Carbon SOP

Monsanto Company
Environmental Sciences Center
St. Louis, MO 63167

Document No. ESC-SOP-95-UT615
Issue/Revision Date: September 1, 1995
No. of Pages: 10

STANDARD OPERATING PROCEDURE

The Analysis of Trichloroethylene (TCE) in Soil, Water, and Carbon using Capillary
GC/Electron Capture Systems and ^{14}C -TCE

Prepared by:	Signed	<u>B. Mason Hughes</u>
	Title	<u>Senior Analytical Consultant</u>
	Date	<u>October 18, 1995</u>
Approved by:	Signed	<u>Sa V. Ho</u>
	Title	<u>Fellow & Business Unit Leader</u>
	Date	<u>10/23/95</u>
Accepted for use by QA Supervisor:	Signed	<u>Janet M. Lee</u>
	Title	<u>Quality Assurance Supervisor</u>
	Date	<u>October 18, 1995</u>

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1.0 SCOPE AND APPLICATION

- 1.1 This method is an adaptation of a direct injection GC/EC method (RCRA SW-846 Method 8010) which can be used to determine the concentration of various volatile halogenated organic compounds in organic solvents. This SOP describes a method which is a deviation from 8010 in that wide bore capillary columns and electron capture detection is used. This method is limited to trichloroethylene (TCE), due to its excellent electron capture sensitivity. Appendix A of ESC-SOP-95-UT613 also describes the results from the determination of the concentrations of TCE in carbon, using ^{14}C -TCE as a measure of extraction efficiency of the TCE using hexane as the extracting solvent. The complete SOP for this procedure is shown in ESC-SOP-95-UT613 entitled *"Quality Control Experiments Used to Evaluate a Procedure for the Analysis of Trichloroethylene (TCE) in Carbon using Capillary GC/Electron Capture Systems and ^{14}C -TCE"*.

2.0 SUMMARY OF METHOD

- 2.1 RCRA SW-846 Method 8010 provides gas chromatographic conditions for the detection of halogenated volatile organic compounds. The present SOP is a modification of 8010.
- 2.2 RCRA SW-846 Method 8010 suggests two optional gas chromatographic packed columns that may also be helpful in resolving the analytes from interferences that may occur and for analyte confirmation. This confirmation is not necessary if an analyte has been confirmed to be present from previous analyses. The present SOP assumes that confirmation analyses have been conducted to identify TCE in samples of interest.

3.0 INTERFERENCES

- 3.1 Refer to SW-846 Method 8000 "Gas Chromatography".
- 3.2 Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. A field sample blank prepared from reagent water and carried through sampling and subsequent storage and handling can serve as a check on such contamination.
- 3.3 Calibration standards can also be contaminated from residual amounts of pure TCE which may be present in pipettes used for making stock solutions. Great care must be taken so that pipettes that have been previously used with pure TCE are not used for low level standards, until they have been confirmed to be clean of this compound.

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4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 Gas Chromatograph: analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detector, analytical columns, recorder, gases, and syringes. A data system for measuring peak heights and/or peak areas is recommended. The following system, or equivalent, can be used:

<u>System Name</u>	<u>Serial Number</u>
HP 5890 Series II Gas Chromatograph	3022A29344 (shipped 6/27/90)
HP 7673 ALS Injector (18593B)	3013A22217
HP 7673 ALS Controller (18594B)	3018A24560
HP-7673 ALS Tray (18596B)	3021A21718

4.1.2 Capillary Column: Supelco's Catalog No. 2-5320 VOCOL 30 m x 0.53 mm I.D. fused silica capillary column with 3.0 μ film thickness, or equivalent.

4.1.3 Detector: Electron Capture Detector (ECD), or equivalent.

4.2 Microsyringe: 10- and 100- μ L Hamilton syringes (#701 and #710, respectively), or equivalent.

4.3 Automatic Pipettes: Pipetteman and Oxford BenchMates, 250 and 1000 μ L, or equivalent.

4.4 Volumetric and Graduated Pipettes: 5- and 10-mL volumetric and graduated pipettes, or equivalent.

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as a water in which an interferent is not observed at the method detection limit (MDL) of the parameters of interest. Burdick and Jackson distilled-in-glass or equivalent was used.

5.2 Stock standards: Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare a 100,000 μ g/mL stock standard of trichloroethylene (TCE) in hexane by adding 0.68 mL TCE to 9.3 mLs hexane using disposable pipettes. Because of the toxicity of some of the organohalides, primary dilutions of these should be prepared in a hood.

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- 5.2.1 Place 9.3 mLs of hexane in a 14-mL vial with teflon-lined lid using a graduated 10-mL pipette. Using a 0.5 mL volumetric pipette and a 100 μ L Hamilton syringe (#710), add 0.68 mLs TCE. Assuming a density of 1.464 g/mL, this volume of TCE corresponds to 1.0 grams.
- 5.2.2 Store, with minimal headspace, at 4 °C and protect from light.
- 5.2.3 Standards should be replaced after 6 months, or sooner if comparison with check standards indicates a problem.
- 5.3 Secondary dilution standards: Using the stock standard solution prepared at 100,000 μ g/mL, prepare secondary dilution standards in hexane and methanol, as needed. The secondary dilution standards should be prepared at concentrations such that the calibration standards prepared in Section 5.4 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace for volatiles and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.
- 5.3.1 Stock (1,000). Purchase from Supelco, or equivalent, or prepare 1,000 μ g/mL secondary dilution standards by placing 0.1 mLs of the Stock Standard in 9.9 mLs of hexane or methanol in 14-mL Pierce vials with teflon-lined lids. (Use a volumetric 10-mL pipette to place 10.0 mLs into the vial and remove 0.1 mLs using a micropipette.)
- 5.3.2 Stock (10). Prepare 10 μ g/mL secondary dilution standards by placing 0.1 mLs of the Stock (1,000) Standards prepared in 5.3.1 in 9.9 mLs of hexane or methanol in 14-mL Pierce vials with teflon-lined lids. (Use a volumetric 10-mL pipette to place 10.0 mLs into the vial and remove 0.1 mLs using a micropipette.)
- 5.4 Calibration standards: A minimum of five concentration levels should be prepared in hexane from the secondary dilution of the stock standard described in 5.3.2. One of the concentration levels should be at a concentration which is near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC.
- 5.4.1 Prepare calibration standards in hexane using the following table.

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Table 1. Volumes of Stock (10) and Stock (0.1) and Solvents used to prepare calibration curves in hexane.

	Concentration ($\mu\text{g/mL}$)	Volume (mLs)	
		Stock (10)	Solvent
	2.5	2.5	7.5
	1.0	1.0	9.0
	0.50	0.50	9.50
	0.25	0.25	9.75
Stock (0.1) →	0.10	0.10	9.9
		Stock (0.1)	
	0.05	5.0	5.0
	0.025	2.5	7.5
	0.010	1.0	9.0
	0.005	0.5	9.5

- 5.5 **Note:** Internal Standards are not normally used for this method. Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.
- 5.6 **Note:** Surrogate Standards are not normally used for this method. However, see Appendix A for a discussion of the use of ^{14}C -TCE to determine carbon extraction efficiencies. Surrogate standards: The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate halocarbons.
- 5.7 Methanol: pesticide quality or equivalent. Store away from other solvents. The date opened, expiration date (normally 1 year), and analysts initials, should be indicated on the bottle.

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Hexane: pesticide quality or equivalent. Store away from other solvents. The date opened, expiration date (normally 1 year), and analysts initials, should be indicated on the bottle.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

- 6.1 See the introductory material in Chapter Four. Organic Analytes, in SW-846 (Reference 3).

7.0 PROCEDURE

- 7.1 TCE is extracted using hexane. The extract is introduced into the gas chromatograph by direct injection. A calibration curve of TCE in the solvent used for extraction is used to calculate the concentration of TCE in the solvent. This information is combined with the sample size, extract volume, dilution factor, and extraction efficiency, to calculate the concentration of TCE in the soil, water, and carbon. The flow sheet shown in Figure 3 summarizes these steps.

7.2 Extraction

- 7.2.1 Soil Samples Between 1 and 3 grams of soil is accurately weighed in a 14-mL Pierce vial with Teflon-lined lid. Ten mLs hexane is added. The vial is manually shaken for 10 seconds. Using these conditions, the extract can be analyzed directly for TCE concentrations which range between 0.05 and 10.0 ug TCE/g soil.

- 7.2.2 Water Samples 2.0 mL water is added to 1.0 mL hexane in a 4-mL vial with Teflon-lined lid. The vial is manually shaken for a few seconds. Using these conditions, the extract can be analyzed directly for TCE concentrations which range between 0.0025 and 0.5 ug TCE/g water.

- 7.2.3 Carbon Samples Between 1 and 3 grams of carbon is accurately weighed in a 14-mL Pierce vial with Teflon-lined lid. Ten mLs hexane is added. The 14-mL vial is placed in an ultrasonic bath for 30 minutes. The extracts can be analyzed after equilibration at room temperature for 4 hours. Using these conditions, the extract can be analyzed directly for TCE concentrations which range between 0.1 and 10.0 ug TCE/g carbon.

7.3 Extract Dilution:

- 7.3.1 For higher TCE concentrations, appropriate dilutions should be made. One-to-ten dilutions may be made by combining 1.0 mL of the extract

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with 9.0 mLs of the extracting solvent. One-to-one hundred dilutions may be made by combining 0.1 mL of the extract with 9.9 mLs of the extracting solvent. Great caution should be exercised in re-using automatic pipetters which may inadvertently become contaminated with TCE from high level samples and/or standards. If possible, completely disposable pipettes should be used. Periodic method blanks should be prepared using representative pipettes and solvents which are used for extract dilution.

7.4 Gas chromatography conditions (Recommended):

7.4.1 Capillary Column:

Helium gas pressure: 4 psi

Column oven temperature: Hold at 40°C for 4 min;
program at 10 °C/min to 65 °C;
program at 20 °C/min to 150 °C.

Injection: Splitless, with backflush turned on at 0.5 minutes.

Injection Port: 250 °C.

7.5 Calibration: Refer to Method 8000 in SW-846 (Reference 3) for proper calibration techniques.

7.5.1 Calibration must take place using the same sample introduction method that will be used to analyze actual samples.

7.5.2 The procedure for internal or external calibration may be used. Refer to Method 8000 for a description of each of these procedures. (This SOP recommends the external standard method of calibration.)

7.6 Gas chromatographic analysis:

7.6.1 Introduce volatile compounds into the gas chromatograph by directly injecting a sample extract.

7.6.2 Follow Section 7.6 in Method 8000 "*Gas Chromatography*" for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

7.6.3 Figures 1 and 2 show chromatograms obtained from the analysis of TCE standards in hexane.

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- 7.6.4 Record the volume of the sample extract and sample volume or sample weight extracted and the resulting TCE peak sizes (in area units or peak heights).
- 7.6.5 Calculation of concentration is discussed in Section 7.8 of Method 8000.
- 7.6.6 If analytical interferences are suspected, or for the purpose of confirmation, analysis using the second GC column or GC/MS is recommended.
- 7.6.7 If the response for a peak is off-scale or outside the calibration range, prepare a dilution of the sample with the extracting solvent. The dilution should be performed on the original extract.

8.0 CALCULATIONS

- 8.1 The concentration of TCE per gram of soil, water, and carbon is calculated using the following equation:

$$\text{TCE Concentration } (\mu\text{g/g}) = [\text{Extract Concentration } (\mu\text{g/mL})] \times [\text{Extract Volume (mL)}] \times \text{Dilution Factor} \times [1/\text{Extraction Efficiency}] \times [1/\text{Sample Size (g)}] \quad (\text{Equation 1})$$

where: Dilution Factor = $[\text{Volume} + \text{Dilution Volume}]/[\text{Volume}]$

Extraction Efficiency = 1.0 for soil and water and 0.333 for carbon

- 8.2. Calculation of the concentration of TCE in dry carbon must be made in most cases. Therefore the amount moisture should be determined before this calculation can be made. For the determination of TCE in carbon associated with the *Lasagna*TM field experiments this information was determined using the procedures outlined in the ESC-SOP-95-UT614 entitled "*Compositing and Sampling Carbon in Sampling Cassettes from the Lasagna*TM Field Experiment at Paducah, Kentucky for DOE Contract No. DE-AR21-94MC31185". Appendix B of the present SOP shows the gravimetric information from which it was determined that a typical amount of moisture in the carbon used in the field experiments is 50%. The following equation is used to correct for % moisture:

$$\text{TCE Concentration } (\mu\text{g/g} - \text{dry weight}) = \text{TCE Concentration } (\mu\text{g/g}) / [\% \text{ Moisture}] \quad (\text{Equation 2})$$

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9.0 QUALITY CONTROL

- 9.1 Refer to Chapter One - QA/QC of SW-846 (Reference 3) for specific quality control procedures and Method 8000 for gas chromatographic procedures.
- 9.2 Mandatory quality control to validate the GC system operation is found in Method 8000, Section 8.6.
 - 8.2.1 The quality control check sample concentrate (Method 8000, Section 8.6) should contain each parameter of interest at a concentration of 1 ug/mL in hexane.
- 9.3 (Surrogate compounds are not normally used with this SOP.) Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if recovery is within limits (limits established by performing QC procedure outlined in Method 8000, Section 8.10).
 - 9.3.1 If recovery is not within limits, the following is required.
 - ° Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
 - ° Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
 - ° Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

10.0 METHOD PERFORMANCE

- 10.1 This method was tested by Monsanto's Environmental Sciences Center as part of the *Lasagna*TM field study funded by the Department of Energy under Contract Number DE-AR21-94MC31185.

11.0 REFERENCES

1. Bellar, T.A., and J.J. Lichtenberg, J. Amer. Water Works Assoc., 66(12), pp. 739-744, 1974.
2. Bellar, T.A., and J.J. Lichtenberg, "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds," in Van Hall, ed., Measurement of Organic Pollutants in Water and Wastewater, ASTM STP 686, pp. 108-129, 1979.
3. "Test Methods for Evaluating Solid Waster", SW-846, Third Edition, November 1986.

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4. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.
5. Provost, L.P. and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.
6. Monsanto Notebook Pages 5,793,901 - 5,793,930.

Figure 1. GC/EC chromatogram from the analysis of a hexane solution containing 0.5 $\mu\text{g/mL}$ TCE and other chlorinated solvents.

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Figure 2. GC/EC chromatograms from the analysis of TCE at 0.25, 0.5, and 1.0 $\mu\text{g/mL}$.

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Figure 3. TCE Analysis Method for Water, Soil, and Carbon

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APPENDIX A. Determination of TCE extraction efficiencies using normal and ^{14}C -TCE

Experiments were conducted using the procedures outlined in ESC-SOP-95-UT613. Table A-1 and Figure A-1 summarizes recoveries measured for TCE and ^{14}C -TCE using that procedure. It appears that for TCE concentrations well above 100 ppm, an extraction efficiency of 33.3% can be used. In addition, ^{14}C -TCE can be used to give additional information to confirm what the extraction efficiency is for carbon samples obtained from the field experiments at Paducah.

Table A-1. TCE Recoveries Using Hexane as Solvent and ^{14}C -TCE for correction.

Experiment	% Recovered	Average	Std Dev	% Rel SD
TCE Calibration	100.00%	0.95	0.04	4.34%
TCE Extraction	19.61%	0.43	0.03	6.03%
TCE Extraction- 14-C corrected	161.65%	3.47	0.88	25.47%
14C-TCE Calibration	100.00%	22,048.70	1,157.08	5.25%
14C-TCE Extraction	21.36%	4,729.71	321.99	6.81%
TCE Calibration	100.00%	91.29	2.28	2.49%
TCE Extraction	32.66%	29.82	2.89	9.69%
TCE Extraction- 14-C corrected	104.27%	95.19	12.67	10.89%
TCE Calibration	100.00%	158.20	3.31	2.09%
TCE Extraction	28.62%	45.27	4.64	10.24%
TCE Extraction- 14-C corrected	162.76%	257.50	91.84	35.67%
TCE Calibration	100.00%	1,534.20	44.01	2.87%
TCE Extraction	37.15%	570.00	66.59	11.68%
TCE Extraction- 14-C corrected	133.98%	2,055.45	776.66	37.79%

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APPENDIX B. MOISTURE DETERMINATION IN CARBON

On March 20 and 21, 1995, carbon samples were removed from the sampling cassettes in the Paducah DOE field experiment and % Moisture was determined using procedures outlined in ESC-SOP-95-UT614. The table below shows that 50% can be used as a good approximation for the amount of moisture in carbon.

	Gravimetric Sample A			Gravimetric Sample B		
	Wet C	Wt. Lost	%Moist.	Wet C	Wt. Lost	%Moist.
{C4-7}-4	2.68	1.30	48.51%	3.20	1.60	50.00%
{C4-7}-5	3.39	1.63	48.08%	4.52	2.24	49.56%
{C4-7}-6	4.05	1.94	47.90%	4.70	2.27	48.30%
{C4-7}-7	3.40	1.61	47.35%	3.54	1.74	49.15%
{C4-7}-8	3.78	1.97	52.12%	4.41	2.33	52.83%
{C4-7}-9	4.79	2.42	50.52%	4.06	2.11	51.97%
{C4-7}-10	4.70	2.43	51.70%	4.58	2.49	54.37%
{C4-7}-11	4.88	2.39	48.98%	5.14	2.52	49.03%
{C4-7}-12	3.91	1.81	46.29%	3.76	1.80	47.87%
{C4-7}-13	5.06	2.31	45.65%	3.86	1.84	47.67%
{C4-7}-14	3.54	1.62	45.76%	3.52	1.72	48.86%
{C4-7}-15	5.10	2.65	51.96%	4.96	2.25	45.36%
{C4-7}-Blank	3.11	0.00	0.00%	3.07	0.00	0.00%
	Average = 48.74%			Average = 49.58%		
	SD = 2.36%			SD = 2.46%		
	%RSD = 4.84%			%RSD = 4.96%		

Appendix F. Carbon Cassette QA/QC Analysis Results

Summary of Replicate Cassette Analyses

Sample ID	Carbon TCE (ug/gram)			
		Average	SD	%RSD
[Control]	4,730.35	4,652.82	109.64	2.36%
[Control]	4,575.30			
[Control]	6,691.18			
[Control]	5,950.85	6,321.02	523.50	8.28%
[Anode-2]	4,897.98	4,616.85	397.57	8.61%
[Anode-2]	4,335.73			
[Anode-2]	10,740.10			
[Anode-2]	9,204.72	9,972.41	1,085.68	10.89%
[Anode-5]	301.55	304.33	80.23	26.36%
[Anode-5]	247.59			
[Anode-5]	1,416.50			
[Anode-5]	1,208.19	1,312.34	147.30	11.22%
{C1-3}	1,370.67	1,197.87	244.37	20.40%
{C1-3}	1,025.07			
[C1-3]	5,605.60			
[C1-3]	4,471.34	5,038.47	802.04	15.92%
[C1-7]	1,035.16	893.66	200.11	22.39%
[C1-7]	752.16			
[C1-7]	1,789.65			
[C1-7]	1,876.60	1,833.13	61.48	3.35%
[C2-2]	2,396.30	2,143.52	357.48	16.68%
[C2-2]	1,890.75			
[C2-2]	14,639.14			
[C2-2]	22,774.15	18,706.64	5,752.32	30.75%
[C2-6]	3,896.20	3,560.11	475.31	13.35%
[C2-6]	3,224.02			
[C2-6]	3,425.44			
[C3-3]	3,140.88	3,283.16	201.21	6.13%
[C3-3]	15,864.89			
[C3-3]	15,373.05			
[C3-3]	15,373.05	15,618.97	347.78	2.23%
[C3-7]	3,601.16	3,285.32	446.66	13.60%
[C3-7]	2,969.49			
[C3-7]	8,953.98			
[C3-7]	6,855.76	7,904.87	1,483.67	18.77%
[C4-3]	1,636.25	1,618.02	25.78	1.59%
[C4-3]	1,599.80			
[C4-3]	5,470.93			
[C4-3]	4,757.88	5,114.41	504.20	9.86%

Summary of Replicate Cassette Analyses

Sample ID	Carbon TCE (ug/gram)			
		Average	SD	%RSD
{C4-7}	36.91	35.79	1.59	4.45%
{C4-7}	34.66			
{C4-7}	614.51			
{C4-7}	516.13	565.32	69.57	12.31%
[Cathode-2]	7,690.13	6,418.34	1,798.59	28.02%
[Cathode-2]	5,146.54			
[Cathode-2]	10,328.41			
[Cathode-2]	12,496.25	11,412.33	1,532.90	13.43%
[Cathode-5]	13,745.27	14,309.21	797.53	5.57%
[Cathode-5]	14,873.15			
[Cathode-5]	2,584.31			
[Cathode-5]	2,881.40	2,732.86	210.07	7.69%
		Average	SD	%RSD
		5,314.07	706.26	12.57%

Appendix G. DuPont's Description of Kriging of Pre-test and Post-test Data

GEOSTATISTICS DATA EVALUATION

Scope and Objectives

Results from the *in-situ* remediation project at the Paducah Kentucky, *Lasagna*TM site indicate that significant trichloroethene (TCE) concentrations have been recovered from impacted soils using the experimental electro-osmosis technique. Pre-treatment soil data TCE concentrations were compared to post-treatment carbon wickdrain sampling filter TCE concentrations. This comparative evaluation provides information for understanding the predictive capabilities of using geostatistics for estimating unknown sample area concentrations. This discussion also highlights the mass of chemical recovered via the *Lasagna*TM remediation technique. The primary objective of this data evaluation was to assess whether kriging mapping methods based on geostatistical models are a reliable predictive tool for estimating unknown sample concentrations.

Geostatistics Data Evaluation

Geostatistical methods provide estimates of unsampled locations by correlating spatial variation relationships, such as distance and direction, and computing weighted averages of underlying statistical distributions of the data set. Kriged output maps (available from the authors) provide interpolation grid displays of unsampled locations for each (pre-test) investigated soil depth. To spatially correlate the structure of a concentration variable a variogram model was developed for each zone or soil interval (i.e., 3' through 15' below ground surface [bgs]). The variogram quantifies correlations across unsampled areas by computing the variance of all pairs of

measurements which satisfy distance and direction for each variable. The variogram model allows for kriging weights to be assigned for unsampled locations. The kriging method used for the *Lasagna*TM project was "ordinary kriging". Ordinary kriging estimates are interpolated values of the weighted moving average of sample values within a local search neighborhood.

It is important to note that the kriging mapping method essentially smoothes the interpolated data to fit the underlying statistical distribution. Because of the smoothing effect of kriged estimates, variability is inherent for each estimated kriged data point. Therefore, the true concentration of unsampled locations is unknown, and some level of uncertainty exists for each interpolated location. This project is unique because previously unsampled locations (pre-test data) can be compared to same-sample locations (post-test data) to measure the effectiveness of the kriging technique for predicting concentration values. Nonetheless, considerations, such as analytical variability, lithologic complexity, and remediation controls add to the uncertainty dilemma and should be understood as potential factors which may influence results. Moreover, such uncertainty factors need to be recognized as potential limitations prior to judging geostatistics as a holistic predictive tool. Before discussing the comparative TCE data results, it is important to address key assumptions and observations of this study:

- Most earth science data sets exhibit lognormal statistical distributions. However, 11 out of the 13 *Lasagna*TM data sets exhibited normal distributions. Normality was assumed for all data sets for consistency.

- Only 12 data values (i.e., pre-test borings) exist at the site. Generally, variogram modeling requires a minimum of 12 to 15 data values for good kriged results. As such, questionable variograms exist for six of the study intervals.
- Several of the kriged output estimation error grids contained relatively high standard deviations. Therefore, the relative uncertainty of the kriged TCE concentrations associated with these higher error grids is high. An example is Zone H8 (10' bgs) with an estimation error of 32.5 versus A1(3' bgs) of 7.85. Coincident with the higher estimation kriged errors were poorer TCE mass recovered percentages (<40%) in Zones G7(9'), H8(10'), and I9(11').
- TCE extraction efficiency in the carbon filters is highly variable and may account for as much as 25% of observed mass recovery.
- Based on percent mass recoveries, it appears that the uppermost and lowermost strata intervals are capturing higher TCE mass than middle strata intervals.

Summary of Pre-test data and Post-test data

Table 1 summarizes the results of TCE concentrations at interpolated (i.e., kriged) locations - Pre-test data; and, TCE concentrations at carbon filter locations - Post-test data. Calculations were made to derive a mass-to-mass comparison of each sampling event result. The (%) mass (shaded) recovered highlights the amount of TCE detected in the carbon filter samples assuming the kriged locations represents a 100% 'real' TCE concentration. As depicted on Table 1, total TCE mass recovered for each interval ranged

from 30% to 90%. In some instances, percent mass recovered exceed 100% primarily due to underestimation of kriged variable.

Table 2 summarizes the results of four discrete soil intervals (3', 6', 10', and 15' bgs) which were examined more closely to understand the predictive capabilities of geostatistical modeling. The kriged data reported on Table 1 were qualified by evaluating whether the resulting kriged estimate was over-or-underestimated based on the spatial structure of the kriged map. This evaluation involved the calculation of each kriged data point and its standard deviation relative to the overall kriged output estimation error. For example, Zone A1 (3' bgs) carbon filter locations C1-3 and C1-7 were overestimated, whereas, C2-2, C2-6, C3-3, and C3-7 were underestimated based on a kriged error plot. Depending on where the location was over-or-undercompensated relative to the overall kriged grid estimation error, a factor of the grid location's standard deviation (i.e., 1X or 2X St.Dev) was applied to derive a 'corrected' kriged TCE concentration at the unsampled location. The 'corrected' value was then calculated for mass and correlated to the mass in carbon filter.

As shown on Table 2, 'corrected' kriged TCE concentrations represent a more realistic estimate of interpolated location concentrations because of error-weight method accounts for the boundaries of uncertainty of the possible statistical range of values. This error-weight method is consistent with statistical central limit theorem principles and is viewed as a viable tool for reducing uncertainty when estimating/predicting potential variability for kriged output values.

Table 1
Lasagna TCE Evaluation: Pre-test Kriged Values versus Post-test Carbon Data

Sample Name	Depth (feet)	No. of Samp.*	PRE-TEST DATA		POST-TEST DATA		Mass Recovered	
			TCE Kriged Cone (ppm or µg/g)	Tot. Mass in Soil (µg per cm ²)	TCE Carbon Cone (ppm or µg/g)	Tot. Mass in Filter (µg per cm ²)	(%)	P/F
C1-3	3	2	28.51	3041.45	1197.87	1521.2949	0.500187378	
C1-7	3	2	45.74	4879.54	893.66	1134.9482	0.232593277	
C2-2	3	2	23.55	2512.31	2143.32	2722.2704	1.083572648	
C2-6	3	2	32.56	3473.50	3560.11	4521.3397	1.30166682	
C3-3	3	2	32.20	3435.10	3283.16	4169.6132	1.213825857	
C3-7	3	2	34.65	3696.46	3285.32	4172.3564	1.128743825	
Zone Ave. (3')							0.910098301	
C1-3	4	2	28.66	3057.45	449.72	571.1444	0.186804167	
C1-7	4	2	50.41	5377.74	755.45	959.4215	0.178406078	
C2-2	4	2	15.56	1659.94	918.13	1166.0251	0.702450149	
C2-6	4	2	48.94	5220.92	3173.98	4030.9546	0.77207745	
C3-3	4	2	38.07	4061.31	1880.92	2388.7684	0.58817682	
C3-7	4	2	61.91	6604.56	2817.04	3577.6408	0.541692528	
Zone Ave. (4')							0.494934532	
C1-3	5	2	44.03	4697.12	522.21	663.5877	0.141275441	
C1-7	5	2	59.92	6392.27	1142.20	1450.594	0.226929401	
C2-2	5	2	15.88	1694.08	1465.91	1861.7057	1.098947925	
C2-6	5	2	57.42	6125.57	2458.20	3121.914	0.509652816	
C3-3	5	2	54.92	5858.87	3524.42	4476.0134	0.763972131	
C3-7	5	2	61.78	6590.69	3497.66	4442.0282	0.673985304	
Zone Ave. (5')							0.569127169	
C1-3	6	2	48.38	5161.18	587.11	745.6297	0.144468842	
C1-7	6	2	77.84	8303.97	1858.20	2359.914	0.284191056	
C2-2	6	2	25.67	2738.48	2208.46	2804.7442	1.024197438	
C2-6	6	2	92.33	9956.44	3083.66	3916.2482	0.393338201	
C3-3	6	2	61.26	6535.22	3102.33	3939.9591	0.602880867	
C3-7	6	2	103.05	10993.37	6173.52	7840.3704	0.713190805	
Zone Ave. (6')							0.527044535	
C1-3	7	2	32.98	3518.31	716.33	909.7391	0.258572752	
C1-7	7	2	48.46	5169.71	2359.25	2996.2475	0.579577481	
C2-2	7	2	16.05	1712.21	1385.65	1759.7755	1.027780179	
C2-6	7	2	59.81	6380.53	2542.06	3228.4162	0.505979315	
C3-3	7	2	52.14	5562.30	2231.32	2833.7764	0.509461266	
C3-7	7	2	67.02	7149.69	4335.45	5506.0215	0.770106326	
Zone Ave. (7')							0.608579553	

*=kriged node values used

Total Mass in Soil (1cm² clay) = 21"*(2.54cm")*2g/cm³*(TCE pre-test-kriged conc.)

Total Mass in Filter (1cm² carbon) = 1"*(2.54cm")*0.5g/cm³*(TCE post-test-carbon conc.)

Table 1
Lasagna TCE Evaluation: Pre-test Kriged Values versus Post-test Carbon Data

Sample Name	Depth (feet)	No. of Samp.*	PRE-TEST DATA		POST-TEST DATA		Mass Recovered	
			TCE Kriged Cone (ppm or µg/g)	Tot. Mass in Soil (µg per cm ²)	TCE Carbon Cone (ppm or µg/g)	Tot. Mass in Filter (µg per cm ²)	(%)	P/F
C1-3	8	2	47.76	5095.04	341.27	433.4129	0.085065652	
C1-7	8	2	58.43	6233.31	1903.31	2417.2037	0.387788141	
C2-2	8	2	28.93	3086.25	1008.58	1280.8966	0.415033325	
C2-6	8	2	73.53	7844.18	3056.81	3882.1487	0.494908161	
C3-3	8	2	51.46	5489.75	2402.65	3051.3655	0.555829592	
C3-7	8	2	83.31	8887.51	4250.99	5397.7573	0.60734191	
Zone Ave. (8')							0.424327797	
C1-3	9	2	79.92	8525.87	482.70	613.029	0.071902222	
C1-7	9	2	79.85	8518.40	1380.02	1752.6254	0.205745844	
C2-2	9	2	78.05	8326.37	1361.77	1729.4479	0.207707308	
C2-6	9	2	90.62	9667.34	3210.24	4077.0048	0.421729742	
C3-3	9	2	73.82	7875.12	1606.75	2046.5725	0.259116369	
C3-7	9	2	93.73	9999.12	4487.62	5699.2774	0.569977898	
Zone Ave. (9')							0.289363231	
C1-3	10	2	128.13	13668.91	1534.28	1948.5356	0.142552376	
C1-7	10	2	116.97	12478.36	1868.82	2373.4014	0.190201389	
C2-2	10	2	132.13	14095.63	1797.78	2283.1806	0.161977904	
C2-6	10	2	138.73	14799.72	6729.07	8545.9189	0.577437877	
C3-3	10	2	121.56	12968.02	1462.72	1857.6544	0.143248885	
C3-7	10	2	137.50	14668.50	7490.67	9513.1509	0.648542857	
Zone Ave. (10')							0.310660215	
C1-3	11	2	153.41	16365.78	1477.10	1875.917	0.114624356	
C1-7	11	2	134.40	14337.79	1823.71	2316.1117	0.161538961	
C2-2	11	2	253.73	27067.92	2672.35	3393.8845	0.125384015	
C2-6	11	2	145.00	15468.60	2957.16	3755.5932	0.242788177	
C3-3	11	2	215.74	23015.14	4753.58	6037.0466	0.26230762	
C3-7	11	2	143.56	15314.98	9159.32	11632.3364	0.759539771	
Zone Ave. (11')							0.27769715	
C1-3	12	2	167.33	17850.76	1925.25	2445.0675	0.13697274	
C1-7	12	2	136.60	14572.49	1988.63	2525.5601	0.173310128	
C2-2	12	2	253.95	27091.39	4658.41	5916.1807	0.218378632	
C2-6	12	2	147.98	15786.51	3675.22	4667.5294	0.295665692	
C3-3	12	2	166.01	17709.95	2637.47	3349.5869	0.189135876	
C3-7	12	2	136.60	14572.49	11511.38	14619.4526	1.003222689	
Zone Ave. (12')							0.336114293	

*=kriged node values used

Total Mass in Soil (1cm² clay) = 21"*(2.54cm/in)*2g/cm³*(TCE pre-test-kriged conc.)

Total Mass in Filter (1cm² carbon) = 1"*(2.54cm/in)*0.5g/cm³*(TCE post-test-carbon conc.)

Table 1
Lasagna TCE Evaluation: Pre-test Kriged Values versus Post-test Carbon Data

Sample Name	Depth (feet)	No. of Samp.*	PRE-TEST DATA		POST-TEST DATA		Mass Recovered	
			TCE Kriged Cone (ppm or µg/g)	Tot. Mass in Soil (µg per cm ²)	TCE Carbon Cone (ppm or µg/g)	Tot. Mass in Filter (µg per cm ²)	(%)	P/F
C1-3	13	2	164.62	17561.66	3259.26	4139.2602	0.235698687	
C1-7	13	2	135.02	14403.93	3283.58	4170.1466	0.289514501	
C2-2	13	2	382.56	40811.50	9813.12	12462.6624	0.475467964	
C2-6	13	2	144.35	15399.26	5765.24	7321.3548	0.529271936	
C3-3	13	2	224.67	23967.80	9988.57	12685.4839	1.01668293	
C3-7	13	2	129.24	13787.32	11037.27	14017.3329	1.016682694	
Zone Ave. (13')							0.593886452	
C1-3	14	2	159.88	17056.00	4494.82	5708.4214	0.334686996	
C1-7	14	2	128.70	17056.00	4494.82	5708.4214	0.403441806	
C2-2	14	2	218.70	23330.92	7411.55	9412.6685	0.39976722	
C2-6	14	2	137.43	14661.03	4614.96	5860.9992	0.646735323	
C3-3	14	2	172.14	18363.90	9351.64	11876.5828	1.137628706	
C3-7	14	2	128.70	13729.72	12298.68	15619.3236	1.137629038	
Zone Ave. (14')							0.676648181	
C1-3	15	2	154.52	16484.19	5038.47	6398.8569	0.147852123	
C1-7	15	2	147.60	15745.97	1833.13	2328.0751	1.038364766	
C2-2	15	2	214.47	22879.66	18706.64	23757.4328	0.827295261	
C2-6	15	2	147.97	15785.44	10282.85	13059.2197	1.389998998	
C3-3	15	2	133.77	14270.58	15618.97	19836.0919	0.637571683	
C3-7	15	2	147.60	15745.97	7904.87	10039.1846	0.637571783	
Zone Ave. (15')							0.779775769	

*=kriged node values used

Total Mass in Soil (1cm² clay) = 21*(2.54cm/1")*2g/cm³*(TCE pre-test-kriged conc.)

Total Mass in Filter (1cm² carbon) = 1*(2.54cm/1")*0.5g/cm³*(TCE post-test-carbon conc.)

Table 2
Lasagna TCE Evaluation: Pre-test Kriged Values versus Post-test Carbon Data

			PRE-TEST DATA					POST-TEST DATA					
Sample Name	Depth (feet)	No. of Samp.*	TCE Kriged Conc (ppm or µg/g)	Kriged Std. Dev./Error Weight (SD)	* (SD)	Corrected Kriged TCE Conc (ppm or µg/g)	Tot. Mass in Soil (µg per cm³)	TCE Carbon Conc (ppm or µg/g)	Tot. Mass in Filler (µg per cm³)	Mass Recovered		Table 1 Mass Recovered	
										(%)	P/F	(%)	P/F
C1-3	3	2	28.51	6.41	*2 (overestimated)	15.69	1673.81	1197.87	1521.2949	0.908881908	P	0.500187378	F
C1-7	3	2	45.74	7.78	*1 (overestimated)	37.96	4049.57	893.66	1134.9482	0.280263686	F	0.232593277	F
C2-2	3	2	23.55	4.09	*1 (underestimated)	27.64	2948.64	2143.52	2722.2704	0.923230653	P	1.083572648	P
C2-6	3	2	32.56	1.62	*1 (underestimated)	34.18	3646.20	3560.11	4521.3397	1.239972554	P	1.30166682	P
C3-3	3	2	32.20	4.06	*1 (underestimated)	36.26	3868.22	3283.16	4169.6132	1.077916109	P	1.213825857	P
C3-7	3	2	34.65	4.66	*1 (underestimated)	39.31	4193.59	3285.32	4172.3564	0.994936464	P	1.128743825	P
Zone Ave. (3')										0.904200229		0.910098301	
C1-3	6	2	48.38	25.90	*1 (overestimated)	22.48	2398.17	587.11	745.6297	0.310916582	F	0.144468842	F
C1-7	6	2	77.84	33.10	*1 (overestimated)	44.74	4772.86	1858.20	2359.914	0.49444409	F	0.284191056	F
C2-2	6	2	25.67	28.30	*0(29.2)	25.67	2738.48	2208.46	2804.7442	1.024199084	F	1.024197438	P
C2-6	6	2	92.33	19.10	*1 (overestimated)	74.23	7918.86	3083.66	3916.2482	0.494547192	F	0.393338201	F
C3-3	6	2	61.26	26.40	*1 (underestimated)	87.66	9351.57	3102.33	3939.9591	0.421315309	F	0.602880867	P
C3-7	6	2	103.05	28.40	*1 (overestimated)	74.65	7963.66	6173.52	7840.3704	0.984518228	P	0.713190805	P
Zone Ave. (6')										0.621656747		0.527044535	
C1-3	10	2	128.13	26.40	*1 (overestimated)	101.73	10852.56	1534.28	1948.5356	0.179546231	F	0.142552376	F
C1-7	10	2	116.97	34.20	*2 (overestimated)	48.57	5181.45	1868.82	2373.4014	0.45805759	F	0.190201389	F
C2-2	10	2	132.13	32.10	*2 (overestimated)	67.93	7246.77	1797.78	2283.1806	0.315061723	F	0.161977904	F
C2-6	10	2	138.73	18.90	*1 (overestimated)	119.83	12783.46	6729.07	8545.9189	0.668513529	F	0.577437877	F
C3-3	10	2	121.56	32.00	*2 (overestimated)	57.56	6140.50	1462.72	1857.6544	0.302524902	F	0.143248885	F
C3-7	10	2	137.50	30.40	*2 (overestimated)	76.60	8182.36	7490.67	9513.1509	1.162642019	P	0.648542857	P
Zone Ave. (10')										0.514390999		0.310660215	
C1-3	15	2	154.52	29.60	*2 (overestimated)	95.32	10168.74	5038.47	6398.8569	0.62926758	P	0.147852123	F
C1-7	15	2	147.60	51.60	*2 (overestimated)	44.40	4736.59	1833.13	2328.0751	0.491508473	F	1.038364766	F
C2-2	15	2	214.47	36.70	*0(37.4)	214.47	22879.66	18706.64	23757.4328	1.038364784	P	0.827295261	P
C2-6	15	2	147.97	20.90	*1 (underestimated)	168.87	18015.05	10282.85	13059.2197	0.724906028	P	1.389998998	P
C3-3	15	2	133.77	33.90	*1 (underestimated)	167.67	17887.04	15618.97	19836.0919	1.108964747	P	0.637571683	P
C3-7	15	2	147.60	34.60	*1 (overestimated)	113.00	12054.84	7904.87	10039.1846	0.832792878	P	0.637571783	P
Zone Ave. (15')										0.804300748		0.779775769	

Appendix H. Comparison of ORNL-Grand Junction and ESC Extract Analysis for Post-*Lasagna*TM Soil Samples

Comparison of ORNL and ESC Analysis Results

Extract ID	ORNL Soil TCE (ug/gram)	ORNL-ESC Soil TCE (%)	Average Soil TCE (ug/gram)	ESC-Avrge Soil TCE (ug/gram)	ESC-Avrge Soil TCE (%)
LP-05 #318	0.18	-2.40%	0.18	0.00	1.20%
LP-05 #320	0.26	-0.77%	0.26	0.00	0.39%
LP-05 #321	0.15	-2.47%	0.15	0.00	1.23%
LP-05 #322	0.04	-28.19%	0.05	0.01	14.09%
LP-05 #324	0.03	-54.13%	0.04	0.01	27.06%
LP-05 #325	0.06	-23.70%	0.06	0.01	11.85%
LP-05 #328	0.00	-165.24%	0.02	0.02	82.62%
LP-05 #329	0.03	-49.04%	0.04	0.01	24.52%
LP-05 #330	0.14	-8.75%	0.14	0.01	4.37%
LP-05 #332	0.17	-2.66%	0.17	0.00	1.33%
LP-05 #333	0.12	-2.83%	0.12	0.00	1.42%
LP-05 #334	0.20	-69.52%	0.30	0.10	34.76%
LP-05 #336	0.29	-43.19%	0.37	0.08	21.59%
LP-05 #337	53.45	-13.63%	57.36	3.91	6.82%
LP-05 #338	73.84	-24.57%	84.18	10.34	12.28%
LP-06 #298	1.22	-1.30%	1.23	0.01	0.65%
LP-06 #299	1.42	-3.98%	1.45	0.03	1.99%
LP-06 #300	0.46	-10.97%	0.48	0.03	5.48%
LP-06 #302	0.13	-3.33%	0.14	0.00	1.66%
LP-06 #303	0.19	0.00%	0.19	0.00	0.00%
LP-06 #304	0.21	-0.15%	0.21	0.00	0.07%
LP-06 #306	0.21	-1.98%	0.21	0.00	0.99%
LP-06 #307	0.32	-5.22%	0.33	0.01	2.61%
LP-06 #308	0.45	-10.25%	0.47	0.02	5.12%
LP-06 #309	0.43	-7.77%	0.45	0.02	3.89%
LP-06 #312	0.53	37.82%	0.44	-0.08	-18.91%
LP-06 #313	0.35	-14.67%	0.37	0.03	7.33%
LP-06 #314	0.51	-25.63%	0.59	0.08	12.81%
LP-06 #316	56.79	-16.17%	61.78	4.99	8.08%
LP-06 #317	9.04	-26.11%	10.40	1.36	13.06%
LP-07 #276	0.14	-79.33%	0.24	0.09	39.67%
LP-07 #277	0.09	-115.79%	0.21	0.12	57.89%
LP-07 #279	0.10	-5.10%	0.11	0.00	2.55%
LP-07 #280	0.08	-21.23%	0.09	0.01	10.61%
LP-07 #281	0.10	-4.47%	0.10	0.00	2.24%
LP-07 #283	0.07	-14.89%	0.08	0.01	7.44%
LP-07 #284	0.10	-7.09%	0.10	0.00	3.55%
LP-07 #285	0.19	-2.06%	0.19	0.00	1.03%
LP-07 #287	0.44	-9.90%	0.47	0.02	4.95%

Comparison of ORNL and ESC Analysis Results

Extract ID	ORNL Soil TCE (ug/gram)	ORNL-ESC Soil TCE (%)	Average Soil TCE (ug/gram)	ESC-Avrge Soil TCE (ug/gram)	ESC-Avrge Soil TCE (%)
LP-07 #288	0.06	-138.50%	0.18	0.12	69.25%
LP-07 #289	2.71	-1.35%	2.73	0.02	0.67%
LP-07 #290	2.03	-0.66%	2.04	0.01	0.33%
LP-07 #293	8.62	-28.59%	10.05	1.44	14.30%
LP-07 #294	41.25	-8.44%	43.07	1.82	4.22%
LP-07 #295	19.60	-2.57%	19.86	0.26	1.29%
LP-08 #353	2.03	-0.18%	2.03	0.00	0.09%
LP-08 #354	1.14	-7.41%	1.18	0.04	3.71%
LP-08 #355	0.32	-1.60%	0.33	0.00	0.80%
LP-08 #358	0.18	-0.41%	0.18	0.00	0.20%
LP-08 #359	0.15	-0.99%	0.15	0.00	0.49%
LP-08 #360	0.28	-0.81%	0.29	0.00	0.40%
LP-08 #363	0.38	-3.78%	0.38	0.01	1.89%
LP-08 #364	0.44	-29.49%	0.51	0.08	14.74%
LP-08 #365	0.42	-30.24%	0.49	0.07	15.12%
LP-08 #366	0.02	-173.45%	0.14	0.12	86.73%
LP-08 #367	17.44	0.17%	17.43	-0.02	-0.09%
LP-08 #368	11.59	-2.02%	11.71	0.12	1.01%
LP-08 #369	35.83	-1.88%	36.17	0.34	0.94%
LP-08 #372	48.12	-8.66%	50.30	2.18	4.33%
LP-09 #341	0.40	-8.69%	0.42	0.02	4.34%
LP-09 #342	0.11	-6.11%	0.12	0.00	3.05%
LP-09 #343	0.23	-4.67%	0.24	0.01	2.34%
LP-09 #344	0.26	-2.66%	0.27	0.00	1.33%
LP-09 #345	0.25	-2.30%	0.25	0.00	1.15%
LP-09 #346	0.53	-9.63%	0.56	0.03	4.82%
LP-09 #347	3.14	-7.64%	3.27	0.12	3.82%
LP-09 #348	13.69	-3.42%	13.92	0.24	1.71%
LP-09 #349	15.23	-20.22%	16.94	1.71	10.11%
LP-09 #350	29.22	-4.21%	29.84	0.63	2.10%
LP-09 #351	32.83	-2.43%	33.23	0.40	1.21%
LP-09 #352	37.77	-12.04%	40.19	2.42	6.02%
LP-11 #220	0.55	-20.29%	0.61	0.06	10.14%
LP-11 #221	0.47	-7.95%	0.49	0.02	3.97%
LP-11 #222	0.58	-17.45%	0.63	0.06	8.73%
LP-11 #223	0.54	-13.97%	0.58	0.04	6.98%
LP-11 #224	0.40	-8.00%	0.42	0.02	4.00%
LP-11 #225	0.44	-5.96%	0.45	0.01	2.98%
LP-11 #229	2.22	-3.16%	2.26	0.04	1.58%

Comparison of ORNL and ESC Analysis Results

Extract ID	ORNL Soil TCE (ug/gram)	ORNL-ESC Soil TCE (%)	Average Soil TCE (ug/gram)	ESC-Avrge Soil TCE (ug/gram)	ESC-Avrge Soil TCE (%)
LP-11 #230	1.84	-1.40%	1.85	0.01	0.70%
LP-11 #231	0.40	-27.23%	0.46	0.06	13.62%
LP-11 #232	1.04	-8.95%	1.09	0.05	4.48%
LP-12 #236	0.22	-3.23%	0.22	0.00	1.61%
LP-12 #237	0.11	-8.00%	0.11	0.00	4.00%
LP-12 #238	0.00	-	0.00	0.00	-
LP-12 #239	0.00	-	0.00	0.00	-
LP-12 #241	0.00	-	0.01	0.01	-
LP-12 #242	0.00	-	0.01	0.01	-
LP-12 #245	0.03	-52.03%	0.03	0.01	26.02%
LP-12 #246	0.29	-4.17%	0.30	0.01	2.09%
LP-12 #247	0.49	-27.98%	0.57	0.08	13.99%
LP-12 #248	1.10	-3.60%	1.12	0.02	1.80%
LP-12 #249	1.64	-1.84%	1.66	0.02	0.92%
LP-12 #251	1.57	-3.89%	1.60	0.03	1.95%
LP-13 #256	2.29	-2.64%	2.32	0.03	1.32%
LP-13 #257	2.56	-3.52%	2.61	0.05	1.76%
LP-13 #260	2.92	-2.52%	2.96	0.04	1.26%
LP-13 #261	0.67	-30.82%	0.79	0.12	15.41%
LP-13 #262	0.04	-43.63%	0.05	0.01	21.81%
LP-13 #264	0.21	-1.54%	0.21	0.00	0.77%
LP-13 #265	0.25	-51.24%	0.33	0.09	25.62%
LP-13 #266	0.25	-53.64%	0.34	0.09	26.82%
LP-13 #268	0.27	200.00%	0.14	-0.14	-100.00%
LP-13 #269	0.44	-29.99%	0.52	0.08	15.00%
LP-13 #270	0.06	-137.19%	0.19	0.13	68.59%
LP-13 #271	0.11	-122.89%	0.28	0.17	61.44%
LP-13 #274	0.23	-0.49%	0.23	0.00	0.24%
LP-14 #387	24.57	-3.61%	25.03	0.45	1.80%
LP-14 #388	33.78	-5.08%	34.66	0.88	2.54%
LP-14 #389	68.54	-14.96%	74.08	5.54	7.48%
LP-14 #390	89.22	-28.58%	104.10	14.87	14.29%
LP-14 #391	80.45	-18.08%	88.44	7.99	9.04%
LP-14 #392	88.11	-29.47%	103.33	15.22	14.73%
LP-14 #393	96.74	-29.42%	113.42	16.68	14.71%
LP-14 #394	78.02	-21.74%	87.53	9.51	10.87%
LP-14 #395	91.23	-27.31%	105.65	14.42	13.65%
LP-14 #396	101.69	-29.16%	119.05	17.36	14.58%
LP-14 #397	56.38	-20.22%	62.73	6.34	10.11%

Comparison of ORNL and ESC Analysis Results

Extract ID	ORNL Soil TCE (ug/gram)	ORNL-ESC Soil TCE (%)	Average Soil TCE (ug/gram)	ESC-Avrge Soil TCE (ug/gram)	ESC-Avrge Soil TCE (%)
LP-15 #374	10.63	-3.99%	10.85	0.22	1.99%
LP-15 #377	43.04	-7.74%	44.77	1.73	3.87%
LP-15 #378	62.65	-17.62%	68.71	6.05	8.81%
LP-15 #379	74.26	-29.58%	87.15	12.89	14.79%
LP-15 #380	86.68	-28.46%	101.06	14.38	14.23%
LP-15 #381	69.20	-28.53%	80.72	11.52	14.27%
LP-15 #382	70.89	-26.44%	81.69	10.80	13.22%
LP-15 #383	68.45	-17.13%	74.87	6.41	8.56%
LP-15 #384	73.40	-9.20%	76.94	3.54	4.60%
LP-15 #385	109.46	-8.09%	114.08	4.61	4.04%
LP-15 #386	36.97	-7.70%	38.45	1.48	3.85%
LP-16 #398	2.56	-65.74%	3.82	1.26	32.87%
LP-16 #399	13.73	-3.33%	13.96	0.23	1.67%
LP-16 #400	4.35	-28.50%	5.07	0.72	14.25%
LP-16 #401	6.53	-13.90%	7.02	0.49	6.95%
LP-16 #402	11.43	-4.19%	11.67	0.24	2.09%
LP-16 #403	16.21	-2.26%	16.39	0.18	1.13%
LP-16 #404	50.81	-3.91%	51.82	1.01	1.96%
LP-16 #405	-	-	-	-	-
LP-16 #406	33.59	-8.72%	35.12	1.53	4.36%
LP-16 #407	30.26	0.56%	30.18	-0.08	-0.28%
LP-16 #408	30.68	-3.98%	31.30	0.62	1.99%
LP-16 #409	19.97	-0.65%	20.03	0.07	0.33%
LP-16 #410	14.24	-2.83%	14.45	0.20	1.41%
LP-16 #411	9.30	-12.80%	9.94	0.64	6.40%
LP-16 #412	25.84	-1.86%	26.09	0.24	0.93%

Average = -17.60%
Std. Dev. = 36.25%
Number = 138

Average = 8.80%
Std. Dev. = 18.13%
Number = 138

Material Balance

Total TCE in Soil and Carbon Volumes (ug/cm²x21" or ug/cm²x1")

Depth	Soil	Carbon	Soil	Carbon	Soil	Carbon	Soil	Carbon	Soil	Carbon	Soil	Carbon	Soil	Carbon
			L16	Anode-5	L5	C1-7	L11	C2-6	L13	C3-7	L12	C4-7	L12	Cathode-5
4			1,131	5,863	2,166	571	437	1,188	4,086	2,389	1,482	2,327	1,482	4,292
5			1,846	1,289	5,313	884	1,878	1,862	6,113	4,476	1,280	2,538	1,280	13,176
6			2,326	854	5,174	746	2,027	2,805	6,913	3,940	2,166	2,980	2,166	8,901
7			2,752	1,193	3,499	910	2,368	1,760	9,783	2,834	2,646	2,619	2,646	8,060
8			4,129	554	2,528	433	3,542	1,281	7,649	3,051	4,257	1,485	4,257	8,931
9			4,897	582	2,432	813	6,774	1,729	9,953	2,041	7,201	1,370	7,201	3,017
10			7,382	1,784	4,822	1,949	9,527	2,283	10,113	1,858	8,204	2,825	8,204	5,988
11			11,596	2,988	7,393	1,876	13,822	3,394	14,669	8,037	8,054	1,735	8,054	5,154
12			11,703	3,833	11,777	2,445	15,394	5,916	14,957	3,350	8,454	2,576	8,454	8,167
13			12,183	6,466	18,072	4,139	15,383	12,463	14,572	12,885	5,750	3,749	5,750	3,084
14			10,412	17,159	12,908	5,708	10,039	9,413	14,498	11,877	5,589	4,207	5,589	9,897
15			8,518	12,685	17,410	6,399	11,148	23,757	13,602	19,836	4,353	6,495	4,353	14,494
Average			6,406	4,601	7,791	2,204	7,703	5,652	10,578	6,198	4,783	2,909	4,783	7,097

Depth	Soil	Carbon	Soil	Carbon	Soil	Carbon	Soil	Carbon	Soil	Carbon	Soil	Carbon	Soil	Carbon
	L15	Control	L15	Anode-2	L5	C1-3	L6	C2-2	L8	C3-3	L7	C4-3	L9	Cathode-2
4	6,571	1,018	6,571	459	2,166	959	523	4,031	1,035	3,578	3,968	5,825	1,482	12,051
5	6,700	2,457	6,700	299	5,313	1,451	2,688	3,122	1,291	4,442	6,835	14,931	4,310	8,304
6	5,110	3,312	5,110	1,088	5,174	2,360	4,353	3,918	320	7,840	7,670	4,906	6,838	7,027
7	4,662	4,006	4,662	1,687	3,499	2,996	5,409	3,228	405	5,506	7,308	3,574	9,110	7,441
8	7,521	3,062	7,521	876	2,528	2,417	2,123	3,882	245	5,399	7,148	1,603	8,470	7,298
9	11,052	4,592	11,052	749	2,432	1,753	4,209	4,077	725	5,099	8,097	3,323	5,046	6,817
10	12,343	4,835	12,343	1,273	4,822	2,373	6,230	8,548	10,209	9,513	7,928	3,378	4,534	6,476
11	14,423	5,189	14,423	1,311	7,393	2,316	14,733	3,756	14,804	11,832	11,852	7,817	5,473	9,542
12	18,114	8,299	18,114	1,922	11,777	2,526	13,964	4,608	18,082	14,819	18,850	14,201	12,080	5,295
13	18,546	5,900	18,546	2,375	18,072	4,170	17,848	7,322	29,732	14,017	17,133	14,939	11,137	3,227
14	20,995	7,718	20,995	2,182	12,908	3,947	16,130	5,881	39,322	15,819	14,797	8,743	8,278	4,226
15	17,410	8,028	17,410	1,687	17,410	2,328	16,895	13,059	25,315	10,039	18,712	13,559	8,534	3,471
Average	11,787	4,869	11,787	1,322	7,791	2,486	8,760	5,456	11,774	8,992	10,841	8,058	7,105	6,748

Appendix I. TCE Concentrations in Soil Borings and Carbon Cassettes used in Material Balance Calculations

Water Summary

TCE, cis-1,2-DCE, and Vinyl Chloride Concentrations in Water from Carbon Cassettes (ug/mL)

Cmpd	Control	Anode-2	Anode-5	C1-3	C1-7	C2-2	C2-6	C3-3	C3-7	C4-3	C4-7 (2 Pores)	C4-7 (1 Pore)	Cathode-2	Cathode-5
TCE-ESC	0.07	0.01	0.00	0.00	0.01	0.11	0.01	0.33	0.01	0.02	0.21	0.00	5.16	0.05
TCE-PGDP	0.17	0.02	0.00	0.02	0.06	0.32	0.02	0.66	0.03	0.17		0.02	1.60	0.24
cis-1,2-DCE- PGDP	0.17	0.02	0.00	0.17	0.13	0.79	0.10	0.11	0.00	0.76		0.09	0.71	0.16
Vinyl Chloride- PGDP	0.00	0.00	0.00	0.01	0.00	0.05	0.00	0.08	0.00	0.09		0.00	0.12	0.04

Compound Concentrations in Water from Carbon Cassettes

Appendix K. Chlorinated Compound Concentrations in Soil from Post-Experiment Soil Borings

During the post-experiment soil sampling, selected boring samples were analyzed for a wide range of chlorinated compounds. The following table summarizes the concentrations of vinyl chloride, *cis*-1,2-dichloroethylene, and trichloroethylene that were detected in these analyses. Note that the location of LP16 is shown in Figure F-3. LP17 and LP18 are not shown in the figure. LP17 was located 5 feet south of L6 and LP18 was located 5 feet south of L9. All results are for the concentrations of these compounds which were detected in water that had been added to core samples. Approximately 20 mLs deionized water was added to several grams of clay from various depths.

Well ID	Depth (ft.)	Water Concentration (µg/L or ppb)		
		vinyl chloride	<i>cis</i> -1,2-dichloroethylene	trichloroethylene
LP16	15.5	<10	<5	<1
LP17	10	"	"	60
	10 (duplicate)	"	"	21
	6	"	"	12
	6 (duplicate)	"	"	13
LP18	10	12	80	6,300
	10 (duplicate)	15	83	5,600
	16.5	<10	18	22,000
	16.5 (duplicate)	"	14	25,000
	6	"	190	6,700
	6 (duplicate)	"	160	4,400
LP8	10.5	"	5	54
	10.5 (duplicate)	"	5	51
	16.5	"	140	26,000
	6.5	"	18	96
	6.5 (duplicate)	"	10	71

Appendix L. Chlorinated Compound Analysis Method Us LMES-

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PADUCAH GASEOUS DIFFUSION PLANT

Technical Services Division
Analytical Department

Analysis of Volatile Organic Compounds
by Capillary Gas Chromatography/Mass Spectrometry
AL-OP-4
Rev. 0

Prepared by: John Wayne Shadrick

Approved by: S. K. Holshouser 11-7-93
S. K. Holshouser, Date
AL Department Manager

Effective Date: 1/21/93

Date	5-1-96	Ed	pages	34
From	John Shadrick			
To	Joy Claver			
Co.				
Phone	6441			
Fax	5064			

In-Hand Procedure: Yes <u> </u> No <u>X</u>	<u> </u> Safety System
Total: <u> </u>	<u> </u> Safety-Related
Partial: <u> </u>	<u>X</u> Quality-Related
Appendix: <u> </u>	<u> </u> Nonquality/Nonsafety-Related
(See Applicability Section For Details)	
Redlined <u> </u> Upgraded <u>X</u>	<u> </u> Temporary/Expiration Date <u> </u>

Record of Changes

Change Letter	Affected Page(s)	Effective Date	Change Approved by
A (IAC)	5, 8, 9, 10, 11, 12, 13, 15, 17, 18, 19, 20, 21, 23, 24, 30, 31, 32, 33, & 34	6/8/95	JKH/19423
B	8, 11, 12, 18, 19, 20, 24, 33	10/23/95	SKH/19423

Next 3-year review required no later than: 1/7/96
Date

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1.0 SCOPE

This procedure is applicable to the analysis of volatile organic compounds in ground and surface water samples, soil samples, and TCLP extracts.

2.0 REFERENCED DOCUMENTS

2.1 Use References

✓ A. *Calibration Procedure Manual*, CP-QTS-1112, "Preparation of the Hewlett Packard 5970 MSD for Analysis"

2.2 Source References

A. Federal Register EPA Test Method, EPA-624, "Purgeables," S-927, Part 136, Appendix A, Method 624, December 11, 1992

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B. SW-846. Test Methods for Evaluating Solid Waste, RCRA 8260, "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique," Volume 1B, September 1994

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C. Basic Safety Information, BSI-2, "Handling Flammable and Combustible Liquids"

D. Basic Safety Information, BSI-8, "Handling Glassware"

E. Basic Safety Information, BSI-5, "Hidden or Unsuspected Hazards in the Chemical Laboratory"

F. *Technical Services Job Hazard Analysis Manual*, L-150, "Handling Organic Chemicals"

G. *Technical Services Job Hazard Analysis Manual*, L-162, "Handling and Transporting Compressed Gas Cylinders Using a Cylinder Cart"

H. *Technical Services Division Standard Operating Procedure Manual*, TSD-103, "Quality Assurance Program"

I. *Technical Services Division Standard Operating Procedure Manual*, CP3-TS-QL1071, "Administration of Consumable Standards"

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2.2 Source References (Continued)

K. *Technical Services Division Standard Operating Procedure Manual, TSD-322, "Temperature Control For Sample Storage"*

3.0 RESPONSIBILITIES

- 3.1 Personnel in the GC/MS lab are responsible for analyzing samples in accordance with this procedure.
- 3.2 The Organic & Process Analysis Section supervisor or his/her appointed representative is responsible for reviewing and approving data resulting from this procedure.

4.0 TERMINOLOGY

4.1 Acronyms

- A. AnaLIS - Analytical Laboratory Information System
- B. GC - Gas Chromatograph
- C. GC/MS - Gas Chromatograph/Mass Spectrometer
- D. MSD - Mass Selective Detector
- E. TCLP - Toxicity Characteristic Leachate Procedure
- F. VOC - Volatile Organic Compound

4.2 Definitions

- A. System Performance Check Compounds (SPCC) - Chloromethane; 1,1-dichloroethane; Bromoform; Chlorobenzene; and 1,1,2,2-Tetrachloroethane
- B. Calibration Check Compounds (CCC) - 1,1-dichloroethene; Chloroform; 1,2-dichloropropane; Toluene; Ethylbenzene; and Vinyl Chloride.

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5.0 SUMMARY OF TEST METHOD

The volatile components of the sample are removed from the water matrix by bubbling helium through the sample at ambient temperature. These compounds are trapped in a tube containing suitable sorbent materials. When purging is completed, the tube is heated and backflushed with helium to desorb the compounds onto a large bore capillary column. The Gas Chromatograph (GC) is temperature programmed to separate the method analytes before they enter the mass selective detector (MSD). The masses of the analytes are measured by the MSD. Data results are stored by file. Data is retrieved and analyzed by the Data System.

6.0 SIGNIFICANCE AND USE

- 6.1 This procedure can be utilized to quantify most volatile organic compounds that have boiling points below 200° C and are insoluble or only slightly soluble in water.
- 6.2 This procedure can be used to determine VOCs in a variety of matrices. The primary applications of this procedure are for surface and ground water samples, soil samples, and TCLP extracts.

7.0 INTERFERENCES

- 7.1 Samples can be easily contaminated by the diffusion of volatile organics through the air. The samples are exposed to air while being transferred from the sample container to the sparging vessel. To minimize this effect, the use of large quantities of volatile organics is not allowed in the GC/MS lab. However, the solvents methylene chloride and carbon disulfide are routinely used in the laboratory building. Consequentially, these compounds are occasionally detected in sequence runs (both sample and blank runs).

NOTE: If contamination from volatile organics through the air is detected in both sample and blank runs, then ensure that system is clean before proceeding to run backup samples.

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- 7.2 Volatile organics (particularly fluorocarbons and methylene chloride) can also come into contact with the samples by diffusion through the septum seal. To check for this type of contamination, a trip blank prepared from organic-free reagent water should be carried through the entire sample collecting and handling process.
- 7.3 When high concentrations of organic compounds are present, the sample sparger position or the sorbent trap can be contaminated. A low-level sample can be contaminated by carry-over if it is analyzed immediately after a high-level sample. Carry-over problems are assessed by the analysis of reagent water blanks. For example, whenever a known concentrated sample is analyzed, it may be followed by the analysis of reagent water to check for cross-contamination. Samples analyzed after a concentrated sample will be closely inspected and if carry-over is suspected then the sample will be reanalyzed.
- 7.4 To minimize carry-over effects, the purge-and-trap (P&T) unit is taken through a bake out cycle. This cycle includes purging the sparger and baking the sorbent tube after each run.
- 7.5 Additional cleanup of sparger positions known previously to contain significant concentrations of volatile organics (eq. positions used for calibration standards) will be rinsed with reagent water and purged for at least 2 minutes. The P&T must be baked out following the cleanup procedure. Very high concentrations (greater than several hundred ug/l) of many volatile organics in a sample may cause contamination of a P&T sparger position that will require special cleanup procedures (see Appendix A, "Clean-up of the Purge and Trap Unit").
- 7.6 Impurities present in the purge or carrier gas may contaminate the plumbing and/or the samples. Therefore, only ultra-high purity helium is used for the purge gas and carrier gas. All supply lines and fittings are made of copper.

8.0 APPARATUS

- 8.1 Purge-and-Trap device - Tekmar LSC 2000 P&T coupled with the ALS 2016 Autosampler, a Tekmar Cryofocusing Module and a Tekmar No. 8 trap, Restek K trap, or other appropriate trap (parameter examples are listed in Appendix B, "Tekmar Parameters").

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8.0 APPARATUS (Continued)

NOTE

Traps normally last 2 to 3 months when used daily. Some signs of a deteriorating trap are:

1. Uncharacteristic recoveries of surrogates, esp. Toluene- d_8
2. A loss of the response of the internal standards during a 12-hour shift
3. A rise in the baseline in the early portion of the scan

- 8.2 Gas Chromatograph (GC) - Hewlett Packard model 5890 Series 2 with capillary column injection port or equivalent (parameters are listed in Appendix C, "GC Parameters")
- 8.3 GC column- 60 meter by 0.53 mm ID RTX-Volatiles (Restek) or other appropriate column
- 8.4 Mass Spectrometer (MS) - Capable of interfacing with the GC, Hewlett Packard model 5970B or equivalent
- 8.5 Data System - Capable of interfacing with the GC/MS, Hewlett Packard HP 1000 computer with RTE-A Rev. F software or equivalent

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NOTE

In recognition of the rapid advances occurring in chromatography, the analyst is permitted to modify GC columns, GC conditions, purge and trap conditions or detectors to improve separations or lower the cost of the measurements. Each time such modifications to the procedure are made, the analyst is required to meet the QC requirements.

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9.0 REAGENTS AND MATERIALS

NOTES

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Stock solutions are purchased as certified solutions from reputable commercially available sources. In the event that a certified solution is unavailable, stock solutions may be prepared from pure standard materials. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

Now available are also certified standard solutions at the working standard concentrations. These are also appropriate for use.

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9.0 REAGENTS AND MATERIALS (Continued)

NOTES (Continued)

Appropriate dilutions may be considered skill of the craft. Vendors and initial concentrations listed in this procedure may be substituted for others of equivalent or higher quality. Appropriate dilutions to the working level is determined by the analyst. Therefore stock concentrations and vendors in this procedure should be considered examples.

Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10° C to -20° C or lower in amber bottles with Teflon-lined crimp caps.

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- 9.1 Organic-free reagent water - ALL references to water in this procedure refer to water in which an interferant is not observed at the method detection limit of the compounds of interest.
- 9.2 Methanol - Purge & Trap grade or equivalent. Store apart from other solvents.
- 9.3 Standards - Refer to CP-QTS-1112, "Preparation of the Hewlett Packard 5970 MSD for Analysis"
- 9.3.1 Internal Standards (IS)
- 9.3.2 Surrogate Standards
- 9.3.3 Internal and Surrogate Standards (ISS)
- 9.3.4 Calibration standards
- 9.3.5 4-Bromofluorobenzene (BFB) standard
- 9.4 Matrix spiking standards - Prepared from Restek TCLP-VOA Mix, Number 30024, or equivalent, containing the following 11 compounds: Benzene; 2-Butanone; Carbon Tetrachloride; Chlorobenzene; Chloroform; 1,4-dichlorobenzene; 1,2-dichloroethane; 1,1-dichloroethene; Tetrachloroethene; and Vinyl Chloride at 2000 µg/ml in methanol. Also allowable are certified standards purchased at the working level 25 µg/mL.
- 9.4.1 Prepare 25.0 ug/ml (high level) working standard by diluting 125.0 µL of stock standard to 10.0 mL using methanol in a volumetric flask.
- 9.4.2 Text deleted.
- 9.5 QA/QC check standard - (QC Standard A and QC Standard B)

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9.0 REAGENTS AND MATERIALS (Continued)

NOTE

The QA/QC check standards are not prepared from the same stock standard as used for the calibration standards (refer to CP-QTS-1112).

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- 9.5.1 Prepare 100.0 ug/mL QC Standard A working standard by diluting 400.0 μ L stock solution (Accustandard's GC/MS for Volatile Organics Catalog No. M-8240A-X12.5, 2.50 mg/ml in methanol, or equivalent) with methanol to 10.0 mL in a volumetric flask.
- 9.5.2 Prepare 100.0 ug/mL QC Standard B working standard by diluting 500.0 μ L stock solution (Restek's VOA Calibration Mix 5 Catalog No. 30010, 2000.0 μ L/mL in methanol, or equivalent) with methanol to 10.0 mL in a volumetric flask.
- 9.5.2a Also acceptable is the purchase of certified standards as one or more mixes at the working concentration 100 μ g/mL.
- 9.5.3 Prepare 10 ppb Laboratory Concentrated Spike (LCS) standard daily just before use by spiking 10 μ L of QC Standard into a 100 mL volumetric flask filled to the mark with reagent water.

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9.6 Helium, purity must be 99.999% or greater

9.7 Liquid nitrogen, L-28 tank

10.0 HAZARDS

10.1 Safety Hazards

- 10.1.1 Many of the compounds used in this procedure have been classified as carcinogenic or are suspected to be carcinogenic. To limit the exposure to these chemicals, always use a diluted stock solution whenever a certified commercial source can be found. The Material Safety Data sheets should be reviewed prior to use of the chemicals.
- 10.1.2 Liquid nitrogen is hazardous due to extreme cold temperatures. Insulated gloves should be worn when handling.
- 10.1.3 Care should be taken when working with the syringes, due to the sharp needles. Basic lab safety should be followed at all times.

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10.2 Technical Hazards

A high concentration of analyte or group of analytes could cause an excessive signal and fault the MS. This would stop the analysis and any remaining analyses in the batch run. When analyzing a sample with no historical data, make large dilutions (ex. 1:10,000) to avoid contaminating the instrument. If the chromatogram shows no peaks, the sample can be re-analyzed at a lower dilution. Should the instrument become contaminated, use the clean-up procedure described in Appendix A, "Cleanup of the Purge and Trap."

11.0 SAMPLING, TEST SPECIMENS, AND TEST UNITS

11.1 All samples shall be analyzed within 14 days from date of collection.

11.2 Samples are stored at 4° C.

12.0 PREPARATION OF APPARATUS

12.1 The capillary column should be conditioned as needed by the following:

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12.1.1 Set GC oven temperature to 160° C.

12.1.2 Leave for approximately 30 minutes before cooling oven.

12.2 Any sparge positions with suspected contamination should be purged with blank water as follows:

12.2.1 Fill sparge position with 5 mL blank water.

12.2.2 Purge sparge position for at least 30 seconds. If a high level sample was analyzed in the sparge vessel from the previous run, purge for at least 2 minutes.

12.2.3 Drain sparge position.

12.2.4 Repeat above steps until all sparge vessels have been purged.

12.3 The trap on the P&T unit should be conditioned each day before use by placing P&T unit in BAKE cycle.

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13.0 CALIBRATION AND STANDARDIZATION

The GC/MS instruments are tuned and calibrated as outlined in CP-QTS-1112, "Preparation of the Hewlett Packard 5970 MSD for Analysis."

14.0 CONDITIONING

All samples and standard solutions must be allowed to warm to ambient (room) temperature before analysis.

15.0 PROCEDURE

15.1 Check tune by analyzing 50 ng BFB.

15.1.1 Spike 5 mL of reagent water with 50 ng BFB (2 μ L of BFB working standard).

15.1.2 Perform P&T analysis:

A. Prepare sequence file. The sequence file contains the necessary information about each sample and the order in which they will be analyzed. As an alternative, BFB may be run as a single sample. In this case, sample order may be ascertained by sample log. Chg.
A

1. From the FMGR prompt on one of the system's terminals type BEDIT and press RETURN key.
2. Enter a name for the sequence file and press ENTER key. If this is a new file, the user will be prompted to press ENTER again to create the file.
3. Fill out the header information (refer to Figure 1, "Header Information"):
 - a. Type the BFB tune file name for the instrument being used to run samples.
 - b. Type at least 2 data cartridges (CRs) to be used for storing the collected data.
 - c. Type 5000 for minimum blocks for CR.
 - d. Start EDIT at sample number 1

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15.0 PROCEDURE (Continued)

- e. Type "Y" for Autostep mode
- f. Type "Y" for Auto File Numbering
- g. Press ENTER key, then press F1 "Sample Forms"

Sequence: DEMOSQ:US Header: 8860930-1701

HEADER INFORMATION

Tune File Namr: APR101

Data File CR's (Enter up to 10):
U1 U2 U3 U4 U5 U6 U7 U8 U9 U10

Minimum blocks for CR check (+blocks or -chunks): 500

Start EDIT at sample number: 1

Use Autostep mode ? Y

Auto File Numbering ? Y

Use standard or alternate logic for bottle numbering (S or A) ? S

Sample Form: 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100 101 102 103 104 105 106 107 108 109 110 111 112 113 114 115 116 117 118 119 120 121 122 123 124 125 126 127 128 129 130 131 132 133 134 135 136 137 138 139 140 141 142 143 144 145 146 147 148 149 150 151 152 153 154 155 156 157 158 159 160 161 162 163 164 165 166 167 168 169 170 171 172 173 174 175 176 177 178 179 180 181 182 183 184 185 186 187 188 189 190 191 192 193 194 195 196 197 198 199 200 201 202 203 204 205 206 207 208 209 210 211 212 213 214 215 216 217 218 219 220 221 222 223 224 225 226 227 228 229 230 231 232 233 234 235 236 237 238 239 240 241 242 243 244 245 246 247 248 249 250 251 252 253 254 255 256 257 258 259 260 261 262 263 264 265 266 267 268 269 270 271 272 273 274 275 276 277 278 279 280 281 282 283 284 285 286 287 288 289 290 291 292 293 294 295 296 297 298 299 300 301 302 303 304 305 306 307 308 309 310 311 312 313 314 315 316 317 318 319 320 321 322 323 324 325 326 327 328 329 330 331 332 333 334 335 336 337 338 339 340 341 342 343 344 345 346 347 348 349 350 351 352 353 354 355 356 357 358 359 360 361 362 363 364 365 366 367 368 369 370 371 372 373 374 375 376 377 378 379 380 381 382 383 384 385 386 387 388 389 390 391 392 393 394 395 396 397 398 399 400 401 402 403 404 405 406 407 408 409 410 411 412 413 414 415 416 417 418 419 420 421 422 423 424 425 426 427 428 429 430 431 432 433 434 435 436 437 438 439 440 441 442 443 444 445 446 447 448 449 450 451 452 453 454 455 456 457 458 459 460 461 462 463 464 465 466 467 468 469 470 471 472 473 474 475 476 477 478 479 480 481 482 483 484 485 486 487 488 489 490 491 492 493 494 495 496 497 498 499 500 501 502 503 504 505 506 507 508 509 510 511 512 513 514 515 516 517 518 519 520 521 522 523 524 525 526 527 528 529 530 531 532 533 534 535 536 537 538 539 540 541 542 543 544 545 546 547 548 549 550 551 552 553 554 555 556 557 558 559 560 561 562 563 564 565 566 567 568 569 570 571 572 573 574 575 576 577 578 579 580 581 582 583 584 585 586 587 588 589 590 591 592 593 594 595 596 597 598 599 600 601 602 603 604 605 606 607 608 609 610 611 612 613 614 615 616 617 618 619 620 621 622 623 624 625 626 627 628 629 630 631 632 633 634 635 636 637 638 639 640 641 642 643 644 645 646 647 648 649 650 651 652 653 654 655 656 657 658 659 660 661 662 663 664 665 666 667 668 669 670 671 672 673 674 675 676 677 678 679 680 681 682 683 684 685 686 687 688 689 690 691 692 693 694 695 696 697 698 699 700 701 702 703 704 705 706 707 708 709 710 711 712 713 714 715 716 717 718 719 720 721 722 723 724 725 726 727 728 729 730 731 732 733 734 735 736 737 738 739 740 741 742 743 744 745 746 747 748 749 750 751 752 753 754 755 756 757 758 759 760 761 762 763 764 765 766 767 768 769 770 771 772 773 774 775 776 777 778 779 780 781 782 783 784 785 786 787 788 789 790 791 792 793 794 795 796 797 798 799 800 801 802 803 804 805 806 807 808 809 810 811 812 813 814 815 816 817 818 819 820 821 822 823 824 825 826 827 828 829 830 831 832 833 834 835 836 837 838 839 840 841 842 843 844 845 846 847 848 849 850 851 852 853 854 855 856 857 858 859 860 861 862 863 864 865 866 867 868 869 870 871 872 873 874 875 876 877 878 879 880 881 882 883 884 885 886 887 888 889 890 891 892 893 894 895 896 897 898 899 900 901 902 903 904 905 906 907 908 909 910 911 912 913 914 915 916 917 918 919 920 921 922 923 924 925 926 927 928 929 930 931 932 933 934 935 936 937 938 939 940 941 942 943 944 945 946 947 948 949 950 951 952 953 954 955 956 957 958 959 960 961 962 963 964 965 966 967 968 969 970 971 972 973 974 975 976 977 978 979 980 981 982 983 984 985 986 987 988 989 990 991 992 993 994 995 996 997 998 999 1000 1001 1002 1003 1004 1005 1006 1007 1008 1009 1010 1011 1012 1013 1014 1015 1016 1017 1018 1019 1020 1021 1022 1023 1024 1025 1026 1027 1028 1029 1030 1031 1032 1033 1034 1035 1036 1037 1038 1039 1040 1041 1042 1043 1044 1045 1046 1047 1048 1049 1050 1051 1052 1053 1054 1055 1056 1057 1058 1059 1060 1061 1062 1063 1064 1065 1066 1067 1068 1069 1070 1071 1072 1073 1074 1075 1076 1077 1078 1079 1080 1081 1082 1083 1084 1085 1086 1087 1088 1089 1090 1091 1092 1093 1094 1095 1096 1097 1098 1099 1100 1101 1102 1103 1104 1105 1106 1107 1108 1109 1110 1111 1112 1113 1114 1115 1116 1117 1118 1119 1120 1121 1122 1123 1124 1125 1126 1127 1128 1129 1130 1131 1132 1133 1134 1135 1136 1137 1138 1139 1140 1141 1142 1143 1144 1145 1146 1147 1148 1149 1150 1151 1152 1153 1154 1155 1156 1157 1158 1159 1160 1161 1162 1163 1164 1165 1166 1167 1168 1169 1170 1171 1172 1173 1174 1175 1176 1177 1178 1179 1180 1181 1182 1183 1184 1185 1186 1187 1188 1189 1190 1191 1192 1193 1194 1195 1196 1197 1198 1199 1200 1201 1202 1203 1204 1205 1206 1207 1208 1209 1210 1211 1212 1213 1214 1215 1216 1217 1218 1219 1220 1221 1222 1223 1224 1225 1226 1227 1228 1229 1230 1231 1232 1233 1234 1235 1236 1237 1238 1239 1240 1241 1242 1243 1244 1245 1246 1247 1248 1249 1250 1251 1252 1253 1254 1255 1256 1257 1258 1259 1260 1261 1262 1263 1264 1265 1266 1267 1268 1269 1270 1271 1272 1273 1274 1275 1276 1277 1278 1279 1280 1281 1282 12

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15.0 PROCEDURE (Continued)

3. It is important to use the correct method file. If the sample is a BFB, then use the BFB method file for the instrument being used. If the sample is a Standard or a Sample, then use the Sample method file for the instrument being used.
4. For the field "Bottle # Front": type "-1"
5. Go to Quant field and either type "Y" if sample or standard or type "N" if BFB. If "N," then go to step 18.
6. In Report field type "M" for medium report (includes quant report and chromatogram), or "F" for full report (includes quant report, chromatogram, and reference spectra comparison for detected analyts). If forms will be generated, type "N" for none. Chg.
A
7. "Aux Reprt": field - disregard.
8. Quant ID File: type ID file name for the instrument being used
9. "Output File": field - ignore, program supplies automatically
10. "Dilution Factor": field, type dilution factor for water sample, for a soil leave at 1.00000
11. "Label Meth (1-5)": field, type 4 (optional) Chg.
A
12. "B (background subtract) option (Y or N)": enter "Y" (optional) Chg.
A
13. "Scale from" field enter "start run time" listed in method file or leave at 0.00 Chg.
A
14. "__ page TIC" field, enter "2" for a 2-page chromatogram
15. "Qion replaces..." field, analyst choice or disregard. Chg.
A
16. "Omit/Only..." field, type "-I-S"
17. Disregard ARCHIVE fields and User Prog 1 and 2 fields
18. "FMGR TR File:" field, type TUNVOA for BFB analysis
- 18a. For samples and QC samples, type appropriate form program
- 18b. Type "=" file name in 3G Chg.
A
- 18c. Type "Y" or "P" name in 4G, "Y" file for samples or blanks, "P" for QC samples
- 18d. Aux: TRFI1
19. Press ENTER key, data will be stored and screen will go to next sample

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15.0 PROCEDURE (Continued)

20. Repeat above steps (1 to 19) for each sample to be analyzed

21. When all data has been entered, press F7 CREATE/REPLACE key to store sequence on data cartridge.

22. Press F8 EXIT/RESTART key

Sequence: DEMO0001 US Sample 11		BEOY Rev: 880930 170172																					
Data File Name: DEMO1		SAMPLE INFORMATION																					
Name: BEOY Demo																							
Misc Data: AGD 881013																							
Method: VOAB		Bottle #	Front: Rear: Samp Size:																				
Quant(Y or N):		Report Format (N,S,M,F,E,P):																					
Quant ID File: VOAB US		Aux Reprt: REPR2																					
		-Output File: Demo1																					
		Dilution Factor: 1000000 (-n means 1/n)																					
REPRT Options:																							
Label Meth (1-5): 8 option (Y or N): Scale from 00000 min. page TIC																							
Qion replaces Rt or Scan# (R or S): Omit/Only (-/+) Types:																							
ARCHIVE: Data File: Quant ID File: Quant Output File:																							
U	After Quant ? Additional Program Data																						
S	User Prog 1:																						
E	User Prog 2:																						
R	FMGR TR File: DEMO1	EG:	4G: Aux:																				
<table border="1"> <thead> <tr> <th>Header</th> <th>Phaser</th> <th>Manibus</th> <th>Nex</th> <th>Unit</th> <th>Screen</th> <th>Control</th> <th>Sample</th> <th>Replican</th> <th>RunTime</th> </tr> </thead> <tbody> <tr> <td>Sample</td> <td>Sample</td> <td>Sample</td> <td>Sample</td> <td>Sample</td> <td>Sample</td> <td>Sample</td> <td>Sample</td> <td>Sample</td> <td>Sample</td> </tr> </tbody> </table>				Header	Phaser	Manibus	Nex	Unit	Screen	Control	Sample	Replican	RunTime	Sample	Sample	Sample	Sample	Sample	Sample	Sample	Sample	Sample	Sample
Header	Phaser	Manibus	Nex	Unit	Screen	Control	Sample	Replican	RunTime														
Sample	Sample	Sample	Sample	Sample	Sample	Sample	Sample	Sample	Sample														

Make changes to existing sample and hit ENTER

Figure 2. Sample information.

C. Load Samples into P&T

1. Attach syringe to the 3-way valve for the position to be loaded
2. Open valve
3. Compress syringe plunger until all of syringe content has been transferred to the sparge position.
4. Close valve

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15.0 PROCEDURE (Continued)

5. Remove syringe
6. Repeat until all samples have been loaded
- D. Start P&T
 1. Ensure P&T is using appropriate method
 2. Enter sample positions to be used
 - a. press F2 ALS
 - b. enter starting position and stopping position
 - c. press F4 EXIT
 3. Press START button
- E. Start system for data collection
 1. From the FMGR prompt on one of the system's terminals type BAMON and press RETURN key
 2. Type BA (number for instrument being used)(sequence file name)
 3. Type GO (number for instrument being used)
 4. To ensure the instrument has started type ST, (number for instrument being used). "Run currently active" appears at the bottom of the screen.
 5. Type EX and press RETURN.

15.1.3 Check spectra for the BFB peak against the criteria in Table 1.

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15.0 PROCEDURE (Continued)

Table 1. BFB Peak Information

Mass	Relative Abundance Required
50	15 to 40 % of mass 95
75	30 to 60 % of mass 95
95	base peak, 100 % relative abundance
96	5 to 9 % of mass 95
173	less than 2 % of mass 174
174	greater than 50 % of mass 95
175	5 to 9 % of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9 % of mass 176

15.1.4 If the tune check meets the above criteria, then the analyst has 12 hours from the time of injection to analyze standards and samples.

15.1.5 If the tune check does not meet the criteria, then the analyst must either:

A. Repeat the 50 ng BFB tune check, or

B. Retune and recalibrate the instrument.

15.2 Perform daily calibration check by analyzing a 50 ppb standard.

15.2.1 Spike 100 mL of reagent water with 50 μ L of CAL-A and CAL-G 100 ppm working standards. To 5 mL of this solution in a syringe, add 10 μ L of ISS 25 ppm working standard to give 50 ppb standard of each analyte.

Chg.
A,B

15.2.2 Perform P&T analysis (refer to Section 15.1.2).

15.2.3 Evaluate check standard.

A. CRT, FF (clear printer)

B. CBCHK, (CB file name) all compounds to be analyzed with 1 ppb limit must meet criteria for 6 point calibration, all others meet for 5 point calibration.

Chg.
A

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15.0 PROCEDURE (Continued)

- A. Check the response factors (RFs) of the SPCCs for a minimum response factor of 0.300 (0.250 for Bromoform).
 - B. Check % relative standard deviation (RSD) for the CCC.
 - C. If the percent difference for each CCC is less than 25%, the initial calibration is assumed to be valid, however, if the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit.
 - D. Check %RSD for each required analyte. %RSD must be <25%.
 - E. Evaluate internal standards' (IS) area responses and retention times (RT).
 - 1. The RT for any IS shall not change by more than 30 seconds from the last check calibration (12 hours).
 - 2. The area response for any IS shall not change by more than a factor of two (-50% to +100%) from the last daily calibration standard check.
 - F. TR, RRTRRV, (Data file name), (Output file name)
 - G. QCAL, (Output file name), (ID file name), C
- 15.2.4 If calibration check standard meets minimum requirements, then update reference spectra and analyze samples by running QREF program.
- 15.2.5 If calibration check standard does not meet any one of the requirements, then the system must be inspected for malfunctions and corrections made as required. Potential problems could include: standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, active sites in the column or chromatographic system, dirty source, and deteriorating trap.
- 15.2.6 If the problem has been corrected, then either re-run calibration check standard or re-calibrate according to CP-QTS-1112.
- 15.2.7 If no source of the problem can be determined after corrective action has been taken, a new five- or six-point calibration must be generated according to CP-QTS-1112. This criterion must be met before quantitative sample analysis begins. If the CCCs are not required analytes by the permit, then all required analytes must meet the 25% difference criterion.

Chg.
A,BChg.
AChg.
A

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15.0 PROCEDURE (Continued)

15.3 Analyze samples.

15.3.1 Prepare samples for P&T analysis.

NOTE

Each batch of samples shall begin with a blank water sample, to include either one duplicate and one matrix spike, or one matrix spike and one matrix spike duplicate, and end with a 10 ppb Laboratory Control Spike (LCS). In addition, soil runs shall include a methanol blank.

A. Water Samples and TCLP extracts

1. Samples with no historical data available should be first analyzed at a minimum of 1:1000 dilution. Dilute according to one of the following: Other dilutions may be made, dependent on knowledge of other samples from the general vicinity.

| Chg.
A,B

 - a. Prepare samples requiring a dilution larger than 1:500 in a volumetric, then transfer to a 5 mL syringe. Go to step 3.

| Chg.
A,B
 - b. Prepare samples directly in a 5 mL syringe filled with organic free reagent water by adding at least 10 μ L (1:500). Go to step 3.

| Chg.
A,B
2. If no dilution is required: Remove the plunger from a 5 mL syringe and attach a closed syringe valve. Open the sample bottle and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Goto step 3.

| Chg.
A
3. Add 50 ppb ISS (10 μ L of the 25 ppm ISS working standard) to the syringe containing the sample.

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15.0 PROCEDURE (Continued)

NOTE

Samples must be stored with zero headspace. Therefore, if there is no sample backup, a syringe backup must be taken at this time. The syringe backup is kept only until the analyst determines that the sample has been analyzed properly. Syringe backups should be analyzed within 24 hours.

B. Water miscible liquids

Analyze as water samples after first diluting at least 50 fold (1:50) with organic-free reagent water.

C. Soil/sediment

1. Weigh approximately 5 grams into a VOA vial (note and record the actual weight used in the logbook)
2. Pipet 10 mL of Methanol into VOA vial
3. Inject 10 μ L of 2500 ug/mL stock surrogate standard into the vial (below surface of methanol). When 100 μ L of the extract is diluted to 5.0 mL, the concentration will be 50 ug/L.
4. If the sample is a matrix spike, add 12.5 μ L of the 2000 ug/mL stock matrix spiking standard. When 100 μ L of the extract is diluted to 5.0 mL, the concentration will be 50 ug/L.
- 4a. Pipet 10 mL methanol into a VOA vial as a methanol blank. Pipet the surrogate into the methanol blank.
5. Shake each VOA vial for 2 minutes
6. Place VOA vials in refrigerator and allow sediment to settle out of methanol layer.
7. Pipet the extract into a GC vial with minimal headspace for storage.
8. Text deleted.

Chg.
AChg.
AChg.
A

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15.0 PROCEDURE (Continued)

9. Prepare for injection:

- a. Remove the plunger from a 5.0 mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with water.
- b. Replace the plunger and compress the water to vent trapped air.
- c. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards.
- d. Add 10 μ L of internal standard solution.
- e. Add 100 μ L of sample extract.

15.3.2 Perform P&T analysis (refer to Section 15.1.2).

16.0 CALCULATION OR INTERPRETATION OF RESULTS

- 16.1 If a soil sample, then back calculate the concentration based on mass of soil weight out. Concentration of compound in soil is determined using the following equation:

$$\text{Analyte Concentration in Soil (mg/kg)} = \frac{C_t \left(\frac{5000 \mu\text{L}}{V_e} \right) V_m \left(\frac{1 \text{mg}}{1000 \mu\text{g}} \right) \left(\frac{1 \text{L}}{1000 \text{ml}} \right)}{m_s \left(\frac{1 \text{kg}}{1000 \text{g}} \right)}$$

Where: C_t = concentration of analyte detected on sample report ($\mu\text{g/L}$) V_e = volume of extract (μL) V_m = Volume of methanol used in extraction (mL) m_s = mass of soil weighed out in grams (g)

For example: following this procedure 10 mL methanol (V_m) is used to extract the soil and then 100 μL of the extract (V_e) is analyzed, the formula simplifies to:

16.0 CALCULATION OR INTERPRETATION OF RESULTS (Continued)

$$\text{Analyte Concentration in Soil (mg/kg)} = \frac{C_r}{m_s} \left(\frac{0.5L \text{ mg g}}{\mu\text{g kg}} \right)$$

16.2 Interpret all sample results by comparing with the following acceptance criteria:

- 16.2.1 Sample was analyzed within 12 hours of the BFB tune check
- 16.2.2 IS areas have not changed by more than a factor of 2 from the daily check standard.
- 16.2.3 RT's for the IS have not changed by more than 0.5 minutes.
- 16.2.4 Surrogate recoveries are within the limits as indicated in Table 2:

Table 2. Surrogate Recovery Limits

Surrogate	QC Limits for Water matrix	QC Limits for TCLP matrix	QC Limits for Soil Matrix
1,2-dichloroethane-d4	76 - 114 %	70-120%	70-120%
Toluene-d8	88 - 110 %	81-117%	81-117%
Bromofluorobenzene	86 - 115 %	74-121%	74-121%

Cbq.
A

- 16.2.5 Inspect spike for recovery limits (refer to Table 3. "Spike Recovery Limits")

Table 3. Spike Recovery Limits

Compounds	Limits for Water Matrix	Limits for TCLP Matrix	Limits for Soil Matrix
Benzene	76-127%	66-142%	66-142%
Carbon Tetrachloride	75-120%	70-125%	70-125%
Chlorobenzene	75-130%	60-133%	60-133%
Chloroform	75-120%	70-125%	70-125%
1,2-dichloroethane	75-120%	70-125%	70-125%
1,1-dichloroethene	61-145%	59-172%	59-172%
Methyl Ethyl Ketone	70-130%	61-145%	61-145%
Tetrachloroethene	75-120%	70-125%	70-125%
Trichloroethene	71-120%	62-137%	62-137%
Vinyl Chloride	70-130%	61-145%	61-145%

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16.0 CALCULATION OR INTERPRETATION OF RESULTS (Continued)

- 16.2.6 Compare sample and sample duplicate or spike and spike duplicate (as applicable) for agreement. Duplicate analyses should agree within a 25 % difference.

$$\frac{C_s - C_d}{C_t}$$

C_s = Compound concentration in sample or spike
 C_d = Compound concentration in duplicate
 C_t = average Compound concentration for both analyses

- 16.2.7 Check 10 ppb LCS for recoveries of requested analytes. Recoveries should be in the 75 to 125 percent range.

- 16.2.8 If any sample fails to meet acceptance criteria Sections 16.2.1 to 16.2.4, then reanalyze sample. If there is no backup sample, then the data must be qualified as conditional or estimated.

Chg.
B

- 16.2.9 If a QC sample fails to meet acceptance criteria Sections 16.2.1 to 16.2.7, then reanalyze the entire batch. If a sample has no backup, then its data must be qualified as conditional or estimated.

Chg.
A

- 16.3 Compare mass spectrum for each compound identified by the quant report to the corresponding standard reference mass spectrum for positive identification.

- 16.4 Examine chromatogram for unidentified peaks. If unidentified peaks are present, and their areas are 10% or greater of the area of the nearest IS, then identify by performing a library search.

17.0 REPORT

Approved results are reported to the requestor by AnaLIS. The reports generated by the procedure are stored in the daily file.

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18.0 PRECISION AND BIAS

The performance of this procedure was demonstrated by analyzing eight independently spiked water samples at a concentration of 1.0 ppb with the compounds listed in Table 1 of Appendix D, "MDL Determinations and Data." Table 2 of Appendix D includes accuracy and precision data for these compounds. Method detection limit (MDL) values were also calculated from this data and are presented in Table 2.

19.0 QUALITY ASSURANCE/QUALITY CONTROL

19.1 Data packages are reviewed by two analysts.

19.2 A 10 ppb Laboratory Concentrated Spike is analyzed at the end of each run.

19.3 Minimum Internal Standard areas must be met.

19.4 Minimum Surrogate recoveries must be met.

19.5 Retention time shift is checked for all analytes.

19.6 Each batch includes a matrix spike and a matrix spike duplicate, or a duplicate and a matrix spike.

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Appendix A
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CLEANUP OF THE PURGE AND TRAP UNIT

A. Steam Clean Procedure

The steam cleaning procedure is specifically designed for the LSC 2000 and requires a Sample Heater. This procedure can also be used with the ALS 2016/2032 if the system includes the Automatic Sample Heaters.

1. Leave the instrument ON with all the valves and lines heated (LSC 2000 valve and line at 200° C and the ALS 2016 valve and line at 175 C).
2. Disconnect the top of the trap or install a #0 (Blank) trap in the concentrator.
3. Place 5-10 ml of blank water in the sparge vessel.
4. Heat the water to 100° C using the sample heater.
5. Purge the water for 20 to 30 minutes.
6. Remove the heater and the water from the sparge vessel.
7. Purge the lines for at least 15 minutes (for each position if running an autosampler) to remove any excess water. Increasing the purge flow will help remove the water more quickly.
8. Reconnect the trap and bake for 30 minutes with the line and valve temperatures still elevated.
9. If any parameters were altered for this procedure (such as, valve/line temperatures, purge flow) set them back to normal operating values.

B. Sample Flush Procedure

The sample flush procedure involves flushing parts of the system with reagent water. This procedure can be used for blockages or contamination problems. Contamination from highly concentrated samples may require back-flushing the P&T system from the trap to the sparger position with methanol and then reagent water.

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1. Flushing Sample Lines

- a. Step to the position that foamed (if using an autosampler)
- b. Remove the glassware
- c. Disconnect the top of the trap on the concentrator
- d. Flush approximately 20 ml of water into the line that connects to the top of the trap. The water will come out at the mount where the glassware was connected on the concentrator or autosampler. It is best to do this with the line and valve temperatures less than 50° C.*

CAUTION

Excessive water introduced to the trap will damage the trap.

- e. After flushing with water, flush with air (using an empty syringe) to blow out any water that may still be in the lines.
 - f. Reconnect the glassware, leaving the trap disconnected so that no water is baked onto the trap (or install a No. 0 trap).
 - g. Bake the position just flushed for approximately 30 minutes. Increase the temperature of the line and valves (ALS2016 valve & line to 175° C and LSC2000 valve & line to 200° C) to aid in removing the water from the flushed lines.**
2. Flushing from Transfer line to the GC
- a. To flush the transfer line:
 1. Disconnect the trap.
 2. Disconnect the transfer line from the GC.
 3. Step the concentrator to desorb.

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4. Flush 20 mL water into the line that connects to the top of the trap. The flushed water will come out the end of the transfer line.
- b. To flush the bottom of the trap line:
 1. Disconnect the trap at the bottom.
 2. Step the LSC2000 to purge position so the vent valve will be open.
 3. Flush 20 mL water into the line that connects to the bottom of the trap. The water will come out the vent.
 4. Increase the LSC2000 line temperature to 150°C.**
 5. Bake the LSC2000 for at least 15 minutes.
 6. Reconnect the transfer line.
3. Flushing the purge lines on an ALS 2016
 1. Remove the left hand panel of the LSC 2000.
 2. Remove the silver oven cover.
 3. Disconnect the ALS 2016 purge line at the 1/16" to 1/16" union in the lower corner in the valve oven area. (Locate the two lines from the 2016 transfer line as they enter the back of the LSC 2000. One of the lines enters the "T" connection at the 9 o'clock position and the other line goes to the union. Disconnect at the union.)
 4. Flush reagent water into this union and toward the transfer line of the 2016 (flush each position as needed).
 5. Reconnect the union and purge the lines for at least 10 minutes to remove the remaining water.

*An increase in temperature creates more back pressure, and increases the pressure needed to flush water through the system. Also, the dry purge valve could be damaged if too much pressure is put on this valve.

**At end of process, reset temperatures to previous settings.

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Appendix B

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Tekmar Parameters

All parameters for the P&T, autosampler, and cryofocusing unit are stored in a Method file on the P&T unit. All temperatures are in Celsius and all times are in minutes. These parameters are adjusted to meet the acceptance criteria of the method. The parameters are dependent on the instrument.

Chg.
A

Screen #1

Press "page down" key for more:

```

Standby: 35°      Prepurge: NI
Preheat: NI      Sample: NI
Purge: 11:00     Dry Purge: 0:00
  
```

Screen #2

Press "page down" key for more:

```

MCM Cooldown: NI
Cap Cooldown: 150°
Desorb Preheat: 150°
Desorb: 4:00 min at 180°
  
```

Screen #3

Press "page down" key for more:

```

Valve: 105°      Line: 105°
Mount: 0         Aux Heater: NI
2016 Valve: 105°      Line: 105°
2032 Valve: NI       Line: NI
  
```

Screen #4

Press "page down" key for more:

```

Inject: 1:00 min at 200°
Bake: 10:00 min at 200°
BGB OFF      BGB Delay: 0 sec
Auto Drain: ON
  
```

Screen #5

```

Cap Union: 180°      MCM Heat: NI
Runs per Sample: 1
  
```

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GC PARAMETERS

All GC parameters for the analysis of BFB are stored in a BFB method file. All temperatures are in Celsius. Time and temperature parameters are adjusted at calibration to meet the criteria of the method.

Chg.
A

```

Injection Port temp: 100.0°
Interface temp: 250.0°

```

```

GC temp program:
Initial temp: 35° for 0.5 min
temp rate: 20°/min
final temp: 180° final time: 10 min

```

```

Oven equilibration time: 0.5 min
RUN time: 10 min
Scan start time: 1.5 min

```

```

Scan Parameters:
Mass Range: 35 to 300
Multiplier voltage: XX Number of A/D samples: XX
GC Peak threshold: 20000 counts

```

* Set when tuning the instrument.

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All GC parameters for the analysis of standards and samples are stored in a sample method file. All temperatures are in Celsius. Time and temperature parameters are adjusted at the time of the calibration to meet the criteria of the method.

Chr.
A

```

Injection Port temp: 100.0
Interface temp: 250.0

```

```

GC temp program
Initial temp: 35 for 5.0 min
Temp rate: 6 /min
Final temp: 180 final time: 30 min

```

```

Oven equilibration time: 0.5 min
RUN time: 30 min
Scan start time: 0:00 min

```

```

Scan Parameters:
Mass Range: 35 to 300
Multiplier voltage: XX Number of A/D samples: XX
GC Peak threshold: 20000 counts

```

* Set these values to the same values used in the BFB method file.

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MDL DETERMINATIONS AND DATA

Table 1. MDL Determinations

PEAK	CMPD	Concentration (µg/L)							
		>SB345	>SB338	>SB339	>SB340	>SB341	>SB342	>SB343	>SB344
1	9 CHLOROFORM	1.13	1.23	1.21	1.11	1.09	1.07	1.29	1.19
2	12 1,1-DICHLOROETHANE	1.18	1.10	1.22	1.06	1.06	0.98	1.20	1.19
3	13 1,1-DICHLOROETHENE	1.03	1.43	1.16	1.15	1.09	-	1.12	1.28
4	14 CIS-1,2-DICHLOROETHENE	1.09	1.23	1.24	1.09	-	1.02	-	1.17
5	15 TRANS-1,2-DICHLOROETHENE	1.15	1.08	1.33	1.19	1.03	1.13	1.21	1.33
6	16 2,2-DICHLOROPROPANE	1.27	1.18	0.69	1.11	1.12	0.66	1.34	1.33
7	17 METHYLENE CHLORIDE	1.21	1.34	1.36	1.21	1.22	1.13	1.42	1.35
8	22 BENZENE	1.11	1.17	1.14	1.12	1.02	1.12	1.13	1.20
9	24 BROMODICHLOROMETHANE	0.98	0.87	1.05	1.02	0.90	0.76	0.95	0.91
10	26 CARBON TETRACHLORIDE	1.02	0.99	1.05	1.04	1.02	1.01	1.14	1.14
11	28 DIBROMOMETHANE	1.04	1.06	1.04	-	-	0.99	1.01	-
12	29 1,2-DICHLOROETHANE	1.11	1.13	1.12	-	1.11	1.02	1.08	1.07
13	31 1,2-DICHLOROPROPANE	-	-	-	-	1.13	1.07	1.06	-
14	32 1,1-DICHLOROPROPENE	1.15	1.11	1.11	1.11	1.19	1.20	1.22	1.19
15	33 CIS-1,3-DICHLOROPROPENE	1.42	1.66	1.56	1.57	1.46	1.45	1.43	1.45
16	35 TOLUENE	1.16	1.17	1.23	1.16	1.08	1.20	1.26	1.32
17	37 1,1,2-TRICHLOROETHANE	0.99	-	1.09	1.12	1.15	1.09	-	-
18	38 TRICHLOROETHENE	1.19	1.52	-	-	1.21	1.19	1.19	1.22
19	41 BROMOFORM	0.64	0.63	0.82	0.72	-	0.57	0.61	0.62
20	42 CHLORODIBROMOMETHANE	0.79	0.75	-	0.74	0.64	0.64	0.73	0.73
21	43 CHLOROBENZENE	1.24	1.14	1.18	1.12	1.13	1.10	1.24	1.17
22	44 1,3-DICHLOROPROPANE	1.17	1.09	1.18	1.01	1.15	-	1.09	1.09
23	45 ETHYLBENZENE	1.14	1.11	1.11	1.07	1.06	1.18	1.19	1.24
24	46 STYRENE	0.38	0.32	1.02	1.04	0.98	1.05	-	1.20
25	47 1,1,1,2-TETRACHLOROETHANE	-	1.12	1.04	1.04	0.98	0.92	1.00	1.27
26	48 TETRACHLOROETHENE	1.05	1.08	1.15	1.01	1.02	1.07	1.15	1.27
27	49 M,P-XYLENE	2.65	2.24	2.22	2.13	2.19	2.27	2.57	2.64
28	50 O-XYLENE	1.27	1.12	1.08	1.05	1.13	1.14	1.26	1.30
29	52 BROMOBENZENE	1.19	1.31	1.14	1.10	1.14	1.12	1.14	1.31
30	53 N-BUTYLBENZENE	1.21	1.05	1.15	1.15	1.11	1.25	1.28	1.40
31	54 SEC-BUTYLBENZENE	1.36	1.14	1.17	1.20	1.11	1.28	1.28	1.43
32	55 TERT-BUTYLBENZENE	1.45	1.24	1.16	1.12	1.08	1.30	1.31	1.40
33	56 2-CHLOROTOLUENE	1.21	1.22	1.16	1.08	1.08	1.21	1.28	1.31
34	57 4-CHLOROTOLUENE	1.29	1.11	1.16	1.11	0.99	1.16	1.29	1.26
35	59 1,2-DICHLOROBENZENE	1.55	1.19	1.13	1.14	1.33	1.20	1.26	1.35
36	60 1,3-DICHLOROBENZENE	1.29	1.18	1.23	1.21	1.13	1.14	1.30	1.34
37	61 1,4-DICHLOROBENZENE	1.43	1.26	1.21	1.20	1.17	1.20	1.39	1.42
38	62 HEXACHLOROBUTADIENE	2.98	1.38	1.57	1.41	1.42	2.02	1.99	2.26
39	63 ISOPROPYLBENZENE	1.21	1.11	-	1.14	1.09	1.21	-	1.34
40	64 P-ISOPROPYLTOLUENE	1.21	1.05	1.13	1.11	1.04	1.12	1.26	1.34
41	65 NAPHTHALENE	-	0.91	-	1.15	1.32	0.96	-	-
42	66 N-PROPYLBENZENE	1.23	1.10	1.12	1.10	1.04	1.15	1.25	1.34
43	67 1,1,2,2-TETRACHLOROETHANE	1.13	-	0.91	1.07	1.01	1.03	1.09	1.08
44	68 1,2,3-TRICHLOROBENZENE	2.16	-	1.37	-	1.27	1.22	-	1.38
45	69 1,2,4-TRICHLOROBENZENE	2.00	1.33	1.35	1.24	1.24	-	1.48	1.44
46	70 1,2,3-TRICHLOROPROPANE	1.51	0.73	1.34	1.21	0.98	1.29	-	1.50
47	71 1,2,4-TRIMETHYLBENZENE	1.41	1.31	1.21	1.18	1.22	1.25	1.30	1.40
48	72 1,3,5-TRIMETHYLBENZENE	1.21	1.19	1.27	1.14	1.14	1.27	1.30	1.44

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Table 2. Calculated MDL Values

	PEAK	CPD	N	R	S	P	MDL
1	9	CHLOROFORM	8	116.500	7.6904	6.6012	0.23056
2	12	1,1-DICHLOROETHANE	8	112.375	8.6179	7.6689	0.25836
3	13	1,1-DICHLOROETHANE	7	118.000	13.4164	11.3698	0.42163
4	14	CIS-1,2-DICHLOROETHANE	6	114.000	8.7636	7.6873	0.29489
5	15	TRANS-1,2-DICHLOROETHANE	8	118.125	10.8158	9.1563	0.32425
6	16	2,2-DICHLOROPROPANE	8	108.750	26.9059	24.7411	0.80663
7	17	METHYLENE CHLORIDE	8	128.000	10.0285	7.8348	0.30065
8	22	BENZENE	8	112.625	5.2355	4.6486	0.15696
9	24	BROMODICHLOROMETHANE	8	93.000	9.1963	9.8885	0.27570
10	26	CARBON TETRACHLORIDE	8	105.125	5.7678	5.4866	0.17292
11	28	DIBROMOMETHANE	5	102.800	2.7749	2.6993	0.10397
12	29	1,2-DICHLOROETHANE	7	109.143	3.8048	3.4860	0.11957
13	31	1,2-DICHLOROPROPANE	3	108.667	3.7859	3.4840	0.26367
14	32	1,1-DICHLOROPROPENE	8	116.000	4.5670	3.9370	0.13692
15	33	CIS-1,3-DICHLOROPROPENE	8	150.000	8.6189	5.7459	0.25839
16	35	TOLUENE	8	119.750	7.3046	6.0999	0.21899
17	37	1,1,2-TRICHLOROETHANE	5	108.800	6.0166	5.5300	0.22544
18	38	TRICHLOROETHENE	6	125.333	13.1250	10.4721	0.44165
19	41	BROMOFORM	7	65.857	8.4346	12.8075	0.26507
20	42	CHLORODIBROMOMETHANE	7	71.714	5.6484	7.8763	0.17751
21	43	CHLOROBENZENE	8	116.500	5.2915	4.5421	0.15864
22	44	1,3-DICHLOROPROPANE	7	111.143	5.9562	5.3590	0.18718
23	45	ETHYLBENZENE	8	113.750	6.2278	5.4750	0.18671
24	46	STYRENE	7	85.571	35.2603	41.2056	1.10811
25	47	1,1,1,2-TETRACHLOROETHANE	7	105.286	11.3829	10.8115	0.35773
26	48	TETRACHLOROETHENE	8	110.000	8.6355	7.8504	0.25889
27	49	M,P-XYLENE	8	118.188	21.7252	18.3819	0.65131
28	50	O-XYLENE	8	116.875	9.4482	8.0840	0.28325
29	52	BROMOBENZENE	8	118.125	8.3399	7.0602	0.25003
30	53	N-BUTYLBENZENE	8	120.000	10.9935	9.1613	0.32958
31	54	SEC-BUTYLBENZENE	8	124.625	11.1347	8.9346	0.33381
32	55	TERT-BUTYLBENZENE	8	125.750	13.2098	10.5048	0.39602
33	56	2-CHLOROTOLUENE	8	119.375	8.3826	7.0221	0.25131
34	57	4-CHLOROTOLUENE	8	117.125	10.4668	8.9364	0.31379
35	59	1,2-DICHLOROBENZENE	8	126.875	13.9534	10.9977	0.41832
36	60	1,3-DICHLOROBENZENE	8	122.750	7.7044	6.2765	0.23097
37	61	1,4-DICHLOROBENZENE	8	128.500	10.9675	8.5350	0.32880
38	62	HEXACHLOROBUTADIENE	8	187.875	55.6094	29.5992	1.66714
39	63	ISOPROPYLBENZENE	6	118.333	9.1579	7.7390	0.30816
40	64	P-ISOPROPYLTOLUENE	8	115.750	10.4437	9.0227	0.31310
41	65	NAPHTHALENE	4	108.500	18.7705	17.3000	0.85231
42	66	N-PROPYLBENZENE	8	116.625	9.8842	8.4752	0.29632
43	67	1,1,2,2-TETRACHLOROETHANE	7	104.571	7.1614	6.8483	0.22506
44	68	1,2,3-TRICHLOROBENZENE	5	148.000	38.6070	26.0858	1.44658
45	69	1,2,4-TRICHLOROBENZENE	7	144.000	26.3122	18.2724	0.82691
46	70	1,2,3-TRICHLOROPROPANE	7	122.286	28.2708	23.1186	0.88846
47	71	1,2,4-TRIMETHYLBENZENE	8	128.500	8.6023	6.6944	0.25789
48	72	1,3,5-TRIETHYLBENZENE	8	124.500	9.8995	7.9514	0.29678

N - Number of Data Points used in calculations

R - Percent Recovery

S - Standard Deviation of the Recovery

P - Percent Relative Standard Deviation