Monsanto

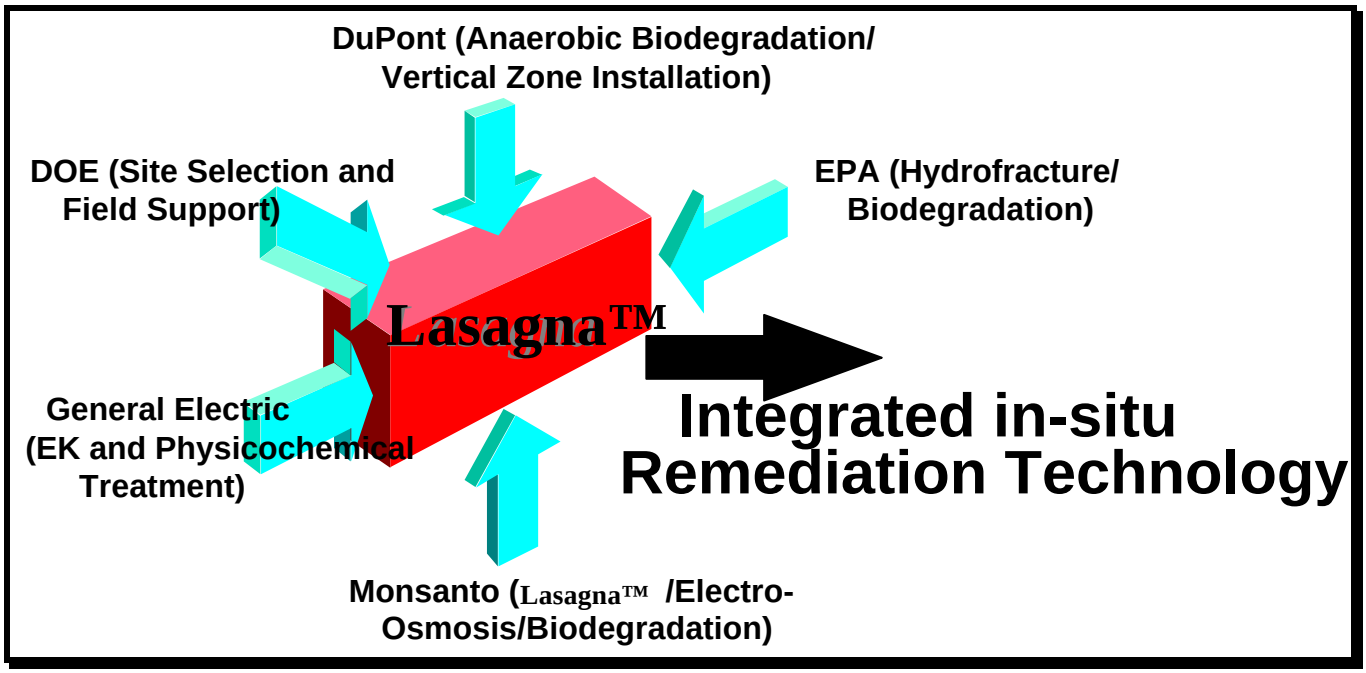
June 1996

Development of an Integrated *in-situ* Remediation Technology

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

B. Mason Hughes, Sa V. Ho, Christopher J. Athmer, and P. Wayne Sheridan (Monsanto Company), Stephen H. Shoemaker and John R. Larson (DuPont), Jay L. Clausen (LMES), John L. Zutman (ORNL-Grand Junction)

DOE Contract Number: DE-AR21-94MC31185



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20 March, 1997

Re: Ordering Information for *"Development of an Integrated in-situ Remediation Technology"* Topical Reports generated under DOE contract number DE-AR21-94MC31185 which was signed September 26, 1994.

Dear Sir/Ms.:

The following table summarizes ordering information for all technical reports written for the above referenced contract. Copies of these reports may be obtained from the Office of Scientific and Technical Information [(423)576-8401] if you are with DOE or a DOE contractor, or from:

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(continued on next page)

Title	Document Number
• **Topical Report for Task #1 entitled "Evaluation of Treatment Zone Formation Options" (September 26, 1994 - May 25, 1996)** Stephen H. Shoemaker, Richard C. Landis, Ronald J. Griffith, Dale S. Schultz, and Gary E. Quinton (DuPont Company)	**DOE/METC/31185—5436, DE97002165**
• **Topical Report for Tasks #2-4 entitled "Electrokinetic Modeling" (September 26, 1994 - May 25, 1996)** Andrew P. Shapiro (General Electric Company)	**DOE/METC/31185—5391, DE97002135**
• **Topical Report for Task #5 entitled "Cost Analysis" (September 26, 1994 - May 25, 1996)** Gary Quinton, Dale Schultz, Richard Landis, Ronald Griffith, and Stephen Shoemaker (DuPont Company)	**DOE/METC/31185—5389, DE97002134**
• **Topical Report for Task #6 entitled "Lab-Scale Development of Microbial Degradation Process" (September 26, 1994 - May 25, 1996)** J. Martin Odom (DuPont Company)	**DOE/METC/31185—5388, DE97002130**
• **Topical Report for Task #7 entitled "Development of Degradation Processes" (September 26, 1994 - May 25, 1996)** M. J. Brackin, M. H. Heitkamp and S. V. Ho (Monsanto Company)	**DOE/METC/31185—5495, DE97002165**
• **Topical Report for Tasks #8 and 10 entitled "Laboratory and Pilot Scale Experiments of the *Lasagna*™ Process" (September 26, 1994 - May 25, 1996)** Sa V. Ho, Christopher J. Athmer, and P. Wayne Sheridan (Monsanto Company) and Andrew P. Shapiro (General Electric Company)	**DOE/METC/31185—5375, DE97002150**
• **Topical Report for Task #9-Part I entitled "TCE Degradation Using Non-Biological Methods" (September 26, 1994 - May 25, 1996)** Andrew P. Shapiro, Timothy M. Sivavec, and Sunita S. Baghel (General Electric Company)	**DOE/METC/31185—5392, DE97002133**
• **Topical Report for Task #9 - Part II entitled "TCE Degradation Using Non-Biological Methods" (September 26, 1994 - May 25, 1996)** Robert G. Orth and David E. McKenzie (Monsanto Company)	**DOE/METC/31185—5393, DE97002131**

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• **Topical Report for Task #11 entitled "Evaluation of TCE Contamination Before and After the Field Experiment" (September 26, 1994 - May 25, 1996)** B. Mason Hughes, Sa V. Ho, Christopher J. Athmer, and P. Wayne Sheridan (Monsanto Company) Stephen H. Shoemaker and John R. Larson (DuPont) Jay L. Clausen (LMES) and John L. Zutman (ORNL-Grand Junction)	**DOE/METC/31185—5496, DE97002166**
• **Topical Report for Tasks #12 and 13 entitled "Large Scale Field Test of the *Lasagna*™ Process" (September 26, 1994 - May 25, 1996)** Christopher J. Athmer, Sa V. Ho, B. Mason Hughes, P. Wayne Sheridan, and P. H. Brodsky (Monsanto Company) Andrew P. Shapiro, Roy F. Thornton, and Joseph J. Salvo (General Electric Company) and Dale S. Schultz, Richard C. Landis, Ron Griffith, and Stephen H. Shoemaker (DuPont)	**DOE/METC/31185—5390, DE97002156**

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 *Lasagna*™ 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 *Lasagna*™ Process	Monsanto

F. Topical Report for Task #11

In January 1995, the Phase I *Lasagna*™ 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).

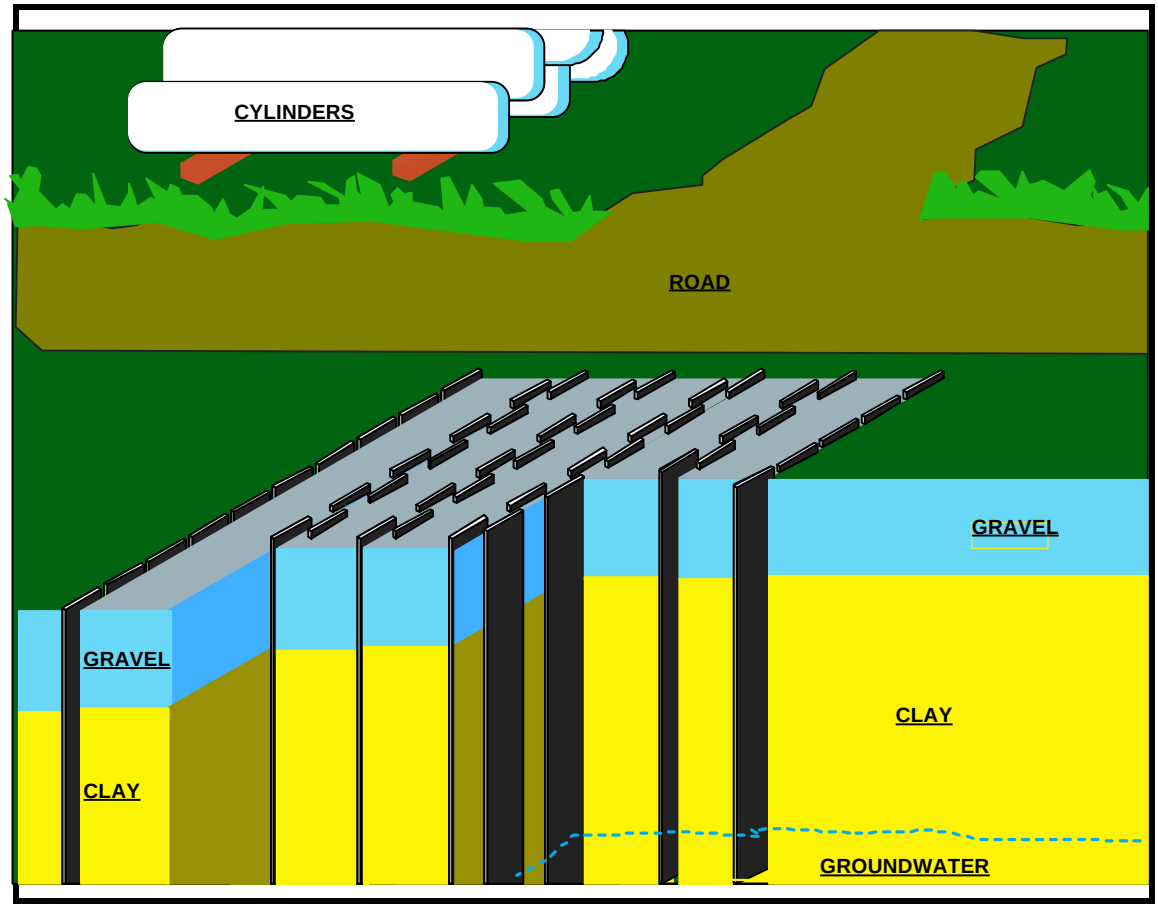

Figure F-1. Three-Dimension View of the Phase I *Lasagna*™ Field Experiment.

A detailed over-view of this site is shown in Figure F-2. In this figure, approximately ½ of the *Lasagna*™ 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™ Process" by Athmer, et. al., in preparation (1996).

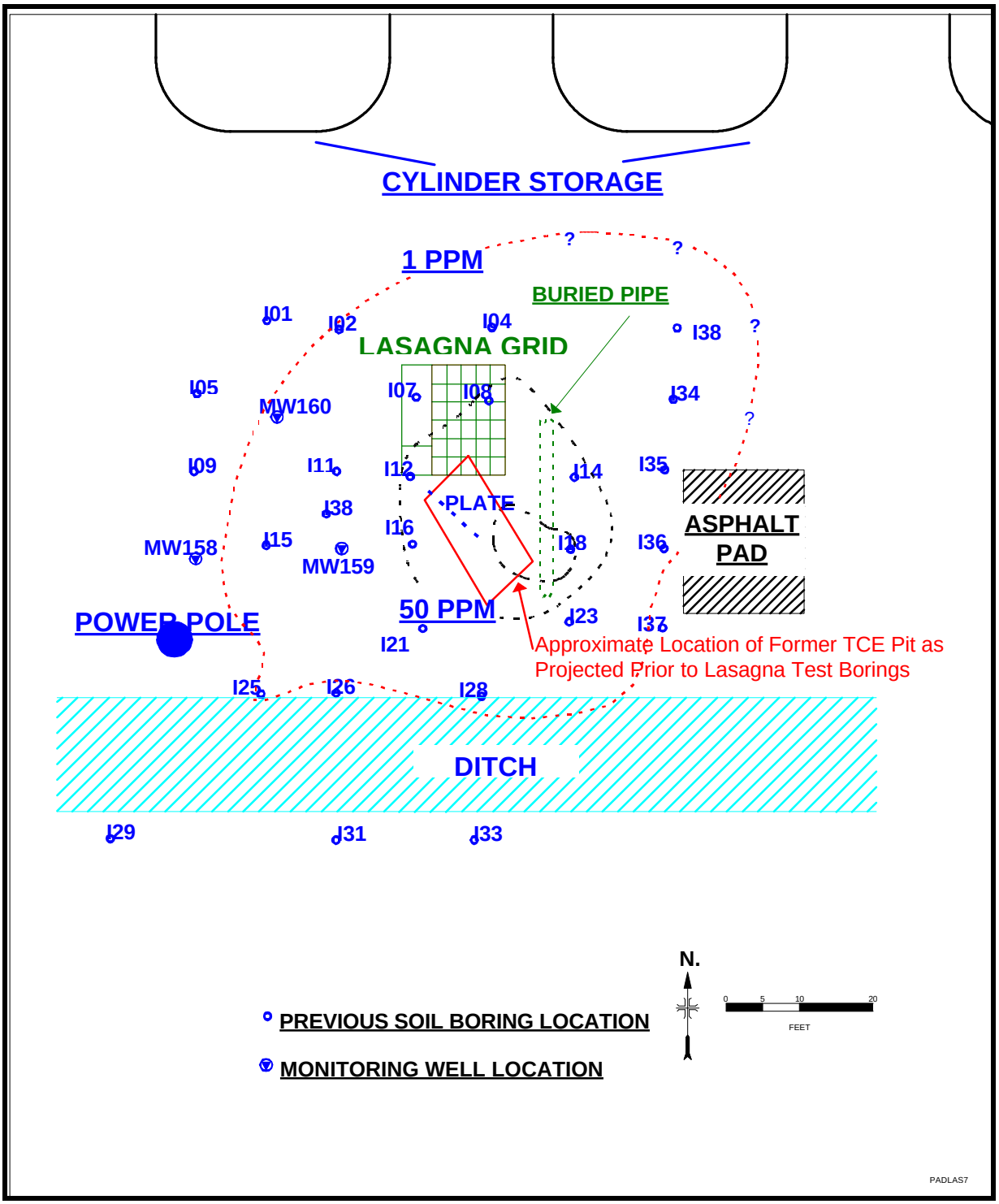

Figure F-2. Overview of the Phase I *Lasagna*™ Field Experiment.

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 15 - 16	84.8 7.3	46.1
I-8	15 - 16	65.4	65.4
I-12	11 - 12 15 - 16	319.6 84.4	202.0
I-14	15 - 16	71.2	71.2
I-16	9 - 10 15 - 16	5.3 77.7	41.5
I-18	15 - 16 18 - 19	121.8 36.9	79.4

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 x 103
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*™ 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.

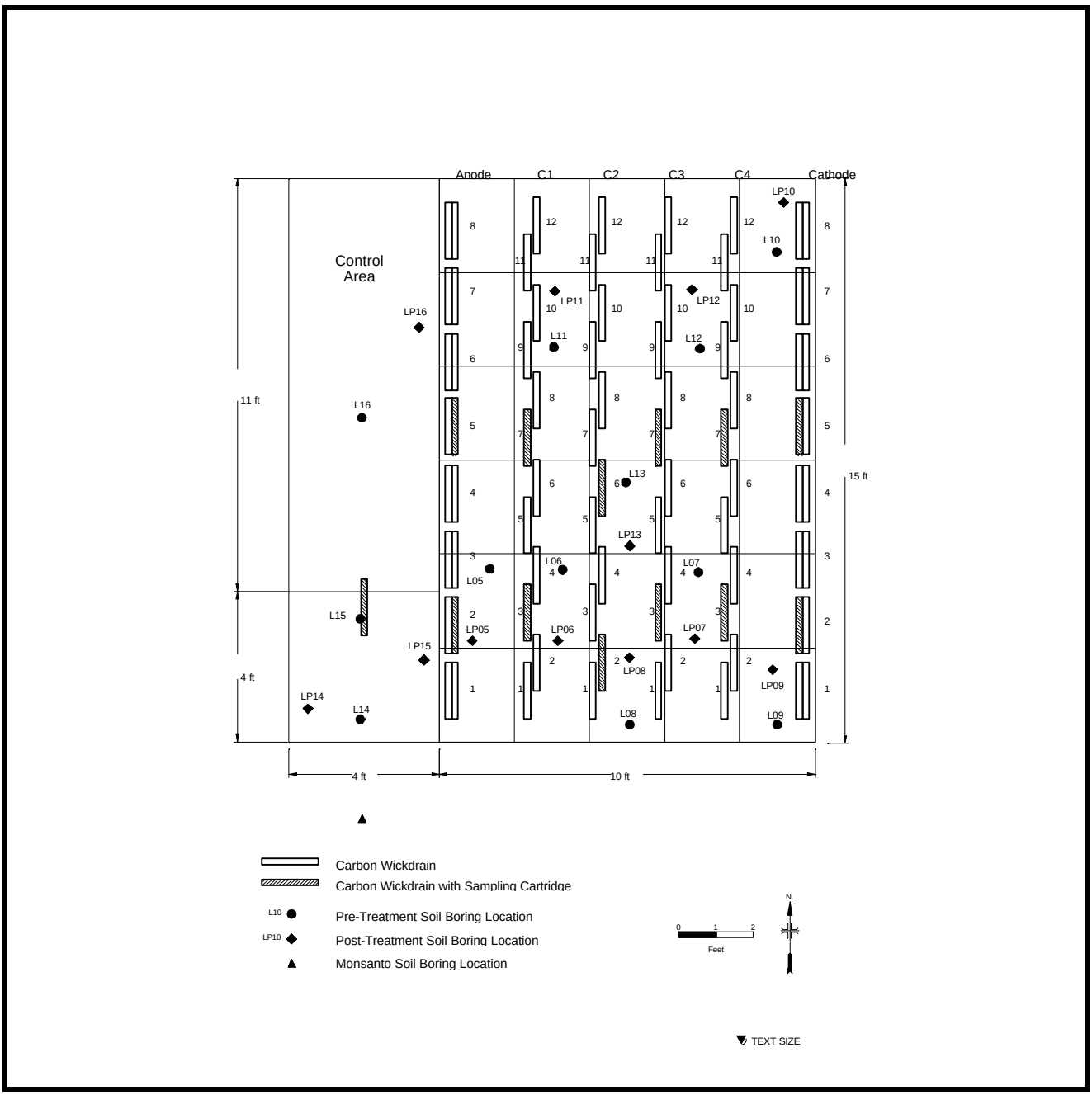

Figure F-3. Locations of Core Samples from the Phase I *Lasagna*™ Field Experiment.

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*™ 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*™ field experiment.

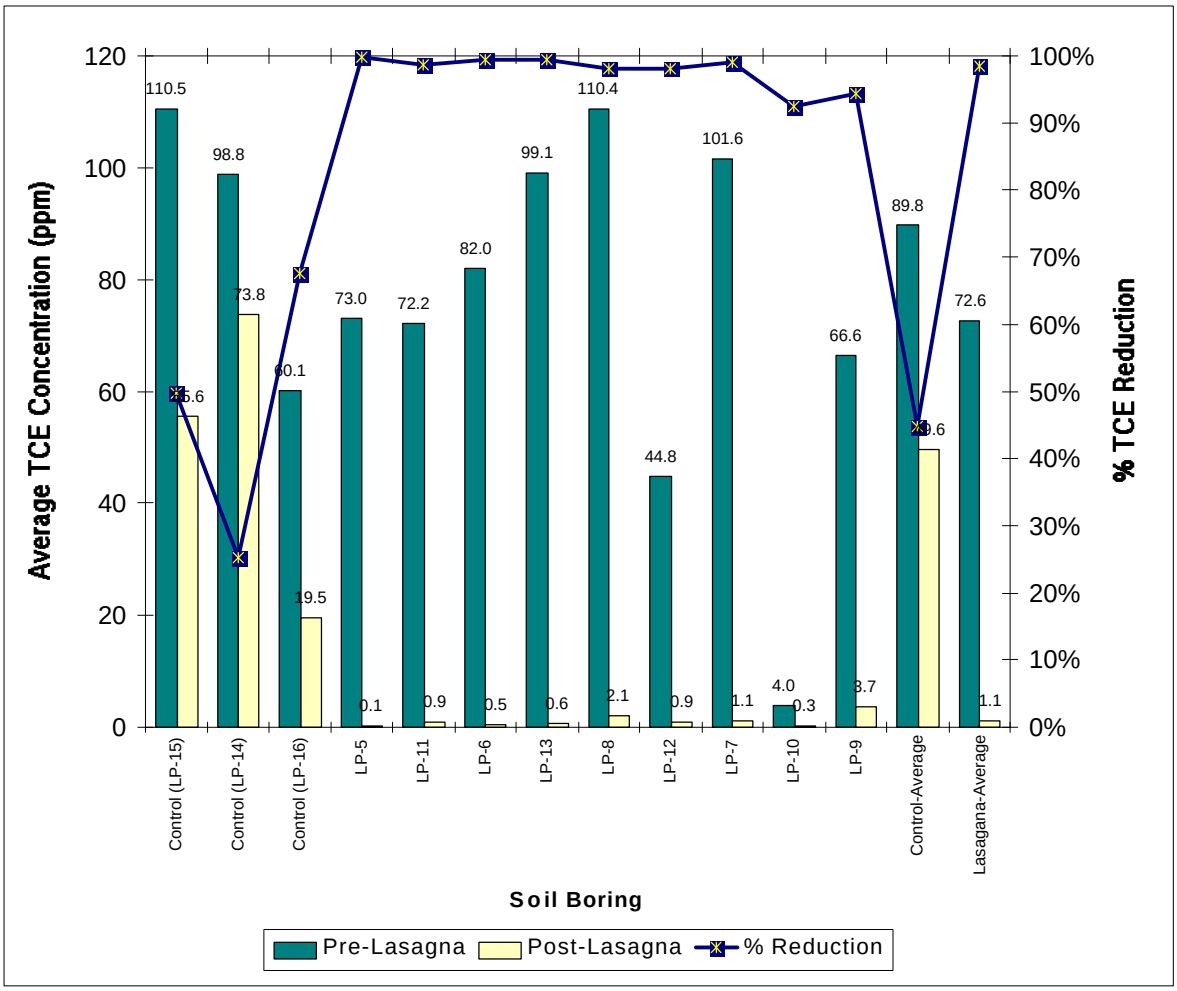

Figure F-4. Average TCE Concentrations Pre- and Post-*Lasagna*™ Field Experiment.

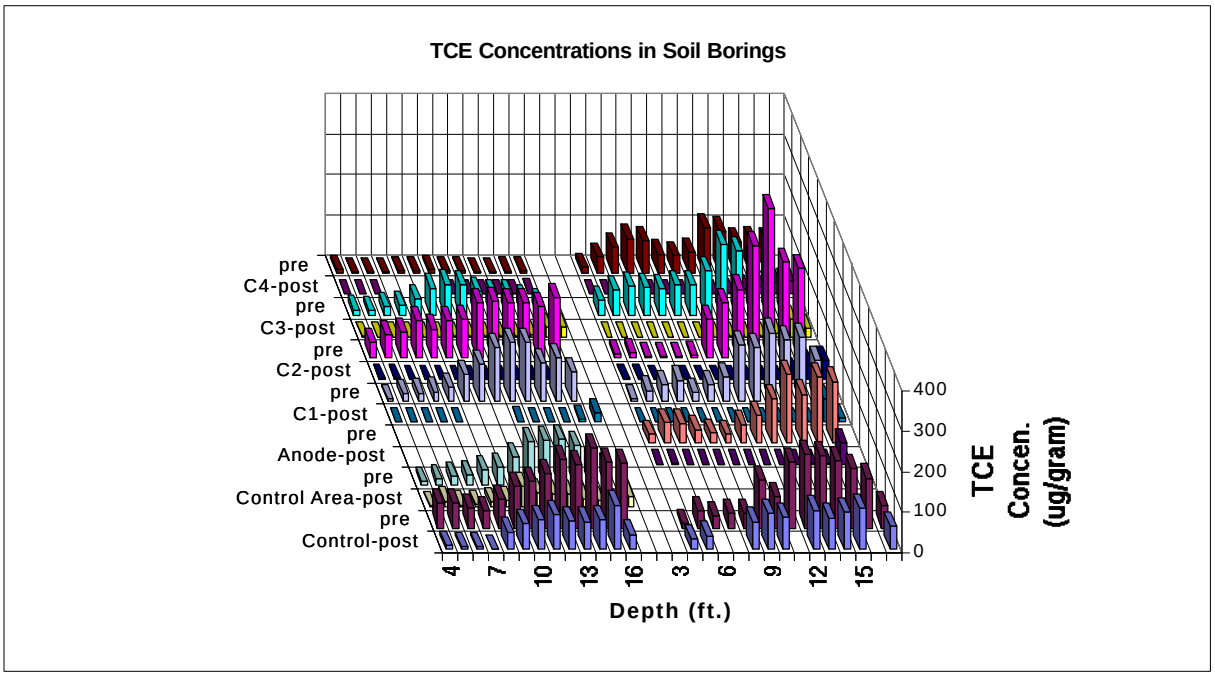

Figure F-5. TCE Concentrations Pre- and Post-*Lasagna*™ Field Experiment.

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.

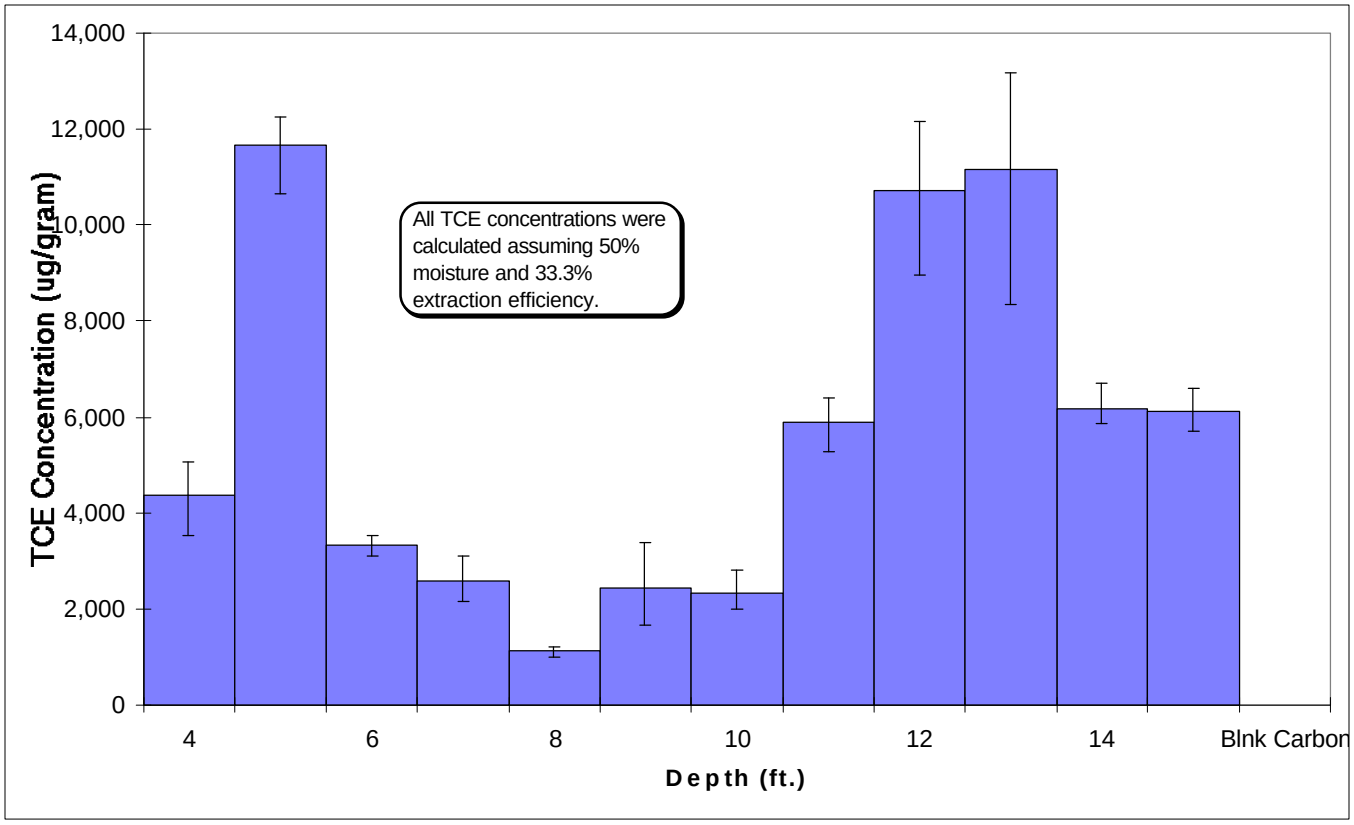

Figure F-6. Concentrations of TCE Measured in Carbon Cassette C4-7 After 2 Pore Volumes.

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.

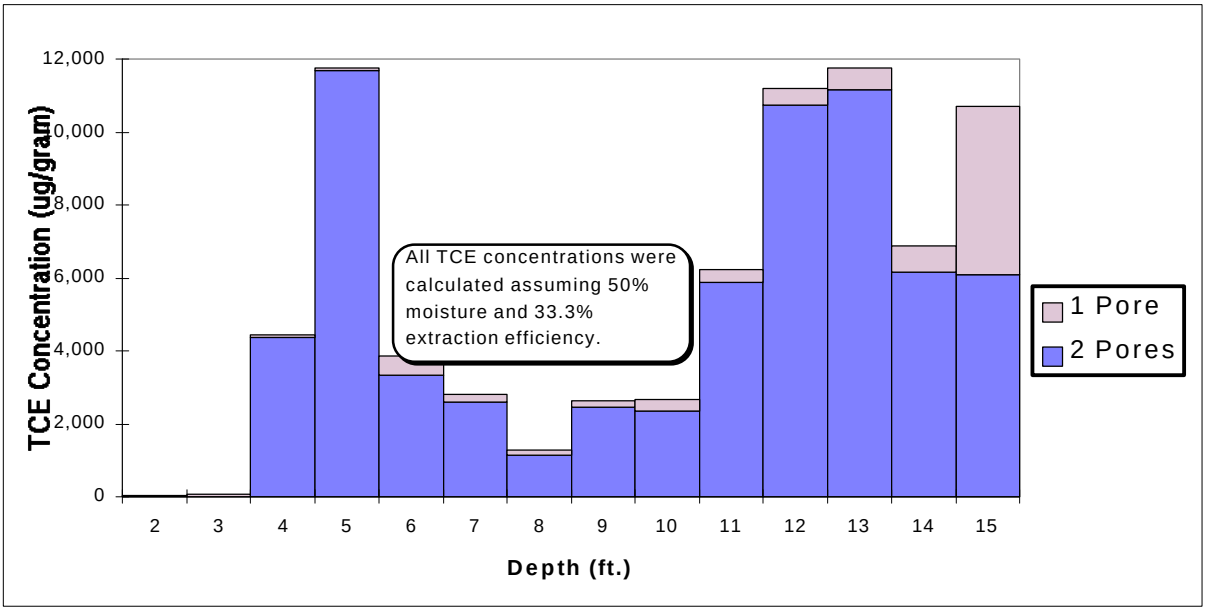

Figure F-7. Concentrations of TCE Measured in Carbon Cassette C4-7 After 2 and 1 Pore Volumes.

Table F-3. Carbon TCE Concentrations (mg/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	South Cartridges
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	North Cartridges
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	

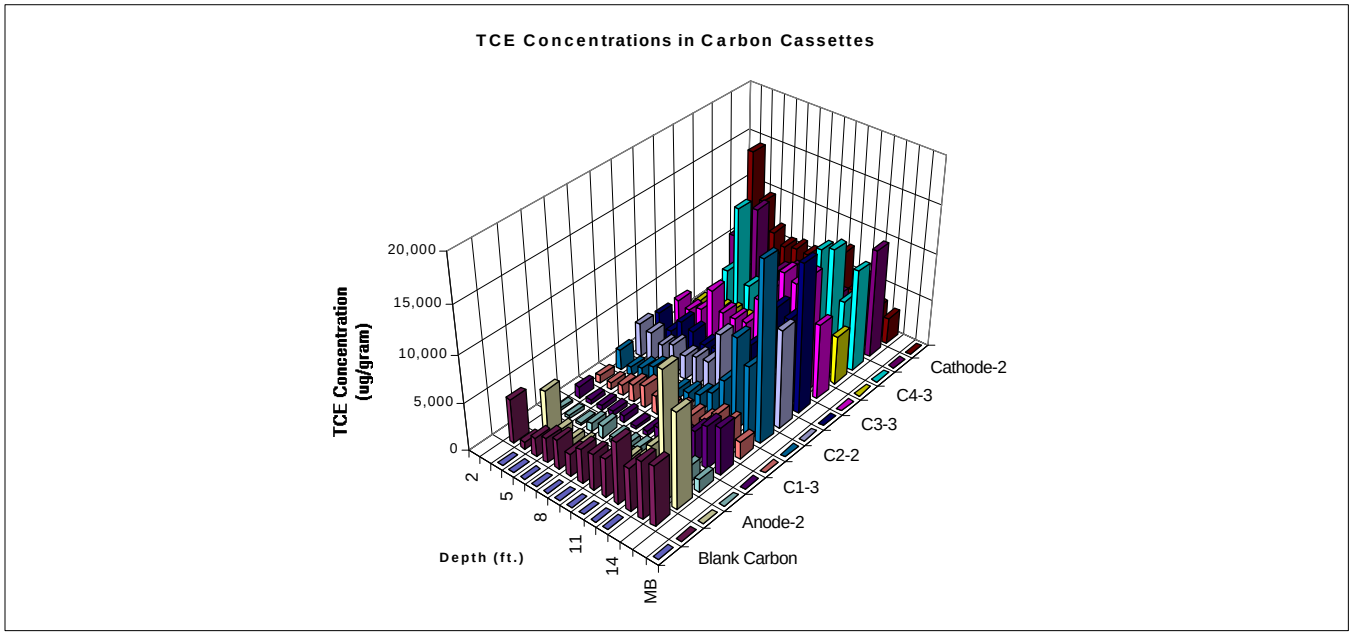

Figure F-8. Concentrations of TCE Measured in all Carbon Cassettes after 3 Pore Volumes.

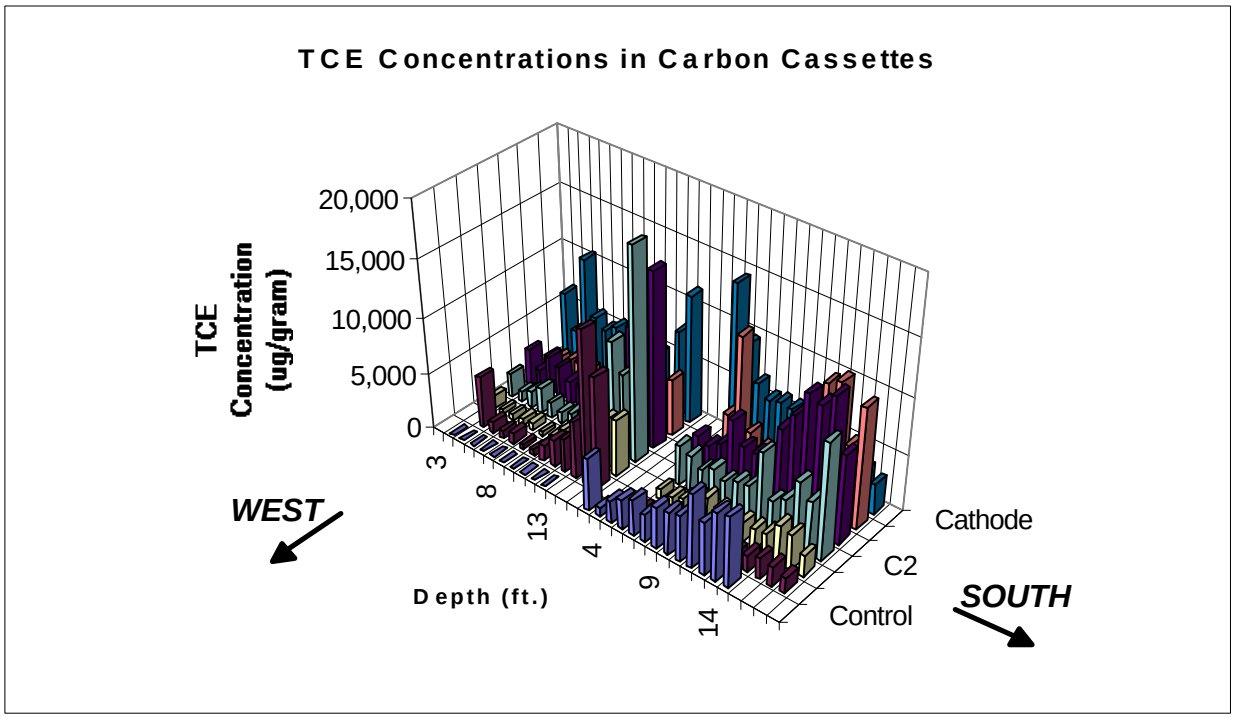

Figure F-9. Spatial Concentrations of TCE Measured in all Carbon Cassettes after 3 Pore Volumes.

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*™ 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*™ 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*™ 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.

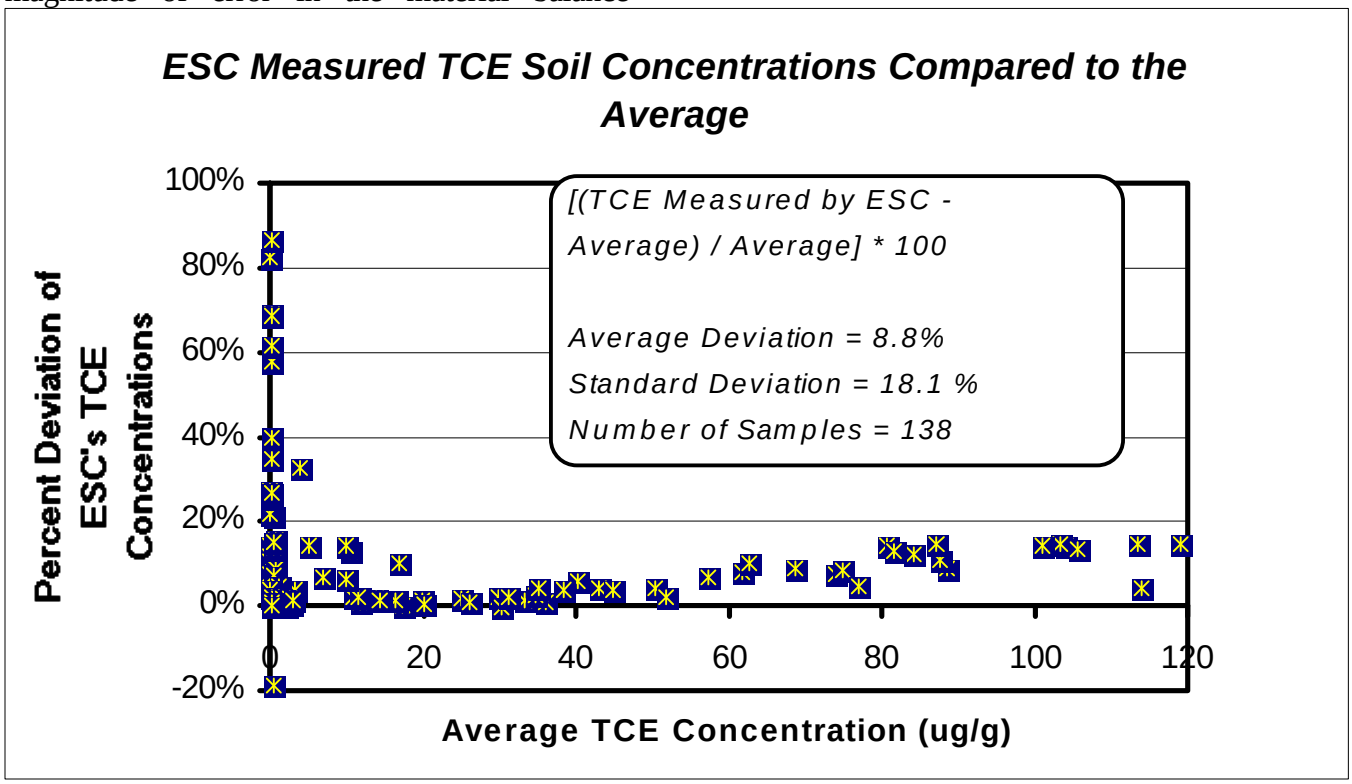

Figure F-10. Deviations of ESC TCE Concentrations from the Average.

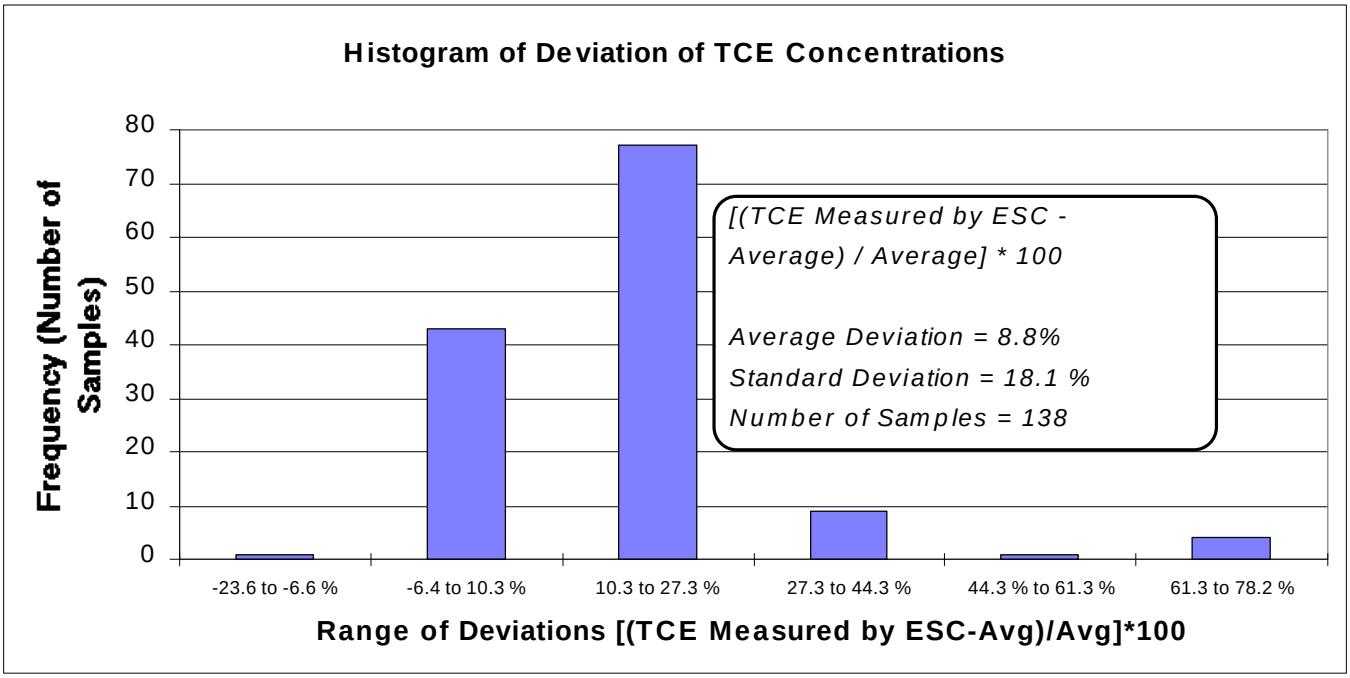

Figure F-11. Histogram of the Frequency Distribution of Deviations in ESC TCE Concentrations from the Average.

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.

TCE_{total} = (soil or carbon weight) x TCE_{concen}

TCE_{total} = volume x density x $TCE_{concen.}$ (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 cm^2 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 cm^2 x 53.3 cm) by the density of soil (2.0 g/cm^3) 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 cm^2 x 2.54 cm) by the density of dry carbon (0.5 g/cm^3) 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*™ 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 cm^2 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 cm^2 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*™ can be used to transfer TCE from contaminated soil into reaction zones. This fact will be exploited in the next phase of *Lasagna*™ process development for the Department of Energy.

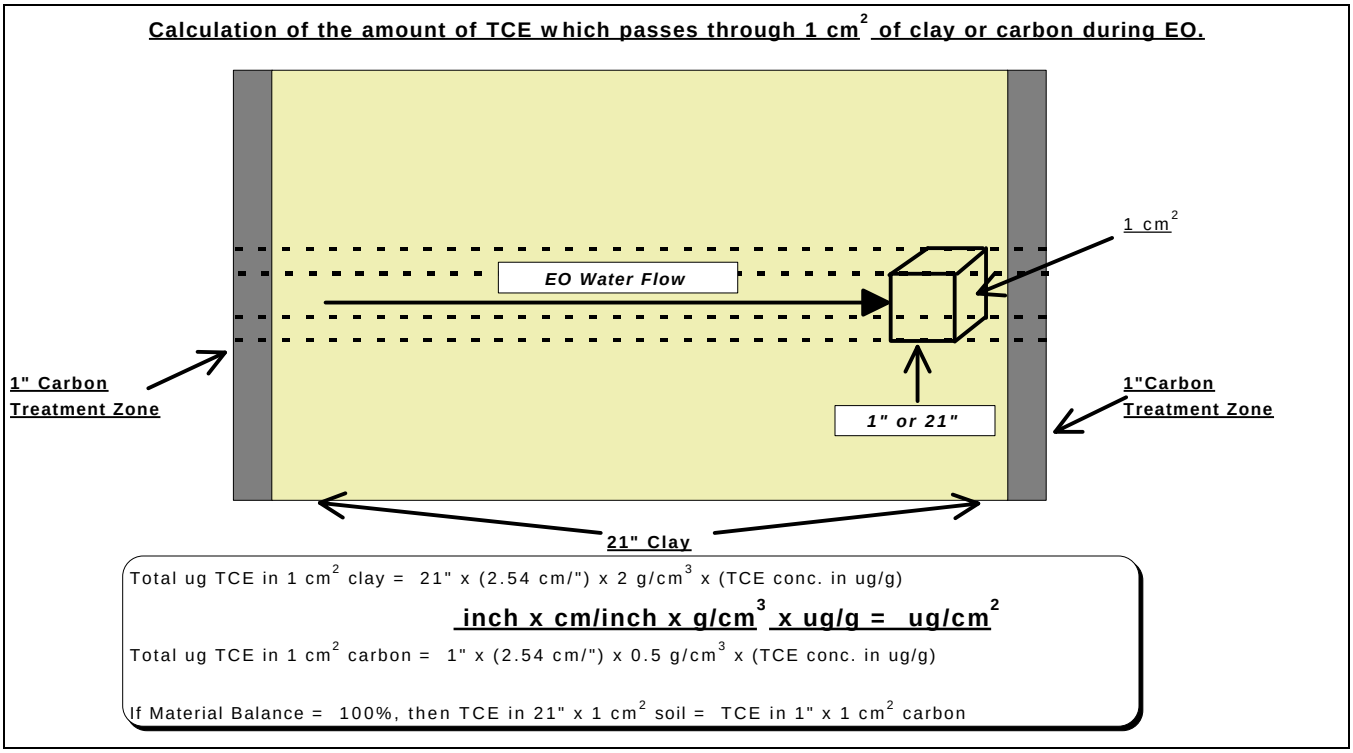

Figure F-12. Diagram of Material Balance Calculation.

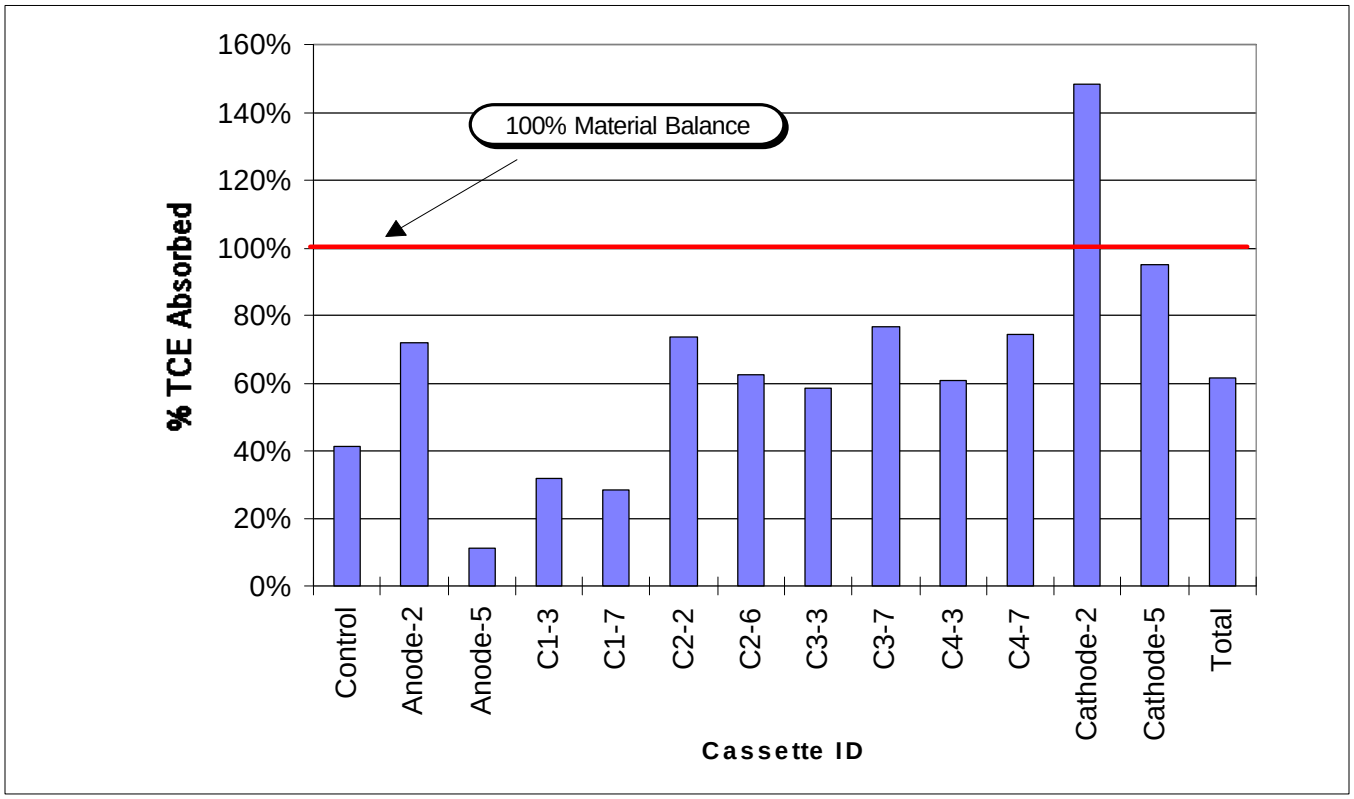

Figure F-13. Results of Material Balance Calculations using Discrete Soil Borings.

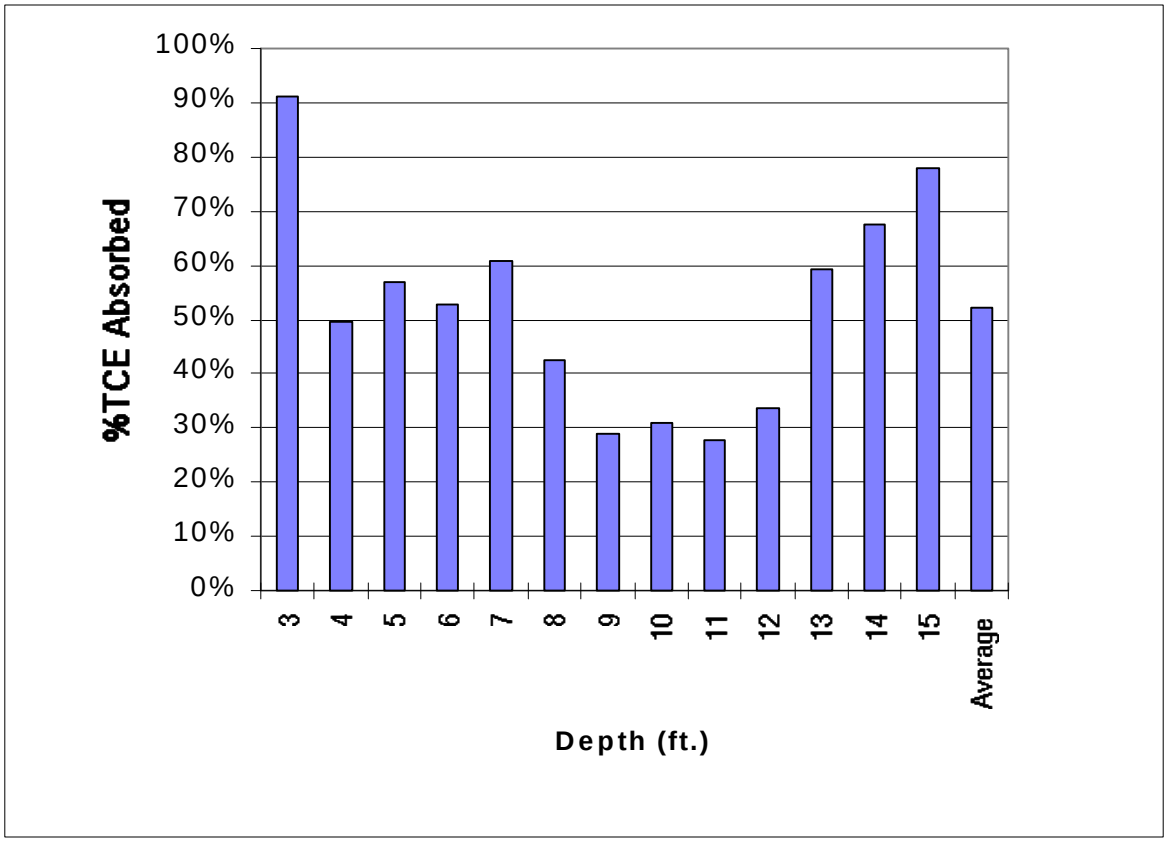

Figure F-14. Results of Material Balance Calculations using Kriged Data From Appendix G.

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-dichloethylene 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 in Appendix L. Appendix J summarizes these results.

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 analyis data shown in Appendix K includes the analysis of soil borings taken outside of, and below the present *Lasagna*™ 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*™ 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*™ 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*™ 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*™ 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

Appendix B. ORNL-Grand Junction Task Reports

Appendix C. Carbon Compositing and Sampling SOP

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

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

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			
[Control]	4,575.30	**4,652.82**	**109.64**	**2.36%**
[Control]	6,691.18			
[Control]	5,950.85	**6,321.02**	**523.50**	**8.28%**
[Anode-2]	4,897.98			
[Anode-2]	4,335.73	**4,616.85**	**397.57**	**8.61%**
[Anode-2]	10,740.10			
[Anode-2]	9,204.72	**9,972.41**	**1,085.68**	**10.89%**
[Anode-5]	301.55			
[Anode-5]	247.59	**304.33**	**80.23**	**26.36%**
[Anode-5]	1,416.50			
[Anode-5]	1,208.19	**1,312.34**	**147.30**	**11.22%**
{C1-3}	1,370.67			
{C1-3}	1,025.07	**1,197.87**	**244.37**	**20.40%**
[C1-3]	5,605.60			
[C1-3]	4,471.34	**5,038.47**	**802.04**	**15.92%**
[C1-7]	1,035.16			
[C1-7]	752.16	**893.66**	**200.11**	**22.39%**
[C1-7]	1,789.65			
[C1-7]	1,876.60	**1,833.13**	**61.48**	**3.35%**
[C2-2]	2,396.30			
[C2-2]	1,890.75	**2,143.52**	**357.48**	**16.68%**
[C2-2]	14,639.14			
[C2-2]	22,774.15	**18,706.64**	**5,752.32**	**30.75%**
[C2-6]	3,896.20			
[C2-6]	3,224.02	**3,560.11**	**475.31**	**13.35%**
[C3-3]	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	**15,618.97**	**347.78**	**2.23%**
[C3-7]	3,601.16			
[C3-7]	2,969.49	**3,285.32**	**446.66**	**13.60%**
[C3-7]	8,953.98			
[C3-7]	6,855.76	**7,904.87**	**1,483.67**	**18.77%**
[C4-3]	1,636.25			
[C4-3]	1,599.80	**1,618.02**	**25.78**	**1.59%**
[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			
{C4-7}	34.66	**35.79**	**1.59**	**4.45%**
{C4-7}	614.51			
{C4-7}	516.13	**565.32**	**69.57**	**12.31%**
[Cathode-2]	7,690.13			
[Cathode-2]	5,146.54	**6,418.34**	**1,798.59**	**28.02%**
[Cathode-2]	10,328.41			
[Cathode-2]	12,496.25	**11,412.33**	**1,532.90**	**13.43%**
[Cathode-5]	13,745.27			
[Cathode-5]	14,873.15	**14,309.21**	**797.53**	**5.57%**
[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*™ 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*™ 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*™ 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*™ 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 summaries 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, <u>total TCE mass recovered for each interval ranged from 30% to 90%.</u> In some instances, percent mass recovered exceed 100% primarily due to underestimation of kriged variable.

Table 2 summaries 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, <u>'corrected' kriged TCE concentrations represent a more realistic estimate of interpolated location concentrations because of error-weight method accounts for the boundaries of uncertainty</u> 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.

Appendix H. Comparison of ORNL-Grand Junction and ESC Extract Analysis for Post-*Lasagna*™ 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%**	**Average =**	**8.80%**
Std. Dev. =	**36.25%**	**Std. Dev. =**	**18.13%**
Number =	**138**	**Number =**	**138**

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

Appendix J. Chlorinated 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.

		Water Concentration (μg/L or ppb)		
Well ID	**Depth (ft.)**	**vinyl chloride**	***cis*-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 Used by LMES-PGDP