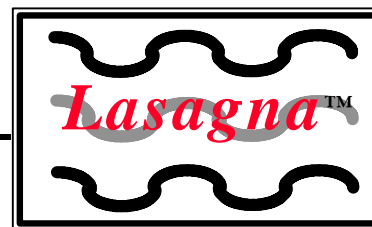


# Monsanto

---



## Rapid Commercialization Initiative (RCI) Final Report for an Integrated *in-situ* Remediation Technology (*Lasagna*™)

In response to: Rapid Commercialization Initiative (RCI)

### FINAL REPORT of the RCI Participants

Submitted to: U.S. Department of Energy  
Morgantown Energy Technology Center  
ATTN: RCI Project Team, MS 107/DDR  
P.O. Box 880  
Morgantown, WV 26507-0880

Submitted by: Monsanto Company  
800 N. Lindbergh Boulevard  
St. Louis, Missouri 63167

March 2, 1998

March 2, 1998

# Rapid Commercialization Initiative (RCI) Final Report for an Integrated *in-situ* Remediation Technology (*Lasagna*<sup>™</sup>)

In response to: Rapid Commercialization Initiative (RCI)

## FINAL REPORT of the RCI Participants

Submitted by: Monsanto Company  
800 N. Lindbergh Boulevard  
St. Louis, Missouri 63167

*Abstract: Contamination in low permeability soils poses a significant technical challenge to remediation efforts. These soils cause poor accessibility to the contaminants and difficulty in delivery of treatment reagents that decrease the effectiveness of existing treatments such as bioremediation, vapor extraction, and pump and treat. The present report describes verification of a new integrated in-situ treatment technology that Monsanto Company hopes to commercialize. This technology uses established geotechnical methods to install degradation zones directly in the contaminated soil and electro-osmosis to move the contaminants back and forth through these zones until the treatment is completed. Conceptually, this integrated technology could treat organic and inorganic contamination, as well as mixed wastes. Once developed, the technology may have significant benefits over existing ones in many aspects including environmental impacts, cost effectiveness, waste generation, treatment flexibility and breadth of applications. A consortium of industry and federal agencies has been formed to combine expertise and resources for accelerating the development of this technology. A previous study (Phase I) conducted in 1995, successfully demonstrated the coupling of electro-osmosis with in-situ adsorption zones. That study was neither audited nor was the data reviewed by outside entities to the extent the present study was reviewed.*

*The present Final Report for the Rapid Commercialization Initiative program uses the continued development of this technology to produce verification information for the RCI Participants. This Phase IIa study was conducted at DOE's Paducah Gaseous Diffusion Plant in Paducah, KY. The contaminant targeted in this project is trichloroethylene (TCE). TCE is a common contaminant found in the nation's soil and groundwater, and is present at 60% of DOE sites. The present report discusses the execution of the Quality Assurance Project Plan (QAPP) and describes the findings of the RCI Participants. This final report forms the basis of the Verification Statement (shown in Appendix A) which will be either signed or concurred with by the various Participants who are among the co-authors of this report. Based upon this Phase IIa data, Phase IIb, which involves the cleanup of the entire area of contamination, will likely proceed.*

## B. Table of Contents

<u>(SECTION TITLE)</u>	<u>(PAGE)</u>
<b>A. TITLE PAGE .....</b>	<b>A-1</b>
<b>B. TABLE OF CONTENTS .....</b>	<b>B-1</b>
List of Tables .....	B-3
List of Figures.....	B-5
<b>C. TECHNICAL SUMMARY .....</b>	<b>C-1</b>
Statement of the Problem.....	C-1
General Approach.....	C-2
Consortium Description .....	C-3
Objective .....	C-3
Project Description .....	C-5
<i>Phase I Results</i> .....	C-5
<i>Phase II Field Study</i> .....	C-6
<i>Site Description and Contamination</i> .....	C-7
<b>D. PROJECT ORGANIZATION AND RESPONSIBILITY .....</b>	<b>D-1</b>
Consortium Organization .....	D-1
RCI Organization.....	D-2
<i>Three Major Teams</i> .....	D-2
<i>Phase IIb Final Cleanup Team</i> .....	D-2
<i>RCI Process and Cost Validation Team</i> .....	D-2
<i>Phase IIa Test Cleanup Team</i> .....	D-2
<i>Discussion of Figure D-3</i> .....	D-2
<i>QA Functions</i> .....	D-2
<i>Personnel</i> .....	D-3
<i>Paducah Department of Energy (DOE) Project Manager - Myrna Redfield</i> .....	D-3
<i>LMES, Environmental Restoration (ER), Program Manager - Fraser Johnstone</i> .....	D-3
<i>LMES, ER, Technical Oversight Coordinator - Jay Clausen (currently Jeff Douthitt)</i> .....	D-3
<i>LMES, ER, Quality Assurance (QA) Specialist - Jennifer Blewett</i> .....	D-3
<i>CDMFPC Federal Analytical Laboratory</i> .....	D-3
<i>ORNL/GJ Analytical Laboratory</i> .....	D-3
<i>LMUS Analytical Laboratory</i> .....	D-3
<i>Oak Ridge DOE Project Manager - Leon Duquella</i> .....	D-4
<i>MSE/Cost Verifier - Stephen Antonioli</i> .....	D-4
<i>RCI/DOE Verification Entity - David Carden</i> .....	D-4
<i>RCI/EPA Representative - Sam Hayes</i> .....	D-4
<i>Third-Party Laboratory</i> .....	D-4
<i>Data Validator</i> .....	D-4
<b>E. COMPARISON OF ACTUAL ACTIVITIES TO THOSE PLANNED IN THE QAPP.....</b>	<b>E-1</b>
Phase II Cleanup Levels .....	E-1
Original Phase IIa Field Experiment Cleanup Levels.....	E-1
Actual Cleanup Levels .....	E-1
Comparison with Original QAPP.....	E-2

<b>(SECTION TITLE)</b>	<b>(PAGE)</b>
<b>F. SAMPLING AND ANALYSIS RESULTS .....</b>	<b>F-1</b>
Sampling Plan .....	F-1
Quality Assurance and Quality Control and Data Usability .....	F-1
Sampling Results.....	F-1
Soil Sample Results.....	F-4
Search for Chlorinated Degradation Products.....	F-10
Water Sample Results .....	F-11
<i>TCE Pore Water Concentrations.....</i>	<i>F-11</i>
<i>TCE Concentrations in Wells 03 - 05.....</i>	<i>F-11</i>
Carbon Sample Results .....	F-14
Flux Chamber Results .....	F-14
Acetylene, Ethane, and Ethylene Degradation Products.....	F-14
Dechlorination of TCE in the Phase IIa Field Test.....	F-16
<i>Introduction.....</i>	<i>F-16</i>
<i>Rate of Dechlorination .....</i>	<i>F-17</i>
<i>Design of treatment zones.....</i>	<i>F-17</i>
<i>Installation &amp; Operation.....</i>	<i>F-19</i>
<i>Dechlorination Results .....</i>	<i>F-19</i>
<b>G. TREATMENT EFFECTIVENESS VERIFICATION.....</b>	<b>G-19</b>
1.0 Introduction.....	G-1
1.1 <i>Objective .....</i>	<i>G-1</i>
1.2 <i>Quality Assurance Project Plan.....</i>	<i>G-1</i>
2.0 Assessment Approach .....	G-1
3.0 Results.....	G-1
3.1 <i>Field Sample Collection Assessment.....</i>	<i>G-2</i>
3.1.1 <i>Sampling Technique Evaluation.....</i>	<i>G-2</i>
3.1.2 <i>Control of Cross Contamination.....</i>	<i>G-2</i>
3.1.3 <i>Field Precision .....</i>	<i>G-2</i>
3.2 <i>Analytical Laboratory Operations Assessment .....</i>	<i>G-5</i>
3.2.1 <i>Analytical Laboratory Technique Evaluation.....</i>	<i>G-6</i>
3.2.2 <i>Laboratory Proficiency Evaluation.....</i>	<i>G-6</i>
3.2.3 <i>Reference Laboratory Data Validation.....</i>	<i>G-6</i>
3.2.4 <i>Accuracy and Precision of Analytical Data.....</i>	<i>G-7</i>
3.2.4.1 <i>Interlaboratory Comparisons .....</i>	<i>G-7</i>
3.2.4.2 <i>Intralaboratory Comparisons .....</i>	<i>G-13</i>
3.2.5 <i>Analytical Method Quality Control for Soil Analysis.....</i>	<i>G-15</i>
3.3 <i>Rollup Assessment and Statistical Evaluation of Data Quality Objectives.....</i>	<i>G-17</i>
3.3.1 <i>Data Quality Issues and Limitations .....</i>	<i>G-17</i>
3.3.2 <i>Evaluation of Trends.....</i>	<i>G-17</i>
3.3.2.1 <i>Treatment Effectiveness .....</i>	<i>G-17</i>
3.3.2.2 <i>Progress in Meeting Action Levels .....</i>	<i>G-18</i>
4.0 Conclusions .....	G-18

<b><u>(SECTION TITLE)</u></b>	<b><u>(PAGE)</u></b>
<b>H. SUMMARY OF INSTALLATION COSTS, LASAGNA™ PHASE IIA TEST.....</b>	<b>H-1</b>
Summary .....	H-1
Introduction.....	H-2
Phase Iia Installation Costs .....	H-2
Phase Iia Test Operating and Maintenance Costs .....	H-5
Discussion of Phase Iia Costs.....	H-6
Simple Core Cost Estimates For A Full Scale Lasagna™ Remediation Based on Depth, Time, and Pore Volumes Required .....	H-7
<b>I. REFERENCES, MERIT OF THE TECHNOLOGY, ABBREVIATIONS AND     DEFINITIONS .....</b>	<b>I-1</b>
Merit of the Technology.....	I-2
<i>Lasagna™ Technology</i> .....	<i>I-2</i>
Abbreviations.....	I-2
Electro-Kinetics .....	I-3
Electro-Osmosis.....	I-3
<i>References</i> .....	<i>I-5</i>
<b>J. CONCLUSIONS .....</b>	<b>J-1</b>
<b>APPENDIX A. VERIFICATION STATEMENT .....</b>	<b>APPENDIX A-1</b>
<b>APPENDIX B. PRE-TEST CONCENTRATIONS OBTAINED     IN MARCH 1996 .....</b>	<b>APPENDIX B-1</b>
<b>APPENDIX C. SECTIONS E - G IN QAPP .....</b>	<b>APPENDIX C-1</b>
<b>APPENDIX D. TABULAR DATA PLOTTED IN THE FIGURES OF SECTIONS     F AND G.....</b>	<b>APPENDIX D-1</b>
<b>APPENDIX E. QA AUDITS.....</b>	<b>APPENDIX E-1</b>
<b>APPENDIX F. MEASUREMENT OF C<sub>2</sub> HYDROCARBONS IN WELL WATER     SAMPLES .....</b>	<b>APPENDIX F-1</b>
Background .....	APPENDIX F-1

### List of Tables

<b><u>(TABLE CAPTION)</u></b>	<b><u>(PAGE)</u></b>
Table C-1. Paducah Soil Characteristics.....	C-9
Table F-1. Summary of Field Operations for the Phase Iia Study.....	F-2
Table F-2. Summary of Water Samples Actually Analyzed.....	F-3
Table F-3. Summary of Carbon Samples, Splits, and Duplicates Actually Analyzed.....	F-3
Table F-4. Summary of Soil Samples, Duplicates and Splits Actually Analyzed. ....	F-4
Table F-5. Summary of Pre- and Post-Lasagna™ Soil Samples for L2A-01 and L2A-02 Soil Cores Analyzed by ORNL/GJ (GC/EC).....	F-5

<b>(TABLE CAPTION)</b>	<b>(PAGE)</b>
Table F-6. Summary of Pre-, Inter-, and Post-Lasagna™ Soil Samples for L2A-03 Soil Cores Analyzed by ORNL/GJ (GC/EC).....	F-6
Table F-7. Summary of Pre-, Inter, and Post-Lasagna™ Soil Samples for L2A-04 and L2A-05 Soil Cores Analyzed by ORNL/GJ (GC/EC).....	F-7
Table F-8. Summary of TCE, DCE, and VC Concentrations Measured in Post-Lasagna™ Soil Samples Analyzed by Weston (GC/PID).....	F-10
Table F-9. Distribution of TCE in Water and on Soil, Using the Langmuir Isotherm Data from Phase I.....	F-11
Table F-10. Summary of TCE Concentrations Measured December 1996 Flux Chamber Measurements. ....	F-14
Table F-11. Summary of C <sub>2</sub> Hydrocarbon Degradation Products Measured in Water Samples. ....	F-15
Table F-12. Summary of Half-Lives of Chlorinated C <sub>2</sub> Degradation Products Measured in Water Samples.....	F-18
Table F-13. Summary of % Conversion of Chlorinated C <sub>2</sub> Degradation Products Measured in Water Samples. ....	F-18
Table F-14. Summary of ppmw (µg/g) of Chlorinated C <sub>2</sub> Degradation Products Measured in Water Samples. ....	F-18
Table G-1. Data Quality Issues and Impacts Upon the Lasagna™ Phase IIa Technology Demonstration .....	G-17
Table G-2. Lasagna™ Treatment Effectiveness -Statistical Data Summary for TCE in Soil for the Zone of Contamination .....	G-18
Table G-3. Lasagna™ Process - Compliance with Cleanup Target - Statistical Data Summary for TCE in Soil .....	G-19
Table H-1. Example of an estimated cost breakdown for a full-scale Lasagna™ remediation, 45 feet deep, with 4 pore volumes required. Time to remediation is 3 years. Total volume is 10,000 yd <sup>3</sup> .....	H-1
Table H-2. Cost Summary for Phase IIa Installation by Contractor and Cost Element.....	H-3
Table H-3. Cost Summary for Phase IIa Installation by Cost Per Unit.....	H-3
Table H-4. Core and Non-Core Costs for Electrode and Treatment Zones .....	H-4
Table H-5. An Estimate of Installation Costs for a Full-Scale Lasagna™ Remediation. <sup>1</sup> .....	H-4
Table H-6. Cost Summary for Phase IIa Operations and Maintenance. ....	H-6
Table H-7. Cost Summary for Phase IIa Test.....	H-6
Table H-8. Estimated Lasagna™ Remediation Core Costs/yd <sup>3</sup> as a Function of Depth, Pore Volumes Required, and Years Allowed for Remediation .....	H-8
Table I-1. Comparison of Electro-Osmotic Permeability and Hydraulic Conductivity for Various Soils <b>(Adapted from J. Mitchell, (1))</b> .....	I-4
Table I-2. Practical Ranges for Electro-osmotic Flushing in Fine-Grained Soils <b>(adapted from Mitchell (13) &amp; Probstein (14))</b> .....	I-5
Table Appendix B-1. Pre-Lasagna™ Soil TCE Concentrations .....	APPENDIX B-3
Table Appendix D-1. Table for Data Shown in Figure G-6.....	APPENDIX D-1
Table Appendix D-2. Table for Data Shown in Figure G-7.....	APPENDIX D-1
Table Appendix D-3. Table for Data Shown in Figure G-1a.....	APPENDIX D-2
Table Appendix D-4. Table for Data Shown in Figure G-1b.....	APPENDIX D-2
Table Appendix D-5. Table for Data Shown in Figure G-1c.....	APPENDIX D-2
Table Appendix D-5. Table for Data Shown in Figure G-2.....	APPENDIX D-3
Table Appendix D-6. Table for Data Shown in Figure G-5a.....	APPENDIX D-4
Table Appendix D-7. Table for Data Shown in Figure G-5b.....	APPENDIX D-4
Table Appendix D-8. Table for Data Shown in Figure G-8.....	APPENDIX D-5
Table Appendix D-9. Table for Data Shown in Figures F-3 and F-4 taken 8/8/96 - 1/10/97.....	APPENDIX D-6
Table Appendix D-10. Table for Data Shown in Figures F-3 and F-4 taken 2/12/97 - 8/6/97.....	APPENDIX D-7
Table Appendix D-11. Calibration Checks for March 1997 Soil Samples.....	APPENDIX D-8
Table Appendix D-12. Laboratory Duplicates for March 1997 Soil Samples Shown in Figure G-11 .....	APPENDIX D-9
Table Appendix D-13. Field Duplicates for March 1997 Soil Samples .....	APPENDIX D-9
Table Appendix D-14. Equipment Rinsates for March 1997 Soil Samples.....	APPENDIX D-10
Table Appendix D-15. Comparison of Passive vs. Sonic Extraction for March 1997 Soil Samples .....	APPENDIX D-10
Table Appendix E-1. Rapid Commercialization Initiative Lasagna™ Project at Paducah Gaseous Diffusion Plant Independent Verification Assessments .....	APPENDIX E-1
Table Appendix E-2. Field Blank Results Lasagna™ Phase IIa Field Sampling .....	APPENDIX E-5

<b>(TABLE CAPTION)</b>	<b>(PAGE)</b>
Table Appendix E-3a. Lasagna™ RCI Program Reference Laboratory Data Validation Results .....	APPENDIX E-6
Table Appendix E-3b. Lasagna™ RCI Program Reference Laboratory Data Validation Results .....	APPENDIX E-7
Table Appendix E-3c. Lasagna™ RCI Program Reference Laboratory Data Validation Results .....	APPENDIX E-8
Table Appendix E-4. Laboratory Hexane Blanks - ORNL/GJ Field Laboratory.....	APPENDIX E-9
Table Appendix E-5. ORNL/GJ Field Laboratory Soil Analysis by Gas Chromatography Instrument Calibration Verification.....	APPENDIX E-10

## List of Figures

<b>(FIGURE CAPTION)</b>	<b>(PAGE)</b>
Figure C-1. Schematic Diagram of the Lasagna™ Technology.....	C-4
Figure C-2. Integrated in-situ Remediation .....	C-5
Figure C-3. Average TCE Concentrations Pre- and Post-Lasagna™ for the Phase I Field Experiment.....	C-6
Figure C-4. Locations of Sampling and Monitoring Sites in the Phase IIA Field Experiments.....	C-8
Figure C-5. Contours of the Highest Measured TCE Concentrations .....	C-9
Figure D-1. Organizational Structure of the Lasagna™ Technology Consortium.....	D-1
Figure D-2. Organizational Structure of the Industrial Consortium for the Phase II Study .....	D-2
Figure D-3. Organizational Chart for QA Oversight of Lasagna™ Phase IIA Field Study .....	D-5
Figure F-1. Summary of Pre-, and Post-Lasagna™ Soil Samples for L2A-01 and L2A-02 Soil Cores Analyzed by ORNL/GJ (GC/EC).....	F-8
Figure F-2. Summary of Pre-, Inter, and Post-Lasagna™ Soil Samples for L2A-03, L2A-04 and L2A-05 Soil Cores Analyzed by ORNL/GJ (GC/EC).....	F-9
Figure F-3. Summary of TCE Concentrations Measured in Wells 03, and 04 Analyzed by (GC/EC).....	F-12
Figure F-4. Summary of TCE Concentrations Measured in Well 05 Analyzed by (GC/EC).....	F-13
Figure F-5. Summary of C <sub>2</sub> Hydrocarbon Degradation Products Measured in Water Samples.....	F-16
Figure G-1a. Field Duplicates - ORNL/GJ Soil Sampling - March 1996.....	G-3
Figure G-1b. Field Duplicates - ORNL/GJ Soil Sampling - March 1997.....	G-4
Figure G-1c. Field Duplicates - ORNL/GJ Soil Sampling - August 1997.....	G-4
Figure G-2. Field Duplicates - Carbon Cassettes .....	G-5
Figure G-3. Summary of TCE Concentrations Measured by Various Laboratories for a Blind QA Sample LA59417.....	G-8
Figure G-4a. Laboratory Intercomparison - TCE in Groundwater Samples - CDMFPC vs. Weston .....	G-9
Figure G-4b. Laboratory Intercomparison - TCE in Groundwater Samples - LMUS vs. Weston .....	G-10
Figure G-5a. Interlaboratory Comparison - Soil Analysis by GC-ECD - March 1997 - ORNL/GJ vs. Weston.....	G-10
Figure G-5b - Interlaboratory Comparison - Soil Extract Analysis by ORNL/GJ and Weston (EC Detector) - August 1997 .....	G-11
Figure G-6. Interlaboratory Comparison - Soil Analysis by ORNL/GJ (EC Detector) and LMUS (P&T) - March 1997.....	G-12
Figure G-7. Interlaboratory Comparisons - Soil Analysis by ORNL/GJ (EC Detector) and Weston (P&T) - August 1997.....	G-12
Figure G-8. Interlaboratory Comparisons - Carbon Cassette Analysis by ORNL/GJ and Weston .....	G-13
Figure G-9. Intralaboratory Comparison - TCE in Groundwater - CDMFPC Field Laboratory .....	G-14
Figure G-10. Intralaboratory Comparison - TCE in Groundwater - LMUS Laboratory .....	G-15
Figure G-11. Duplicate Analysis of the Same Soil Extract - ORNL/GJ Field Laboratory .....	G-15
Figure G-12. Soil Sample Extraction Technique Evaluation for TCE ORNL/GJ Method vs. Sonication Method.....	G-16
Figure H-1. Plotting of the Study Values Listed in Table H-7.....	H-8
Figure Appendix F-1. GC/MS Chromatogram of C <sub>2</sub> Degradation Products When TCE is Present Below the Solubility Limit (280 ppmw (µg/g) TCE in Water).....	APPENDIX F-2
Figure Appendix F-2. GC/MS Chromatogram of C <sub>2</sub> Degradation Products When TCE is Present as DNAPL (Prepared With 5,800 ppmw (µg/g) TCE in Water).....	APPENDIX F-3
Figure Appendix F-3. Degradation Products When TCE is DNAPL or Dissolved.....	APPENDIX F-3

---

**Integrated *in-situ* remediation technologies will greatly reduce cleanup costs for remediation of contaminated sites, produce less disruption to the environment, and minimize worker exposure to hazardous materials.**

---

### Statement of the Problem

During the last decade a great deal of research has been conducted to develop *in-situ* technologies for treating contaminated soils and groundwater. *In-situ* methods are attractive because of the potential lower cost, less disruption to the environment, and reduced worker exposure to hazardous materials. However, promising treatments such as bioremediation, vapor extraction, air sparging, and pump and treat, have been found rather ineffective when applied to low permeability soils present at many contaminated sites.

In recent years, electro-osmosis (movement of water through soil matrices induced by an applied electric field — see Section I for a more detailed discussion), has received increasing attention as an *in-situ* method for cleaning up contamination in low-permeability soils<sup>1,2,3,4,5,6</sup>. Electro-osmosis is a classical civil engineering method that has been used since the 1930s for dewatering clays, silts, and fine sands<sup>7</sup>. In the new application, water introduced into

the soil at the anode region would flow under electro-osmosis through the contaminated soil, flushing the contaminants to the cathode area for further treatment or disposal aboveground. Major advantages of electro-osmosis include uniform flow distribution in a heterogeneous soil because the flow is independent of pore size; controllable flow direction by the placement of the electrodes and the voltages applied; and low power consumption<sup>5,8</sup>. During electro-osmosis, however, there are a large number of chemical and electrochemical processes occurring that can negatively affect the electro-osmotic process and the effectiveness of the intended remediation. These include ion exchange, development of osmotic and pH gradients, soil drying, precipitation of salts and secondary minerals, electrolysis, soil fabric changes, etc.<sup>3-6, 8-9</sup>. Due to these complications and because it is not a destructive method, electro-osmosis *alone* does not appear to provide a practical and complete treatment technology.

A consortium of industrial companies and government agencies are currently developing a novel, *in-situ* technology aimed at cleaning up contamination in low-permeability soils. The approach, called “integrated *in-situ* remediation technology”<sup>10</sup> (designated *Lasagna*<sup>TM</sup>)<sup>11</sup>, consists of technical components discussed in the following sections.

---

<sup>1</sup> Shapiro, A.P., Renauld, P., and Probstein, R. “*Preliminary Studies on the Removal of Chemical Species from Saturated Porous Media by Electro-osmosis*” *Physicochemical Hydrodynamics*, Vol. 11, No. 5/6, pp. 785-802 (1989).

<sup>2</sup> Hamed, J., Acar, Y.B., and Gale, R.J. “*Pb(II) Removal from Kaolinite Using Electro-kinetics*” *J. Geotech. Eng., ASCE*, Vol. 112, pp. 241-271, February (1991).

<sup>3</sup> Bruell, C. J. and Segall, B. A. “*Electro-osmotic Removal of Gasoline Hydrocarbons and TCE from Clay*” *J. Environ. Eng.*, Vol. 118, No. 1, pp 68-83, Jan/Feb 1992.

<sup>4</sup> Segall, B. A. and Bruell, C. J. “*Electro-osmotic Contaminant Removal Processes*” *J. Environ. Eng.*, Vol. 118, No. 1, pp 84-100, Jan/Feb 1992.

<sup>5</sup> Acar, Y.B., Li, H., and Gale, R.J. “*Phenol Removal from Kaolinite by Electro-kinetics*” *J. Geotech. Eng., ASCE*, Vol. 118, No. 11, pp. 1837-1852, November 1992.

<sup>6</sup> Shapiro, A.P., and Probstein, R.F. “*Removal of Contaminants from Saturated Clay by Electro-Osmosis*” *Environ. Sci. Technol.* 27, pp. 283-291, 1993.

---

<sup>7</sup> Casagrande, L. “*Electro-osmotic Stabilization of Soils*” *J. BSCE*, 39:51-83 (1952).

<sup>8</sup> Shapiro, A.P. 1990. “*Electro-osmotic Purging of Contaminants from Saturated Soils.*” Ph.D. Thesis, Department of Mechanical Engineering, M.I.T., Cambridge, Mass.

<sup>9</sup> Mitchell J.K.. *Potential Uses of Electro-kinetics for Hazardous Waste Site Remediation*, Position Paper prepared for USEPA-University of Washington *Workshop on Electro-kinetic Treatment and its Application in Environmental-Geotechnical Engineering for Hazardous Waste Site Remediation*, Seattle, WA, August 4-5 1986.

<sup>10</sup> Brodsky, P. H. and S. V. Ho. “*In-Situ Remediation of Contaminated Soils*” U. S. Patent 5,398,756, March 21, 1995.

<sup>11</sup> *Lasagna*<sup>TM</sup> is a trademark of Monsanto Company.



## General Approach

As described below, the *Lasagna*<sup>TM</sup> technology may be installed either in a horizontal or a vertical configuration and may be applicable to organic, inorganic, or mixed wastes. Mixed contamination of organics and heavy metals is very common, yet currently not treatable due to the incompatibility of methods required to treat these two types of contaminants. For instance, microorganisms can be effective for degrading organic contaminants, but are often inhibited by low levels of many metals. This complication can be effectively handled by the *Lasagna*<sup>TM</sup> process in many ways. One could use multiple treatment zones, some for trapping metals and some for degrading organics, or identical treatment zones containing mixed treatment materials to both trap metals and degrade the organics, or first immobilize the metals by adsorption/chelation then introduce microbes into the treatment zones to destroy the organics. Many other spatial and/or temporal variations are possible.

Another example of very difficult contamination involves highly nonpolar contaminants such as polyaromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs). Biodegradable surfactants can be used to mobilize these organics, probably in the form of micelles. If the surfactants are nonionic, the micelles should move with the electroosmotic flow from anode to cathode. If the surfactants are ionic, say, anionic, the corresponding micelles would migrate towards the anode. Either way, the contaminants transported along with the surfactants would be intercepted by the treatment zones and degraded. Surfactants are, however, costly if used only once and can severely interfere with microbial degradation activity. With *Lasagna*<sup>TM</sup>, the organics can be mobilized by a small amount of surfactant into treatment zones where they are trapped and the surfactant reused in place until all the contaminants are removed from the soil. Microbes can then be introduced into the treatment zones to degrade the organics. An even better approach is to use treatment zones as "delivery" zones to introduce microbes or treatment reagents by electrokinetics into the contaminated soil to destroy the organics where they reside without resorting to the use of surfactants.

For soils contaminated with heavy metals and radionuclides, the *Lasagna*<sup>TM</sup> technology could be used to concentrate these contaminants in narrow treatment zones in the soil for either physical recovery and disposal or immobilization in place. The former can be accomplished with cartridges that are designed to be pulled out from the soil once the process is completed. The latter can be accomplished by irreversible chemical reactions, or by in-place vitrification of the thin treatment zone strips. To facilitate vitrification, electrically conducting materials

could be mixed into the treatment zones during the emplacement.

*In situ* decontamination can occur if :

- **zones are created in close proximity to one another that are sectioned through the contaminated soil region, and turned into sorption/degradation zones by introducing appropriate materials (sorbents, catalytic agents, microbes, oxidants, buffers, etc.).** The vertical degradation zones can be placed using several approaches such as funnel-and-gate based on sheet piling, trenching or slurry walls. Hydraulic fracturing and related technologies may provide an effective and low-cost means for creating degradation zones horizontally in the subsurface soil.
- **electro-osmosis is utilized as a liquid pump for flushing contaminants from the soil into the treatment zones for degradation.** Since these zones are deliberately located near one another, the time taken for the liquid to move by electro-osmosis from one zone to the adjacent one can be short. In the horizontal configuration, the zones above and below the contaminated soil area can be injected with graphite particles during the hydrofracturing process to form the electrodes in place. For highly non-polar contaminants, surfactants can be introduced into the water or incorporated into the treatment zones to solubilize the organics.
- **liquid flow is reversed by switching electrical polarity to effect multiple passes of the contaminants through the treatment zones for complete sorption/destruction.** This step also serves to minimize complications associated with long-term application of one-directional electro-osmosis.

Whether the electrodes and the treatment zones are horizontal or vertical depends upon the site/contaminant characteristics. In general, the vertical configuration using trenching, sheet piling, etc., is probably more applicable to shallow contamination (within 50 ft {15.2 meters} or so from ground surface), whereas the horizontal configuration using hydrofracturing or related methods has the unique capability for handling deep contamination. Schematic diagrams of the two configurations are shown in Figure C-1. [The term *Lasagna*<sup>TM</sup> originates from the layered configuration of the electrodes and degradation zones.] This technology is potentially effective for treating organic or inorganic contamination, as well as mixed wastes.

The *Lasagna*<sup>TM</sup> technology provides a potential solution for remediation of organics in low permeability soils which is one of the technical challenges facing DOE. Removing the organics from the soils also removes the source of further groundwater contamination. Low permeability soils are found at many DOE sites. These sites include: Portsmouth Gaseous Diffusion Plant, Fernald Feed Materials Production Center, and Mound Plant in Ohio;

Argonne National Laboratory in Illinois; Oak Ridge National Laboratory and Facilities in Tennessee; Brookhaven National Laboratory in New Jersey; Paducah Gaseous Diffusion Plant in Kentucky; Savannah River Site in South Carolina; and Pinellas Plant in Florida.

## Consortium Description

A consortium has been formed consisting of Monsanto, E. I. du Pont de Nemours & Co., Inc. (DuPont) and General Electric (GE) (the "Consortium"), with participation from the Environmental Protection Agency (EPA) and the Department of Energy (DOE). The five members of this group are leaders in their represented technologies and hold significant patents and intellectual property that, in concert, form an integrated solution for remediation. The overall objective of the consortium is to sufficiently develop the integrated *in-situ* remediation technology so that it can be utilized for site remediation. Figure C-2 shows the major contributions of the participating members. In addition to providing site and support for field experiments of the *Lasagna*<sup>TM</sup> technology, DOE also contributes technically through a number of relevant on-going research programs.

## Objective

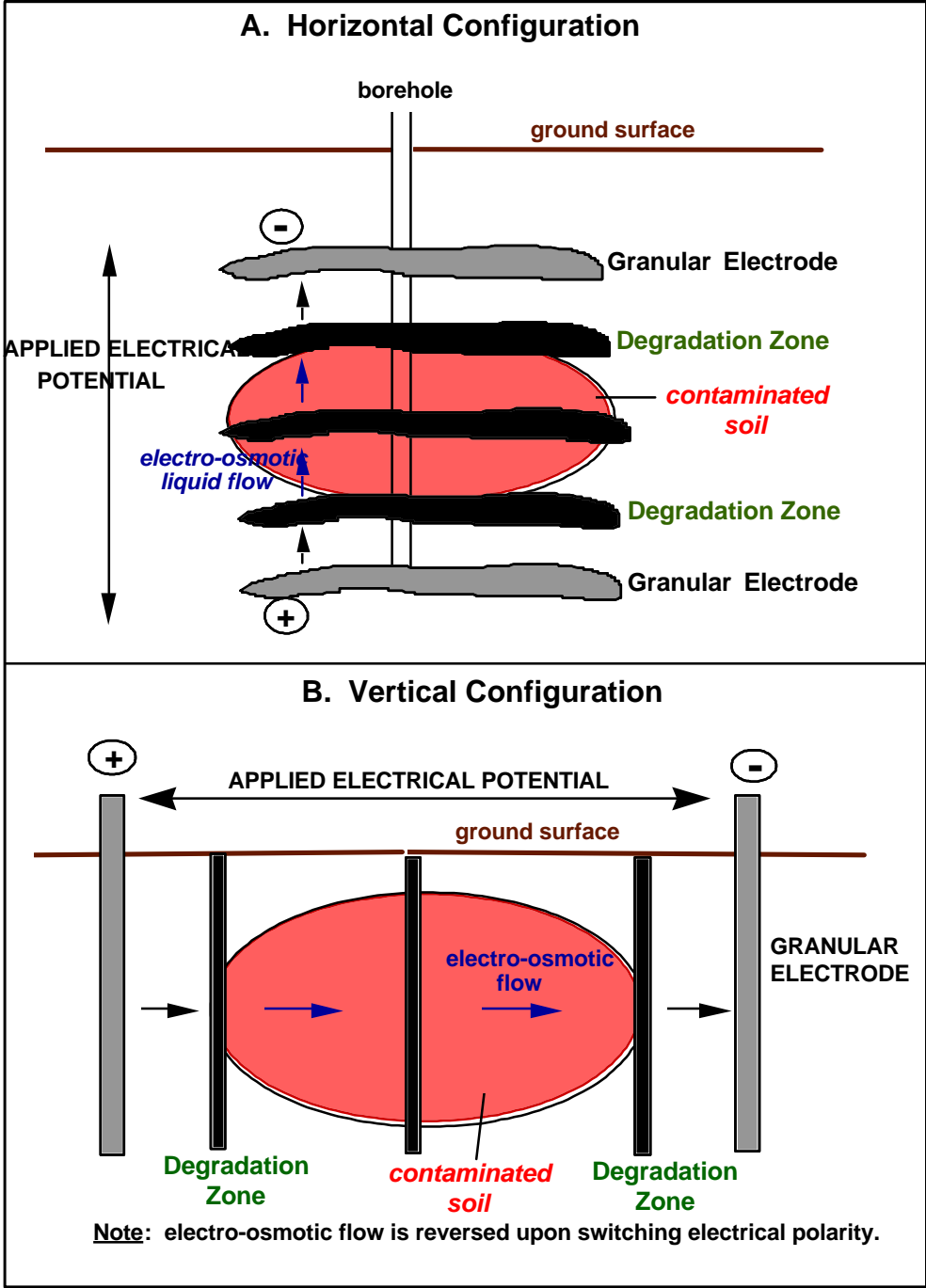
The primary objective of the present Rapid Commercialization Initiative (RCI) project for the Paducah Site is to verify the ability of the *Lasagna*<sup>TM</sup> technology to remove soil trichloroethene (TCE) to levels below the site specific action level (5.6 ppmw ( $\mu\text{g/g}$ )) set by the Commonwealth of Kentucky. This action level can be used for this site as long as ground water monitoring and modeling indicates that this level will not result in groundwater concentrations of 5 ppbw ( $\mu\text{g/L}$ ) TCE at the Plant's property line. A secondary objective is to document to what levels of cleanup and what destruction and removal efficiency (DRE) can be demonstrated in this test. These objectives were met by a four-step process:

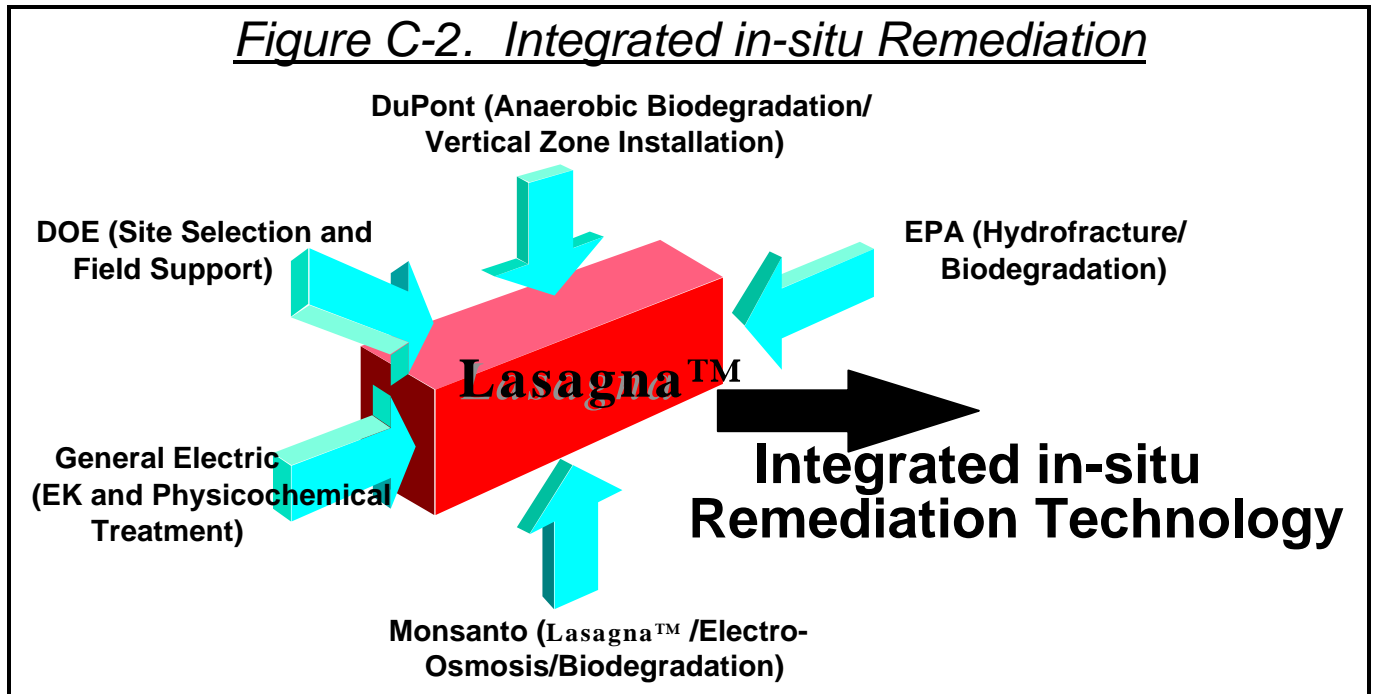
- (a) Ensuring that the sampling approach identified would provide data of sufficient quality and quantity to make decisions regarding treatment effectiveness at a high level of statistical confidence;
- (b) Independently verifying through onsite audit that the project field sampling and field analytical plans were implemented correctly;
- (c) Independently verifying project laboratory analytical results through analysis of split samples at a qualified independent laboratory;

(d) Completing a roll up assessment report that discusses the results of the verification activities, evaluates whether the data indicate success in meeting action levels,

and assesses any limitations of the data set in making decisions concerning project success.

**Figure C-1. Schematic Diagram of the Lasagna™ Technology**





## Project Description

On September 26, 1994 a contract was awarded to Monsanto for the Phase I evaluation of this integrated *in-situ* remediation technology. (Research was funded by the Morgantown Energy Technology Center under contract number DE-AR21-94MC31185.) Section I gives 10 references of reports that give details on the results of this study. This phase was completed successfully with accomplishments as follows.

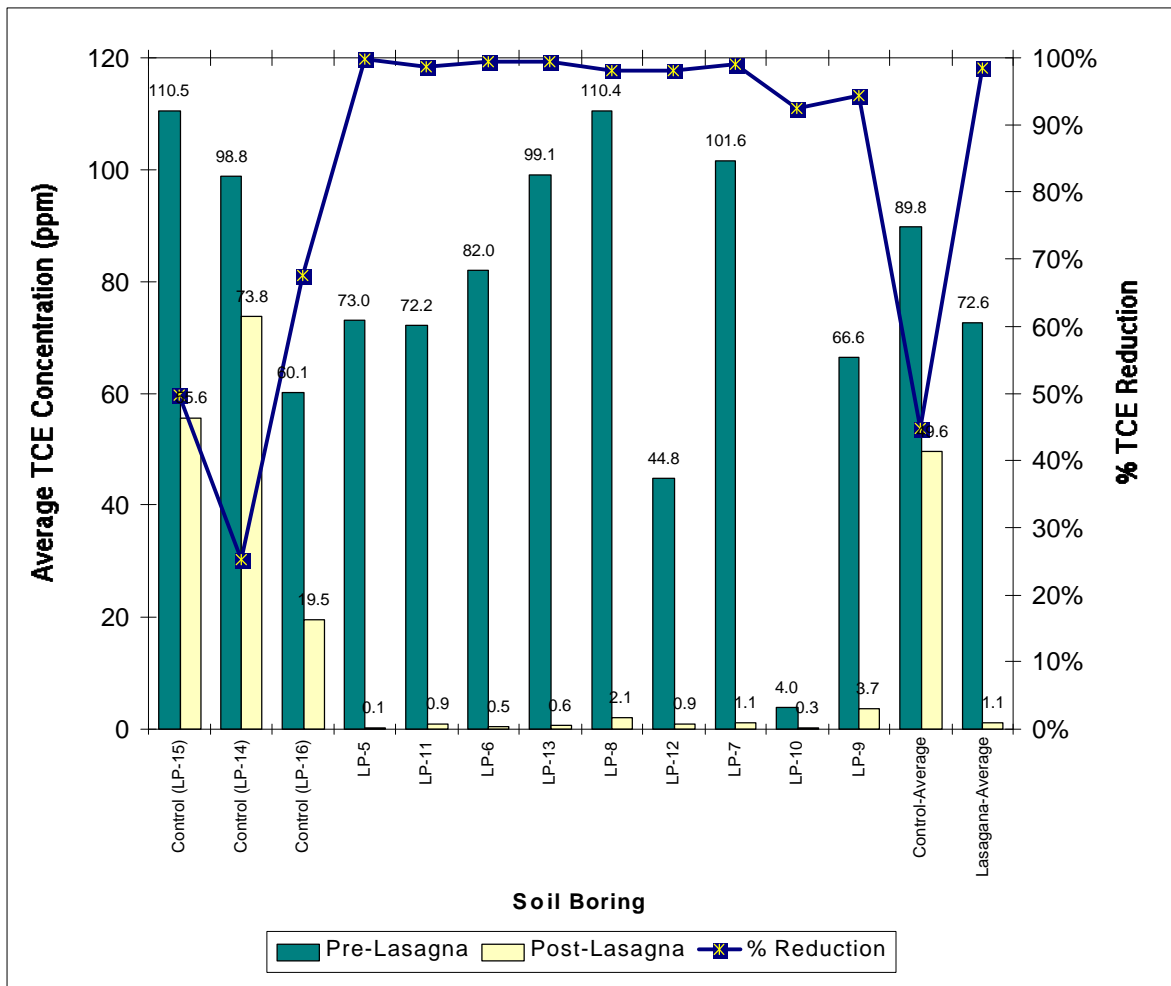
### Phase I Results

During the Phase I study, a number of important milestones were reached. These included:

- Demonstration of good Material Balance for movement of TCE from the contaminated soil into carbon treatment zones,
- Installation of treatment zones and electrodes to a 15-ft. (4.6-meter) depth, and
- Cleanup of 83 yd<sup>3</sup> soil from an average concentration of 72.6 to 1.1 ppmw ( $\mu\text{g/g}$ ), which is well below the target cleanup level of 5.6 ppmw ( $\mu\text{g/g}$ ) (see Figure C-3).

Note: the Phase I study and its results were not verified as part of this RCI project. Phase IIa is the focus of this present report.

**Figure C-3. Average TCE Concentrations Pre- and Post-Lasagna™ for the Phase I Field Experiment**



## Phase II Field Study

The current RCI project used part of the Phase II activities for continued development of this technology (which has been funded by DOE-OR and the Subsurface Contaminants Focus Area) to produce and verify soil cleanup and cost data for rapid commercialization of this technology. The Phase II study is divided into IIa and IIb. Phase IIa is designed to provide information to the consortium for the final design of the full-scale cleanup and to demonstrate to EM-40 that the full-scale cleanup (Phase IIb) should proceed. Phase IIa demonstrated that:

- Treatment zones and electrodes can be emplaced to a depth of 45 ft. (13.7 meter),
- Selected portions of the Phase IIa volume of 1,050 yd<sup>3</sup> can be cleaned up to less than the 5.6 ppmw (µg/g) target level,
- Iron can be used to degrade TCE, and

- DNAPL as well as dissolved TCE can be remediated.

Since the completion of this Phase IIa study, the Paducah Plant has selected the *Lasagna*™ process as their preferred method of cleanup in the proposed Record of Decision (ROD). If the ROD is signed full-scale cleanup (Phase IIb) may begin where approximately 10,000 yd<sup>3</sup> of low-permeability clay will be treated.

The contaminant targeted for the Phases I and II research is trichloroethylene. Greater than 60% of the DOE facilities have chlorinated solvents in the soils and sediments and greater than 80% of the facilities have detected chlorinated solvents in the groundwater. Trichloroethylene is the most prevalent chlorinated solvent found in the soils and groundwater throughout the DOE complex (reference DOE/ER-0547T). Many of the other RCI sponsoring organizations have similar contaminated sites.

If Phase II studies are successful, further work could be pursued to develop *Lasagna*<sup>TM</sup> technology for treating soils contaminated with heavy metals, chlorinated aromatics, and mixed wastes. Figure C-4 shows the Phase IIa field experiment layout, with sampling and monitoring sites.

Section D of this final report gives a complete description of the project organization and how RCI was incorporated into the execution of this study to validate the data obtained from the Phase IIa field test. Section E compares what was planned in the QAPP with what was actually accomplished, and Section F describes the results from the analysis of samples which were taken. Section G shows how this data was used to validate the data upon which the feasibility of using the *Lasagna*<sup>TM</sup> process for cleaning up the remainder of the Phase II site is based. Section H gives cost information for this process, developed by MSE. Appendix A shows the Verification Statement that was written as a result of this final report. Appendix B summarizes the pre-test soil data taken in March 1996, several months before beginning installation of Phase IIa. Appendix C shows sections from the original QAPP. The goals shown in this appendix form the basis for much of the discussions given in Sections E - G of this final report. Appendix D shows the tabular data for all figures plotted in Sections F and G. Appendix E gives supplementary auditing data, and Appendix F discusses the importance of measuring C<sub>2</sub> hydrocarbon degradation products in well water samples.

## Site Description and Contamination

The Phase IIa field test was located at the Paducah Gaseous Diffusion Plant cylinder drop-test pad area. The area is mostly clay with gravel and clay overburden and has been used as a truck road. The contaminant at this site is trichloroethylene (TCE). The results of several soil sampling events and well installations provided the sub surface TCE concentrations needed to define the vertical and lateral extent of the TCE plume. The soil TCE concentrations in this area range from below 1 ppmw ( $\mu\text{g/g}$ ) to approximately 1,500 ppmw ( $\mu\text{g/g}$ ). With a soil density of 2 g/cc, a porosity of 0.4 and a moisture content of 20%, a soil concentration of 225 ppmw ( $\mu\text{g/g}$ ) would result in TCE saturated pore water (1,100 ppmw ( $\mu\text{g/g}$ )). There is definite indication of dense non-aqueous phase liquid (DNAPL) in the area. Table C-1 lists the characteristics of the Paducah soil and Figure C-5 is a contour map showing the highest TCE concentrations found in the soil based on several series of soil borings and well installations.

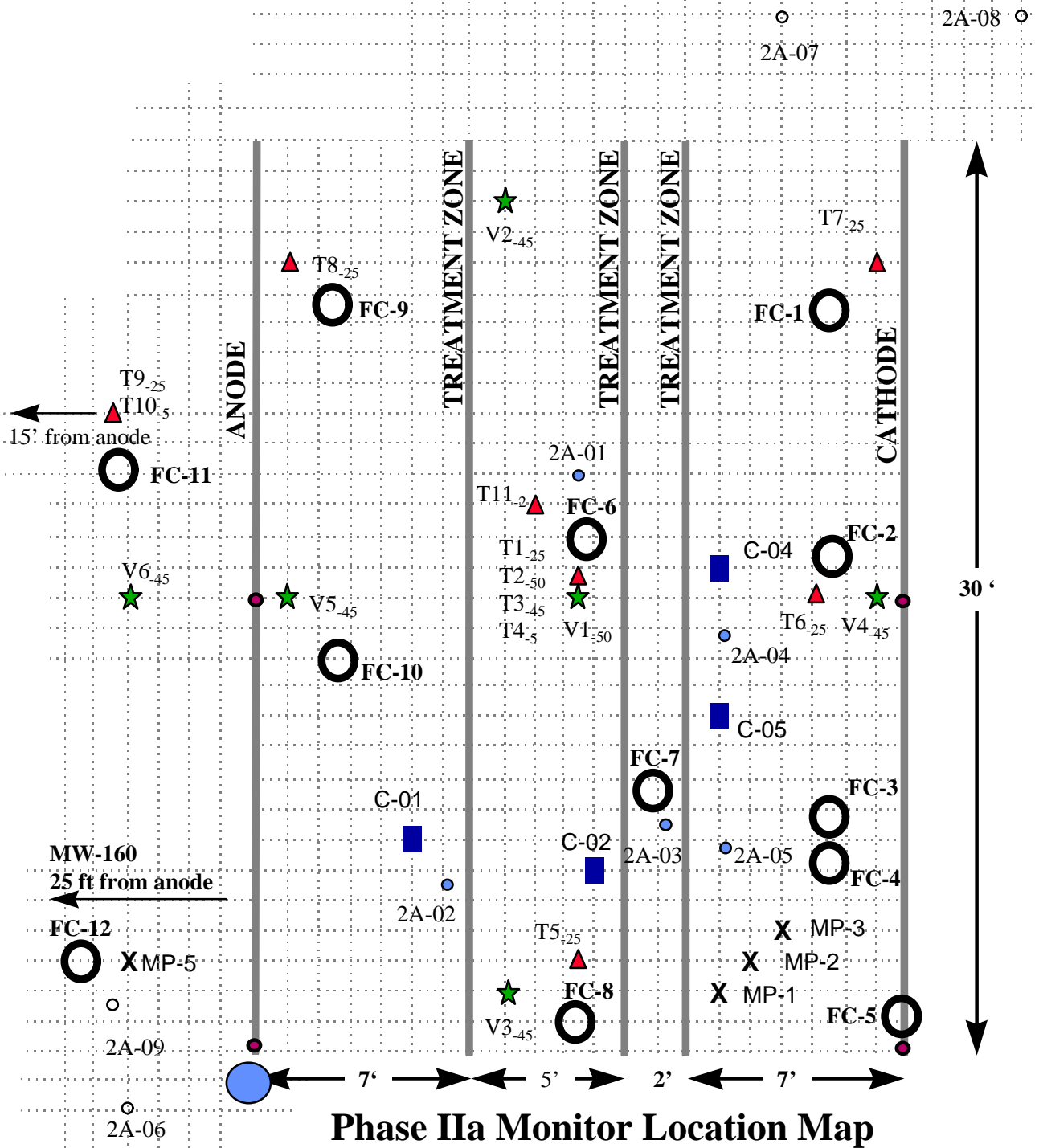
Nine pretest soil boring were made to confirm the northern edge of the plume and to determine the baseline of TCE concentration in the test area. Soil samples were taken at 1 foot intervals to a depth of 40 feet. The TCE contamination appears to taper off to below detection limits at about 30 to 35 feet deep. It was determined from this data that the *Lasagna*<sup>TM</sup> system should be installed to a depth of 45 feet. The regional aquifer is approximately 60 feet below grade in this area. More detailed information on the operation of the Phase IIa field experiment may be found in the final report for the project.<sup>12</sup>

---

<sup>12</sup> Athmer, C. J., Ho, S. V., Hughes, B. M., Sheridan, P. W., Brodsky, P. H., Shapiro, A. P., Salvo, J. J., Schultz, D. S., Landis, R. C., Griffith, R., and Shoemaker, S. H., "Topical Report for Task 7.2 entitled "Field Scale Test" (January 10, 1996 - December 31, 1997), final report for DOE Contract Number DE-AC05-96OR22459".

Figure C-4. Locations of Sampling and Monitoring Sites in the Phase IIa Field Experiments

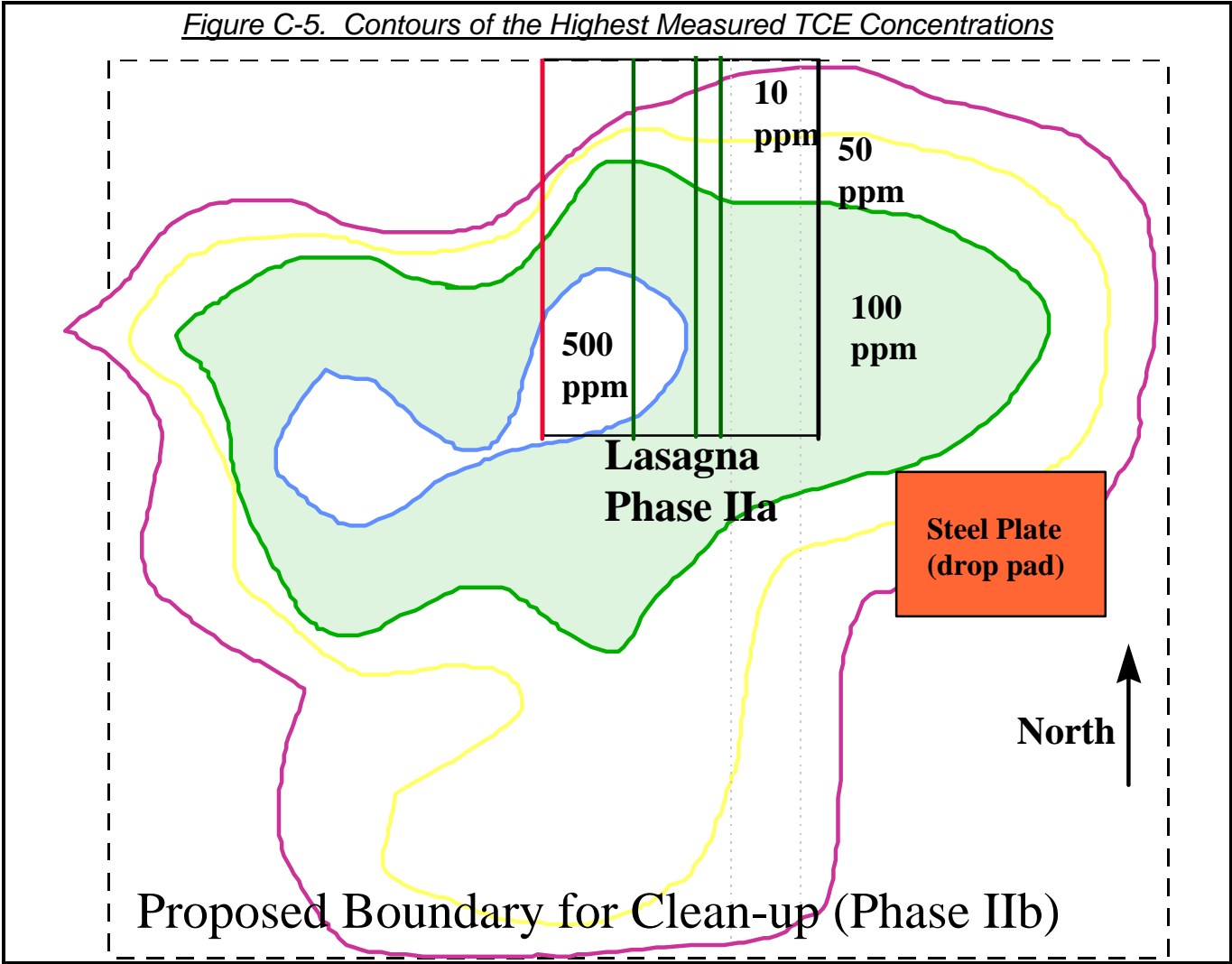
- 4 CARBON SOCKS 25 FEET DEEP
- ▲ 6 THERMOCOUPLES 5 TO 50 FEET DEEP
- ✕ 4 MEMBRANE PROBES 25 FEET DEEP
- 5 WATER WELLS 25 FEET DEEP
- ★ 6 VOLTAGE PROBES 45 OR 50 FEET DEEP
- 4 ANODE/CATHODE WELLS 25 FEET DEEP
- ADDITIONAL PRE-TEST SOIL BORINGS
- FLUX CHAMBER LOCATIONS



**Table C-1. Paducah Soil Characteristics**

Soil Type:	Clay Loam
Sand Content:	22 %
Silt Content:	46%
Moisture Content:	15 -18%
Organic Carbon Content:	0.2%
Cation Exchange Capacity:	13.4 meq/100g.
Porosity:	0.4
Bulk Density:	2.0 g/cm <sup>3</sup>
Electroosmotic Conductivity:	1.2 x 10 <sup>-5</sup> cm <sup>2</sup> /v-sec
Hydraulic Conductivity (estimated):	1 x 10 <sup>-7</sup> cm/sec

**Figure C-5. Contours of the Highest Measured TCE Concentrations**





## D. Project Organization and Responsibility

Integrated *in-situ* remediation technologies, which combine electro-osmosis with zero valent iron degradation techniques, may greatly reduce cleanup costs for remediation of sites contaminated with chlorinated solvents.

### Consortium Organization

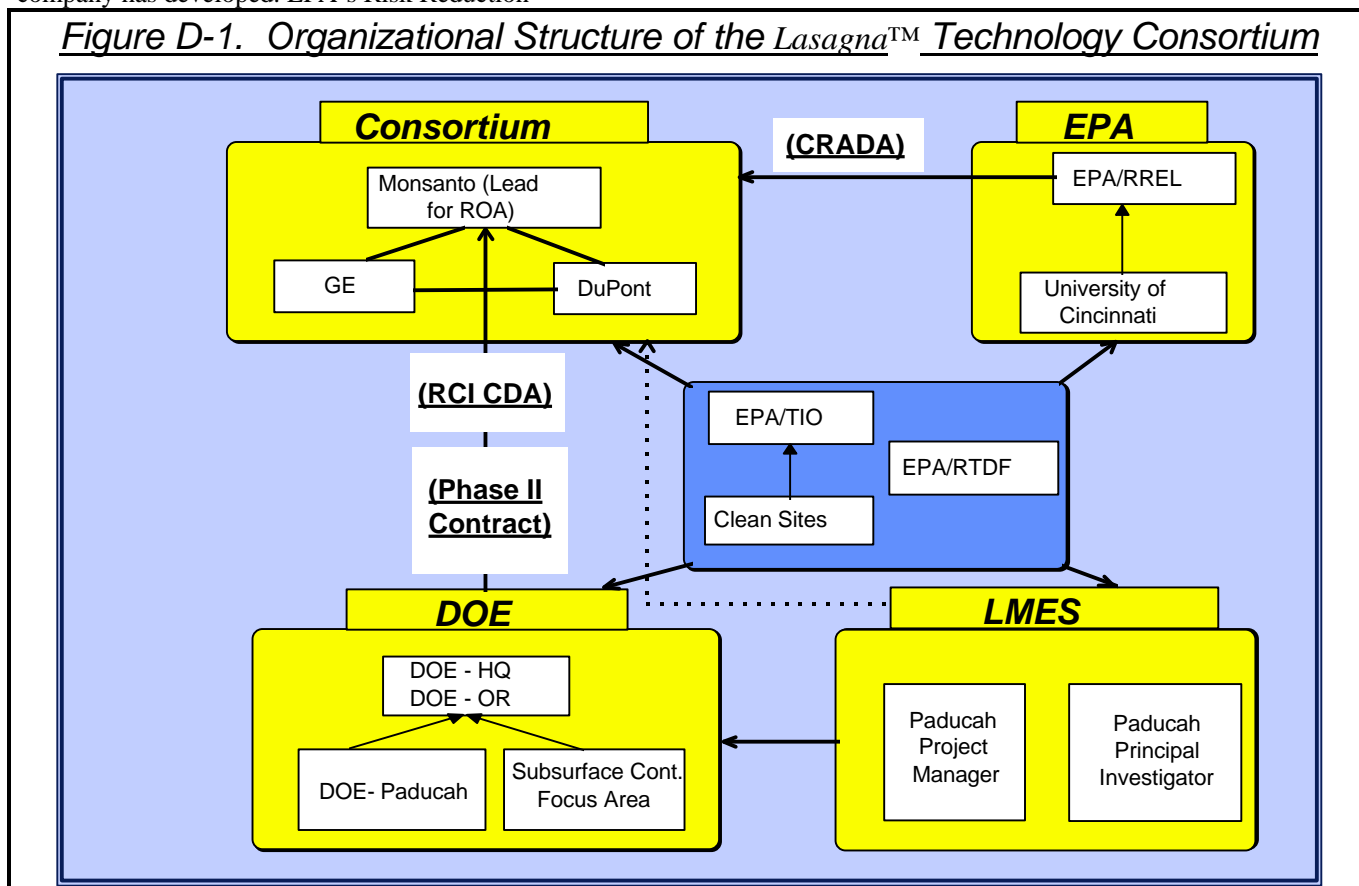
Figure D-1 shows a diagram of the structural organization of the research consortium which has been developing important elements of the *Lasagna*<sup>TM</sup> *in-situ* remediation technology. Four major industrial and government entities are shown interacting through the EPA's Remediation Technologies Development Forum (RTDF). This latter program is shown at the center of the diagram with Clean Sites playing an important role as a contractor to EPA's Technology Innovation Office (TIO).

The industrial partners, consisting of Monsanto, General Electric, and DuPont, shown in the upper left of the diagram have formed an industrial consortium to develop the *Lasagna*<sup>TM</sup> technology. This technology was created by combining important technologies that each company has developed. EPA's Risk Reduction

Engineering Laboratory (RREL) is shown in the upper right corner of the diagram. Their important contribution is in the development of hydrofracturing for the emplacement of large, horizontal, electrodes and reaction zones which may permit the *Lasagna*<sup>TM</sup> technology to be used in a horizontal configuration.

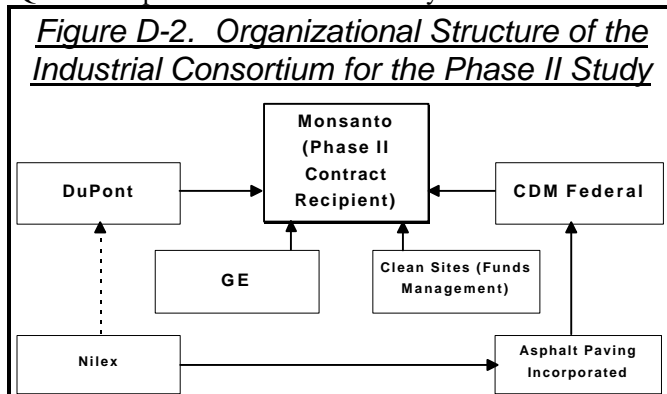
The Department of Energy and its contractor, Lockheed Martin Energy Systems (LMES), are shown at the bottom of the figure. It is through the DOE that the Consortium has obtained significant funding to continue the development of this technology. They have also been instrumental in developing the overall strategy for evaluating the *Lasagna*<sup>TM</sup> technology. DOE's on-going research programs relevant to the *Lasagna*<sup>TM</sup> technology will be utilized as appropriate. LMES will be providing critical sampling and analysis support, as well as coordinating field activities at the Paducah site.

**Figure D-1. Organizational Structure of the *Lasagna*<sup>TM</sup> Technology Consortium**



## RCI Organization

A number of questions arose from EPA's auditor, Sam Hayes, concerning how the various QA Organizations and Project Managers interacted for the Phase IIa field study. Figure D-2 shows more details on the industrial consortium, and Figure D-3 presents the multiple organizations and responsibilities that a number of individuals have. The following paragraphs describe how QA is incorporated into this field study.



### Three Major Teams

#### Phase IIb Final Cleanup Team

The field study that is being conducted at Paducah uses the resources from three major teams. The first one shown in Figure D-3 is the Phase IIb team which is responsible for the total cleanup of the site. Ms. Myrna Redfield is the DOE-Paducah Project Manager. She is responsible for interacting with the Commonwealth of Kentucky and the Region IV CERCLA office of EPA to obtain the appropriate permits and the Record of Decision (ROD) which will be used to ultimately cleanup up the SWMU 91 area. Funding for this cleanup will come from EM-40 funds, if the ROD is signed.

#### RCI Process and Cost Validation Team

The Rapid Commercialization Initiative (RCI) Participants are the second team which is involved in this field test. This team is responsible for obtaining process and cost validation data which demonstrate whether the process is effective and to estimate the cost of the SWMU 91 cleanup. The RCI program should also expedite the use of the *Lasagna*<sup>TM</sup> process for cost effective cleanup of TCE-contaminated clay at government and commercial sites. A more detailed description of the role and the members of the RCI Participants is given in Appendix A.

#### Phase IIa Test Cleanup Team

The third member is the Phase IIa team which is responsible for the test cleanup of a small portion of the total SWMU 91 area. The funding of this test cleanup was

obtained from EM-50 and is a continuation of an initial study funded from DOE's Research Opportunities Announcement (ROA) program. The continuation of this research extends the depth of cleanup from 15 to 45 ft., and extends the method of treatment from carbon adsorption to iron degradation of TCE.

### Discussion of Figure D-3

A number of conventions are used in Figure D-3 that help understand the interactions of the QA oversight of the Phase IIa study with these organizations. A much larger group is involved in this study, but cannot be shown due to the complexity of the figure. All QA functions are shown as ovals in the figure. In addition, the dotted-dashed lines indicate which functions are being audited by the various QA functions. Finally, persons with multiple responsibilities are connected to their organizations through various bold solid lines with arrow heads. For example, the LMES Project Manager, Fraser Johnstone, functions in two roles, since he is the LMES project manager for the Phase IIa test cleanup and, if the ROD is signed, for the Phase IIb final cleanup. Similarly the LMES Project Hydrogeologist, Jay Clausen (or his replacement, Jeff Douthitt) functions as a part of the Phase IIb LMES final cleanup, while also functioning in the Phase IIa team to provide all analytical information for the test field study.

The RCI Technology Holder Project Manager, B. Mason Hughes, not only serves as a part of the Phase IIa test cleanup team, but he also is a part of the RCI Participants. In these capacities he provides a technical link between the RCI Participants and the Phase IIa test cleanup team which is producing the data for process and cost validation.

### QA Functions

The primary QA for the Phase IIa field study is being provided by the LMES ER QA Specialist, Jennifer Blewett. A detailed description of her role and responsibilities is shown in the following section. In addition to her daily oversight of all data that is generated by the CDMFPC Federal, ORNL/GJ, and Lockheed Martin Utilities Services (LMUS) Analytical Laboratories, she also reviews all QA plans for the LMES activities. The Verification Entity, David Carden, serves as an independent QA reviewer who is a member of the RCI Participants and reports to the DOE RCI Project Manager, Skip Chamberlain. He also reviews data generated by a third-party analytical laboratory, Roy F. Weston Laboratories, which is used to provide QA data that will be used to assess the precision and accuracy of data generated from the laboratories used by LMES. In addition, LDC Data Validators review all data that is generated by Roy F. Weston Laboratories and produces an assessment of the completeness of the data generated from

this third-party laboratory. A later section gives more information on Mr. Carden's duties. An additional QA auditor is also used by the RCI Participants. This auditor, Mr. Sam Hayes, is provided by the EPA, and generated the Technical Systems Review to which this response is addressed. The following sections describe a number of the persons shown in Figure D-3 with an emphasis on the QA function of this program.

## Personnel

### Paducah Department of Energy (DOE) Project Manager - Myrna Redfield

The DOE Project Manager provides technical and management oversight for the Sampling and Analysis Plan (SAP) and also is the primary interface between U.S. Environmental Protection Agency (USEPA), Kentucky Department of Environmental Protection (KDEP), and Lockheed Martin Energy Systems (LMES).

### LMES, Environmental Restoration (ER), Program Manager - Fraser Johnstone

The LMES ER Program Manager has overall responsibility for the SAP implementation process and interfaces with DOE program management and regulatory agencies. Additional programmatic responsibilities include technical, quality, financial, and scheduling matters. The LMES ER Program Manager reports to the Paducah DOE Project Manager as well as to the DOE Oak Ridge Project Manager.

### LMES, ER, Technical Oversight Coordinator - Jay Clausen (currently Jeff Douthitt)

The LMES ER Technical Oversight Coordinator has programmatic responsibility for technical integration of the investigation activities and is the principal interface with the *Lasagna*<sup>TM</sup> Consortium through Monsanto Enviro-Chem, and the DOE Verification Entity. The LMES ER Technical Oversight Coordinator reports directly to the LMES ER Program Manager. At the end of the Phase IIa study, Mr. Jeff Douthitt replaced Mr. Clausen in this capacity.

### LMES, ER, Quality Assurance (QA) Specialist - Jennifer Blewett

The LMES ER QA Specialist is responsible for evaluating quality concerns, issues, problems, and performing project surveillances. The LMES ER QA Specialist oversees data collection and analyses performed by Oak Ridge National Laboratory/Grand Junction (ORNL/GJ), Camp Dresser McKee Federal (CDMFPC), and Lockheed Martin Utility Services (LMUS). The LMES ER QA Specialist reports to the LMES ER Program Manager.

### CDMFPC Federal Analytical Laboratory

CDMFPC Federal oversees operation of the *Lasagna*<sup>TM</sup> test plot and water sampling. They are also responsible for site accessibility, safety, and QA measures. QA measures include implementation of the CDMFPC QA program, reviewing, identifying, and solving problems. They are responsible for ensuring that QA requirements in the SAP and Quality Assurance Plan (QAP) are followed; ensuring that sampling and decontamination procedures are effectively implemented; ensuring that appropriate QA/QC requirements and technical commitments are met; ensuring that all applicable state federal codes, standards, and regulations are appropriately specified and effectively implemented; interfacing with the LMES ER QA Specialist on all quality related matters, and initiating stop-work actions when conditions or procedures adverse to quality warrant immediate action. The CDMFPC Analytical Laboratory reports to the LMES ER Program Manager.

### ORNL/GJ Analytical Laboratory

ORNL/GJ conducts soil and carbon sampling and analysis. They are also responsible for site accessibility, safety, and QA measures. QA measures include implementation of the ORNL/GJ QA program, reviewing, identifying, and solving problems. They are responsible for ensuring that QA requirements in the SAP and Quality Assurance Plan (QAP) are followed; ensuring that sampling and decontamination procedures are effectively implemented; ensuring that appropriate QA/QC requirements and technical commitments are met as outlined in the Memorandum of Understanding between ORNL/GJ and LMES ER (KY/EM-97); ensuring that all applicable state and federal codes, standards, and regulations are appropriately specified and effectively implemented; interfacing with the LMES ER QA Specialist on all quality related matters; and initiating stop-work actions when conditions or procedures adverse to quality warrant immediate action. The ORNL/GJ Analytical Laboratory reports to the LMES ER Program Manager.

### LMUS Analytical Laboratory

LMUS conducts soil and water split sample analyses. QA measures include implementation of the LMUS QA program, reviewing, identifying, and solving problems. They are responsible for ensuring that QA requirements in the SAP and Quality Assurance Plan (QAP) are followed; ensuring that sampling and decontamination procedures are effectively implemented; ensuring that appropriate QA/QC requirements and technical commitments are met; ensuring that all applicable state and federal codes, standards, and regulations are appropriately specified and effectively implemented; interfacing with the LMES ER QA Specialist on all quality related matters; and initiating stop-work actions when conditions or procedures adverse to quality

warrant immediate action. The LMUS Analytical Laboratory reports to the LMES ER Program Manager.

**Oak Ridge DOE Project Manager - Leon Duquella**

The DOE Project Manager provides technical and management oversight for the Sampling and Analysis Plan (SAP) QAP and is also the primary interface between LMES, Paducah DOE Project Manager, the *Lasagna*<sup>TM</sup> Consortium, Monsanto Enviro-Chem, the DOE Verification Entity, and the Rapid Commercialization Initiative Project Manager.

**MSE/Cost Verifier - Stephen Antonioli**

The organization responsible for verifying the costs of the Phase IIa *Lasagna*<sup>TM</sup> field test is MSE-TA (MSE Technology Applications, Inc.). Andy Johnson and Stephen Antonioli of MSE interacted directly with the CDMFPC project manager, Robert Hines, to obtain accurate cost information for conducting the field test. MSE is located in Butte, MT and is a subsidiary of MSE, Inc. It is staffed with engineers and scientists with a variety of backgrounds and provides services to local, state and federal government agencies as well as to private companies.

**RCI/DOE Verification Entity - David Carden**

The DOE Verification Entity provides QA oversight over sampling and analysis activities. The DOE Verification Entity is responsible for evaluating quality concerns, issues, problems, and performing project surveillances. The DOE Verification Entity reviews data collection and analyses performed by Oak Ridge National Laboratory/Grand Junction (ORNL/GJ), Camp Dresser McKee (CDMFPC), Lockheed Martin Utility Services (LMUS), and Roy F. Weston Laboratories. The DOE Verification Entity also interfaces with the Paducah DOE Project Manager, LMES Program Manager, *Lasagna*<sup>TM</sup> Consortium, and RCI Industry Project Manager. The DOE Verification Entity reports to the DOE Rapid Commercialization Initiative Project Manager and had participated in the preparation of the final validation report from the RCI Participants.

**RCI/EPA Representative - Sam Hayes**

RCI's EPA representative provides an additional QA oversight for the RCI Participants and assures that all field activities are properly documented and consistent with regulatory federal and state agencies' requirements. All RCI Participants members participated in writing a final process validation report which incorporates the QA findings from the EPA representative along with the Verification Entity's findings.

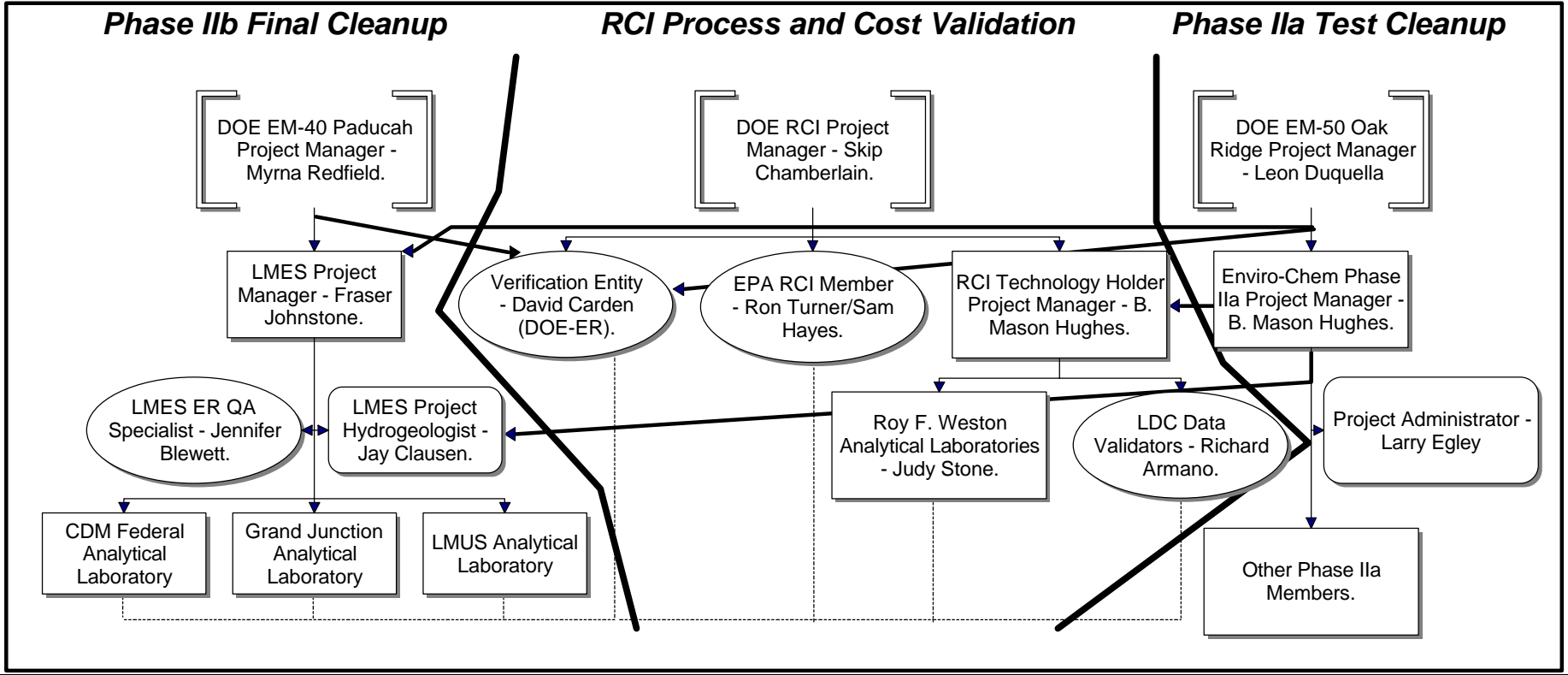
**Third-Party Laboratory**

**Roy F. Weston Laboratory** serves as the Verification Entity's third-party laboratory. The project manager for this laboratory is Ms. Judy Stone. She serves as project coordinator of all split sample analyses shown in this QAPP. She also interfaces with Mr. Carden and Dr. Hughes and is responsible for communicating the analytical requirements outlined in this QAPP to the analysts providing analytical results. Roy F. Weston Laboratory is an independent laboratory and has no vested interest in the success of the Phase IIa study. The expenses of these third-party analyses are funded by Monsanto Co.

**Data Validator**

**Laboratory Data Consultants, Inc.** serves as the data validator for the split samples analyzed by Weston laboratories. A full EPA Level 4 review was conducted. Ms. Linda Rauto, chemist, coordinates all daily validation activities. Mr. Richard Amano, principal chemist, acts as the overall project manager. The expenses of this data validation are borne by Monsanto Co.

Figure D-3. Organizational Chart for QA Oversight of Lasagna™ Phase IIa Field Study



## E. Comparison of Actual Activities to Those Planned in the QAPP

The evaluation of a new integrated *in-situ* remediation technology, which combines electro-osmosis with zero valent iron degradation techniques, was made by using well-defined QA objectives. This section compares the actual to the planned activities given in the QAPP.

The QA Objectives for the Phase IIa study were developed to produce validated data for DOE, DOD, and state environmental agencies to evaluate the process' success and thus to allow the use of Lasagna™ for remediation of low permeability soils with TCE contamination. For the Paducah site, a wide range of TCE concentrations were measured during the Phase I test. Therefore, soil data was obtained by ORNL/GJ from five soil borings at 1-ft depth intervals before and after the Phase IIa field experiments to more accurately determine the average TCE concentration.

### Phase II Cleanup Levels

Ultimately, in order for the Lasagna™ process to be a success for this site, the mean TCE soil concentrations must be below approximately 5.6 ppmw (µg/g). (This is the action level determined by LMES, DOE, and the Commonwealth of Kentucky, based on groundwater modeling calculations.) This will be determined by statistical analysis of the soil concentration data after 1 - 2 years of operation of the Phase IIb field experiment and applying the following decision rule:

- **If the upper 95% confidence level of the mean of the soil sample population is less than 5.6 ppmw (µg/g) then the Lasagna™ process can be judged to be successful as a remediation technology for the specific application at Paducah.**

If this criterion is not met after 1 - 2 years of Lasagna™ operation, it will be possible to extend the treatment time, without re-installing the electrodes and treatment zones. In order to evaluate the data for use by other states, a destruction and removal efficiency (DRE) will also be calculated in several ways. The overall DRE will be calculated, and DRE's will be calculated at specific depths from the paired pre- and post-Lasagna™ soil concentrations. Efforts will be made to show whether TCE was destroyed or re-located at the site.

### Original Phase IIa Field Experiment Cleanup Levels

The QAPP described how to evaluate the data from the Phase IIa field experiment. This approach was based on the anticipated length of operation of this phase, originally between 3 and 6 months. The decision rule mentioned

above was to be applied only to the post-experiment core samples 2A-04 and 2A-05, since during 3 - 6 months of operation, contaminated water should theoretically pass through two treatment zones before arriving at these two sampling points. The mean pre-test TCE concentration of these two points (shown in Appendix B) was approximately 34 ppmw (µg/g). (This mean was calculated by averaging soil concentration data between 5' or 6' and 40' bgs.) The Phase IIa field experiment was to be considered a success by applying the following decision rule:

- **If the upper 95% confidence level of the mean of the soil sample population is less than 5.6 ppmw (µg/g) for 2A-04 and 2A-05 then the Lasagna™ process can be judged to be successful as a remediation technology for the specific application at Paducah.**

The core samples 2A-01, 2A-02, and 2A-03 were located such that less than one pore volume water should flow past these three points in 3 - 6 months. The average pre-experiment TCE concentrations for these three points were 18.3, 41.3, and 52.3 ppmw (µg/g), respectively. (These means were calculated by averaging soil concentration data between 4' and 35' bgs for 2A-01, between 5' and 35' bgs for 2A-02, and between 4' and 39' bgs for 2A-03.) Due to the low volume of treated water passing these points, the above decision rule was not to be used for these latter 3 points. Therefore, a successful process may not result in the same mean concentrations for these 3 core samples as was anticipated for the other 2 cores. It was predicted that the average soil concentrations at the core sampling points 2A-01 and 2A-02 would not be reduced over the pre-test concentrations in the 3 - 6 month period and that sampling point 2A-03 would be partially cleaned up. It was expected that the mean concentration of the post-Lasagna™ core samples at point 2A-03 would be reduced below 25 ppmw (µg/g). However, there were no plans to use this criterion for assessing the success of the Phase IIa experiment, especially if the means for points 2A-04 and 2A-05 were below the 5.6 ppmw (µg/g) target. Therefore only the results from 2A-04 and 2A-05 were planned to be used to indicate whether the full Phase II should be installed after the first 3 - 6 months of Phase IIa operation.

### Actual Cleanup Levels

A number of problems were encountered in the field which caused the Lasagna™ Consortium and RCI

Participants to modify the above decision rule cleanup levels of Phase IIa. The major problem was associated with the very large amount of TCE DNAPL which was encountered at the site. After the first 3 months of operation, carbon samples indicated that there were much larger amounts of free phase TCE present in the 15 - 25' depth region, than would be predicted from the TCE concentrations measured in the pre-test soil samples which are shown in Appendix B. All of the cleanup times and cleanup levels which were shown in the original QAPP were based upon the assumption that TCE was dissolved in the pore water in the clay formation, and that three months of operation would replace all of the contaminated water which was up gradient of wells 3, 4, and 5 with clean water which had passed through one or two iron treatment zones. [This assumption was made from the rates of reaction, and EO water velocity (and thus reaction time) which would occur after one pass through the treatment zone under normal operating conditions.]

The presence of pure-product DNAPL, however, meant that non-ideal operation of the process may result in much longer treatment times, since TCE must be dissolved into the water before it could be efficiently transported through the iron treatment zones. Further complications were also observed during installation of the IIa electrodes and treatment zones, when soil cores were examined in more detail. The sand layer at 20' {6.1 meters} appears to be a source of DNAPL and may offer a source and transport channel of contamination into the Phase IIa site from the nearby contamination in the remaining portions of the Phase II site. Therefore the criteria for success of the Phase IIa experiment was considered too rigid to indicate whether the *Lasagna*<sup>TM</sup> process could be used successfully to clean up the entire Phase IIb site.

## Comparison with Original QAPP

Appendix C shows the original Sections E - G in the original QAPP. That appendix shows the original data quality objectives, planned sampling and analysis activities, and how the treatment effectiveness would be verified. The next section of this report (Section F) will compare the planned to the actual activities and Section G will discuss how the Quality Assurance data from analysis of water, carbon, air, and soil samples analyzed by up to four different laboratories were used to verify the success of the *Lasagna*<sup>TM</sup> Process.

## F. Sampling and Analysis Results

---

The results of analysis of a large number of water, soil, and carbon samples were used to evaluate the effectiveness of the *Lasagna*<sup>TM</sup> process. Standard statistical evaluation procedures were used to determine the accuracy of the obtained data.

---

### Sampling Plan

This section of the final report is designed to summarize all data which was collected under the guidance and supervision of the RCI Participants. Tables F-1 and F-2 in Appendix C show the sampling and analyses which was anticipated before beginning the Phase IIa field studies. As mentioned in Section E, the large amount of unanticipated DNAPL caused the original plans to be altered, increasing the operating time from 3 to approximately 11 months, and as a result, impacted the total numbers of samples which were actually taken. Table F-1 in this section gives a summary of the operating times for the Phase IIa field experiment. Tables F-2 through F-4 in this section summarize the total numbers of water, carbon, and soil samples which were taken in the Phase IIa field experiment. The totals include all QA samples which were analyzed

### Quality Assurance and Quality Control and Data Usability

Quality Assurance and Quality Control (QA/QC) was implemented throughout all sampling and analysis activities in accordance with the RCI Quality Assurance Plan. QA/QC activities included routine field and laboratory audits by EPA and the DOE Independent Verification Entity, field and laboratory quality control samples, replicate analyses, etc. For a detailed discussion of the QA/QC program and its results, see section G. Section G also discusses how the data shown in the present section can be used to establish confidence levels for the data upon which the RCI Participants are drawing conclusions on the success of the *Lasagna*<sup>TM</sup> technology.

### Sampling Results

The following paragraphs describe the soil results, the search for chlorinated organic degradation products (i. e. *cis*-dichloroethylene and vinyl chloride), water results, carbon results, flux chamber results, and the

results of analyses for acetylene, ethane, and ethylene degradation products.

In addition to the primary data that the RCI Participants are using to draw conclusions on the success of this technology, these sections rely heavily upon samples which have been split between at least two different analytical organizations. The soil QA data consists of the primary soil samples which were analyzed on-site by ORNL/GJ and soils that were analyzed by LMUS and Weston using Purge-and-Trap GC/MS and GC/PID analytical techniques. In addition, a number of hexane extracts of the soil samples were analyzed in duplicate by ORNL/GJ and split between ORNL/GJ and Weston Laboratories.

The section describing the water sample results show the analyses conducted by CDMFPC, LMUS, and Weston Laboratories. The water concentrations of TCE were used to follow the extent and rate of the cleanup process. The carbon results were similarly used to indicate operational details of the *Lasagna*<sup>TM</sup> process. Duplicates and split extracts were analyzed by ORNL/GJ and Weston.

The flux chamber results showed the extent of TCE volatilization during operation of the process. Only one set of results are presented although a second round of samples were also analyzed. However, the QC results from the second round were insufficient to draw conclusions and are therefore not shown in this report. It is recommended that when the full site cleanup occurs, there should be an additional attempt to monitor the extent of TCE volatilization during operation.

A later part of this section discusses the measurement of acetylene, ethane, and ethylene in the five monitoring wells. These compounds are known to be the final form of the carbon from iron dechlorination of TCE. This information is shown to confirm that degradation is occurring according to the mechanism established for this reaction as is discussed in the final parts of this section.



**Table F-1. Summary of Field Operations for the Phase IIa Study**

<b>Date</b>	<b>Time</b>	<b>Distance Water Front Travel (inches)</b>	<b>Operation</b>
08/14/96	12:10	0.0	Power on
10/1/96	9:00	11.6	Power off for 4.5 hrs to change generator
11/5/96	8:05	23.8	Power off for 4.5 hours for maintenance
11/13/96	8:30	26.2	Power off 6 hrs for carbon sock sampling
12/13/96	9:50	35.8	Reverse Polarity
12/19/96	8:00	34.2	Power off to install new anode rod
12/19/96	14:30	34.2	Back to Normal Polarity
12/23/96	7:30	35.5	Power off 3 hrs for rod repair
2/26/97	9:00	50.3	Power off for Soil Sampling
3/17/97	9:00	50.3	Power on
3/18/97	9:55	50.5	Reverse Polarity
3/31/97	14:30	48.5	Back to Normal Polarity
4/5/97	18:00	49.5	Power off for undetermined reason
4/9/97	13:00	49.5	Power on
5/27/97	12:00	57.7	Power off for anode work
5/28/97	14:35	57.7	Power on
08/06/97	7:27	67.5	Power off

**Total run time was 331.45 days out of a possible  
356.80 days (8/14/96 12:10 to 8/6/97 7:27)**

Table F-2. Summary of Water Samples Actually Analyzed.

Date	CDMFPC	LMUS	Weston	Grand Total
8/7/96	2	0	0	2
8/8/96	6	3	2	11
8/12/96	6	2	2	10
8/27/96	8	3	3	14
9/10/96	7	4	3	14
9/24/96	8	2	2	12
11/13/96	8	2	3	14
12/12/96	8	5	3	16
1/10/97	2	3	2	7
2/13/97	6	11	4	21
3/7/97	0	13	0	13
4/30/97	6	6	2	14
6/6/97	6	5	5	16
7/9/97	7	5	5	17
8/9/97	6	5	5	16
<b>Grand Total</b>	<b>86</b>	<b>69</b>	<b>41</b>	<b>197</b>

Table F-3. Summary of Carbon Samples, Splits, and Duplicates Actually Analyzed.

Week of .....	# Samples	Sample Type (Method)
13 November 1996	77	Carbon Cassette Samples ( <u>GC/EC</u> )
	10	Duplicate Analyses
	5	Split Analyses (Weston)
13 December 1996	46	Carbon Cassette Samples ( <u>GC/EC</u> )
	3	Duplicate Analyses
9 January 1997	63	Carbon Cassette Samples ( <u>GC/EC</u> )
	5	Duplicate Analyses
24 February 1997	62	Carbon Cassette Samples ( <u>GC/EC</u> )
11 March 1997	62	Carbon Cassette Samples ( <u>GC/EC</u> )
	8	Split Analyses (Weston)
11 August 1997	116	Carbon Cassette Samples ( <u>GC/EC</u> )
	13	Split Analyses (Weston)
<b>Totals</b>	<b>470</b>	<b>Carbon Cassette Samples, Replicate, and Split Analyses (<u>GC/EC</u>)</b>

Table F-4. Summary of Soil Samples, Duplicates and Splits Actually Analyzed.

Week of .....	# Samples	Sample Type (Method)
7 March 1997	145	Soil Cores <u>(GC/EC)</u>
	13	Duplicate Analyses
	13	Split Analyses (ORNL/GJ)
	13	Split Analyses (LMUS-P&T)
11 August 1997	243	Soil Cores <u>(GC/EC)</u>
	22	Duplicate Analyses
	22	Split Analyses (ORNL/GJ)
	20	Split Analyses (Weston-P&T)

## Soil Sample Results

The vast majority of soil samples were measured by ORNL/GJ using hexane to extract the TCE from the soil. This procedure is superior to standard water analysis methods, since significant TCE losses can occur while transporting soil samples to the laboratory. The ORNL/GJ analytical procedure calls for placing an aliquot of contaminated soil directly into a pre-weighed vial containing 10 mLs hexane while in the field. This minimizes TCE loss. To confirm that these results are comparable to those obtained from EPA's Method 624 Purge-and-Trap GC/MS analysis method, samples were split and analyzed using GC/MS techniques by LMUS and Weston Laboratories. This comparison yielded mostly acceptable results (see Section G for a complete discussion).

Tables F-5 through F-7 and Figures F-1 and F-2 summarize the soil sample results obtained by ORNL/GJ.

L2A-01 and L2A-02 were only sampled at the beginning and end of the study, while the remaining three sites were sampled an additional time, indicated as "Inter" in the tables and figures. The bottom of the tables also summarize the average TCE concentrations, % reduction and average TCE concentrations measured. Note that when considering locations L2A-03 through L2A-05, an average concentration of 6.6 ppmw ( $\mu\text{g TCE/g soil}$ ) was measured. This is only 1.0 ppmw ( $\mu\text{g TCE/g soil}$ ) larger than the average TCE concentration that the Commonwealth of Kentucky requires for site closure. However, the concentration of 6.6 ppmw ( $\mu\text{g TCE/g soil}$ ) was reached after 11 months of operation which is only one-half the time that the full cleanup is anticipated to require. It is the opinion of the technology holder that this feasibility study indicates that full cleanup of the site should be achieved if operated for the full two-year time period.

**Table F-5. Summary of Pre- and Post-Lasagna™ Soil Samples for L2A-01 and L2A-02 Soil Cores Analyzed by ORNL/GJ (GC/EC).**

**Lasagna™ TCE Concentrations (ug/g) As a Function of Depth**

Depth(ft)	L2A-01-pre	L2A-01-post	Depth(ft)	L2A-02-pre	L2A-02-post
4	0.04		4		0.05
5	0.06	0.63	5	0.11	0.47
6	0.07	0.46	6	1.53	0.00
7	0.04	0.08	7	8.58	0.69
8	0.04	0.23	8	16.99	0.21
9	0.04	0.27	9	17.53	0.33
10	0.02	0.14	10	37.33	0.12
11	0.02	0.03	11	49.01	0.14
12		0.01	12	38.79	0.45
13	0.00	0.00	13	96.06	1.67
14	0.01	0.00	14	27.18	5.72
15	0.01	0.00	15	39.89	0.36
16		0.00	16	39.93	7.66
17	0.00	0.00	17		1.52
18	0.00	0.01	18	52.93	2.51
19	0.00	0.01	19	43.30	12.08
20	0.01	0.15	20	118.25	8.76
21	0.06	0.03	21	50.13	100.66
22	0.80	3.19	22	34.22	12.50
23	1.02	10.59	23	41.38	85.58
24	13.69	7.28	24	148.16	73.75
25	85.83	0.90	25	99.56	50.90
26	113.04	1.36	26	57.01	31.20
27	124.92	0.48	27	56.32	68.61
28		0.73	28	0.70	41.54
29		0.14	29	21.67	49.96
30	70.19	0.16	30	25.09	73.45
31	46.65	1.06	31	23.90	56.98
32	15.22	1.52	32	6.77	44.87
33	14.88	0.28	33	4.81	106.23
34	11.13	0.60	34		30.11
35	13.52	0.06	35		11.62
36		0.00	36		0.00
37		0.00	37		0.00
38		0.00	38		0
39		0.00	39		0
			40		0

<b>Average:</b>	18.3	0.9	41.3	24.5
<b>% Removal:</b>	-	95.25%	-	40.81%

**Table F-6. Summary of Pre-, Inter-, and Post-Lasagna™ Soil Samples for L2A-03 Soil Cores Analyzed by ORNL/GJ (GC/EC).**

**Lasagna™ TCE Concentrations (ug/g) As a Function of Depth**

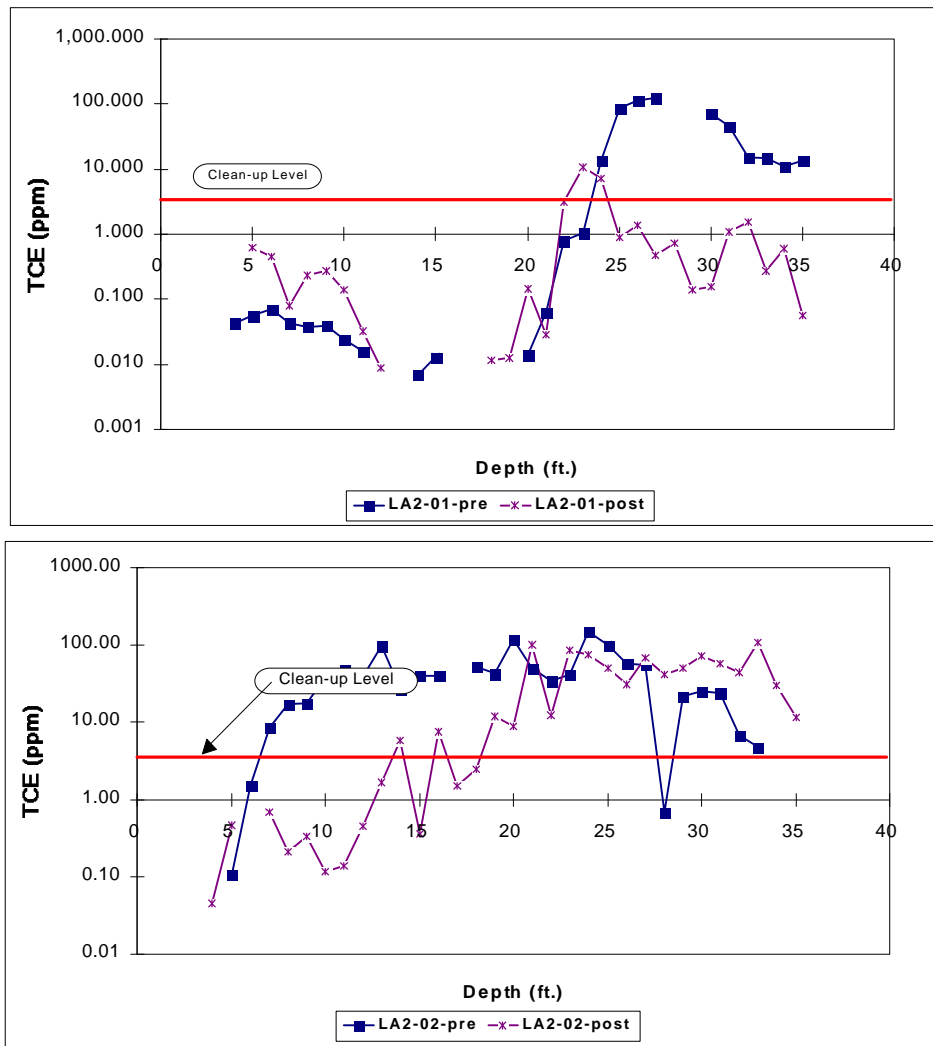
Depth(ft)	L2A-03-pre	L2A-03-inter	L2A-03-post
4	0.00	0.09	0.00
5	0.51	0.28	0.00
6	0.39	0.29	0.00
7	0.78	0.10	0.00
8	0.73	0.07	0.00
9		0.07	0.01
10	0.94	0.07	0.01
11	5.16	0.02	0.01
12	2.34	0.03	0.05
13	4.01	0.00	0.02
14	2.18	0.02	0.00
15	2.64	0.13	0.00
16		0.08	0.00
17		2.31	0.06
18	17.68	3.45	0.04
19	31.91	2.91	1.07
20	357.07	1.14	0.07
21			0.06
22			0.00
23	218.57	27.40	0.41
24	250.47	20.45	1.48
25	199.16	7.44	1.30
26	101.41	3.05	0.61
27	64.48	0.34	0.00
28	63.87	0.06	0.00
29	101.50	0.01	0.00
30	71.07	0.00	0.00
31	57.05	0.00	0.00
32	13.57	0.00	0.00
33	11.27	0.00	0.00
34	17.25	0.03	0.00
35	11.47	0.06	0.01
36	4.83		0.00
37	2.74		0.00
38	4.00		0.00
39	3.75		0.00

<b>Average:</b>	52.3	2.3	0.1
<b>% Removal:</b>	-	95.69%	99.72%

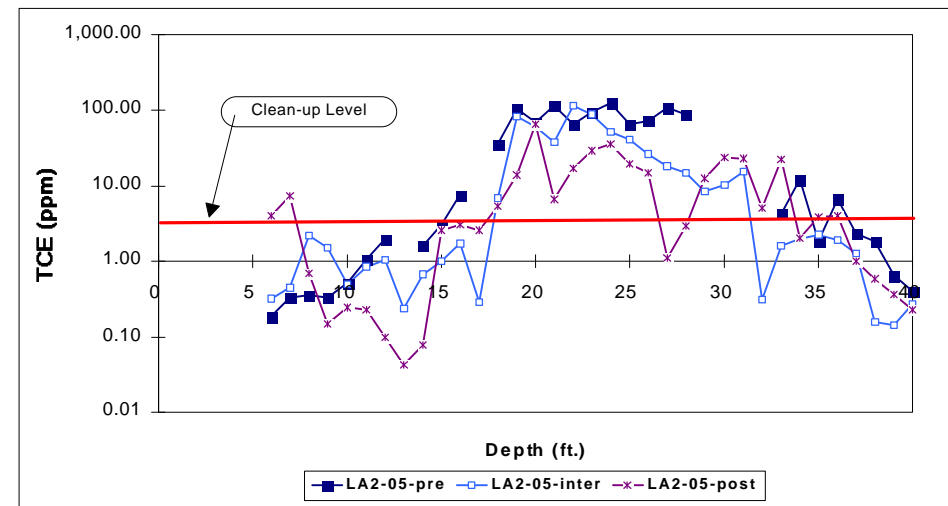
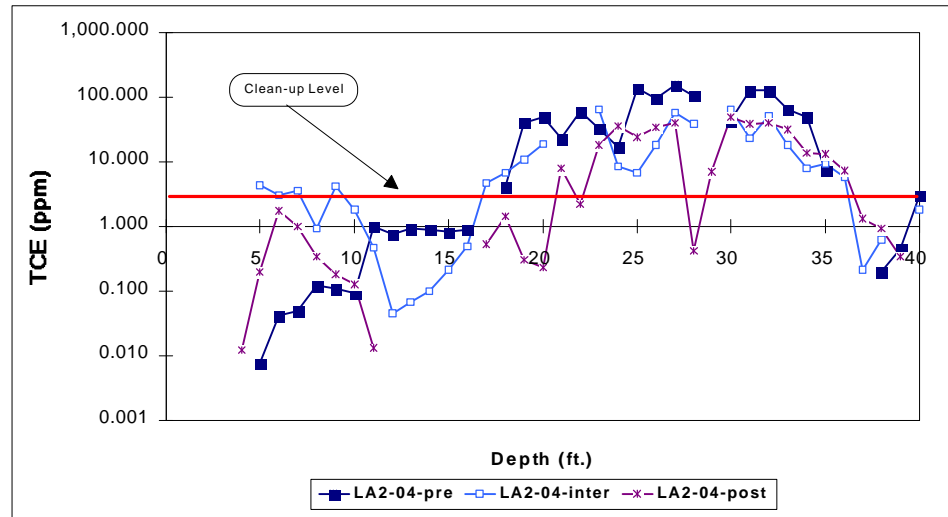
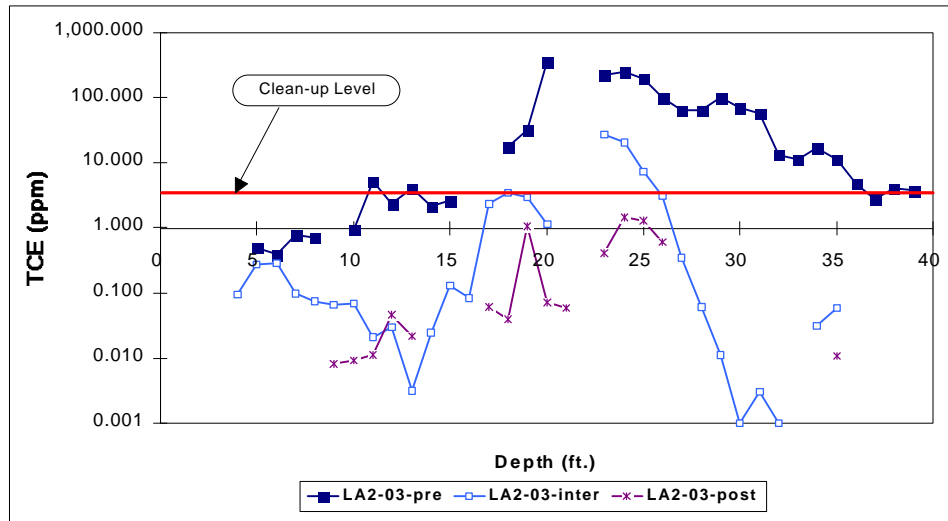
**Table F-7. Summary of Pre-, Inter, and Post-Lasagna™ Soil Samples for L2A-04 and L2A-05 Soil Cores Analyzed by ORNL/GJ (GC/EC).**

Lasagna™ TCE Concentrations (ug/g) As a Function of Depth						
Depth(ft)	L2A-04-pre	L2A-04-inter	L2A-04-post	Depth(ft)	L2A-05-pre	L2A-05-inter
4			0.01			
5	0.01	4.41	0.19	5		
6	0.04	3.07	1.73	6	0.19	0.32
7	0.05	3.58	0.99	7	0.33	0.45
8	0.12	0.92	0.34	8	0.36	2.17
9	0.11	4.21	0.18	9	0.33	1.52
10	0.09	1.81	0.13	10	0.52	0.50
11	1.00	0.48	0.01	11	1.08	0.85
12	0.77	0.04	0.00	12	1.98	1.04
13	0.91	0.07	0.00	13		0.23
14	0.90	0.10	0.00	14	1.61	0.67
15	0.82	0.22	0.00	15	3.11	1.02
16	0.89	0.49	0.00	16	7.54	1.72
17		4.64	0.52	17		0.29
18	4.11	6.67	1.44	18	35.11	6.97
19	40.23	10.66	0.30	19	106.31	81.76
20	48.59	19.04	0.23	20	70.02	60.72
21	23.17		7.81	21	117.89	38.00
22	59.95		2.21	22	66.43	116.41
23	32.32	65.82	17.99	23	95.27	89.90
24	16.59	8.60	35.96	24	123.37	51.85
25	136.42	6.66	23.99	25	65.10	40.93
26	94.37	17.91	34.17	26	73.27	26.53
27	152.03	56.33	39.91	27	108.90	18.21
28	102.22	38.64	0.41	28	88.59	14.89
29			7.13	29		8.36
30	43.64	63.98	49.35	30		10.22
31	129.40	23.13	38.57	31		15.35
32	129.33	50.36	39.96	32		0.31
33	63.68	17.88	31.26	33	4.35	1.60
34	48.92	8.01	13.69	34	12.27	1.99
35	7.14	9.38	13.37	35	1.87	2.25
36	0.00	5.66	7.23	36	6.59	1.88
37	0.00	0.21	1.31	37	2.30	1.29
38	0.20	0.62	0.94	38	1.82	0.16
39	0.44	0.00	0.34	39	0.65	0.14
40	3.09	1.80		40	0.41	0.27
						0.15
<b>Average:</b>	33.6	12.8	10.3		34.4	16.2
<b>% Removal:</b>	-	61.80%	69.25%		-	52.78%
	<b><u>Average Concentration for 03, 04, &amp; 05 Pre-Lasagna:</u></b>					<b><u>40.1</u></b>
	<b><u>Average Concentration for 03, 04, &amp; 05 Inter-Lasagna:</u></b>					<b><u>10.4</u></b>
	<b><u>Average Concentration for 03, 04, &amp; 05 Post-Lasagna:</u></b>					<b><u>6.6</u></b>
	<b><u>Average Concentration for 01 - 05 Pre-Lasagna:</u></b>					<b><u>36.0</u></b>
	<b><u>Average Concentration for 01 - 05 Inter-Lasagna:</u></b>					<b><u>10.4</u></b>
	<b><u>Average Concentration for 01 - 05 Post-Lasagna:</u></b>					<b><u>9.0</u></b>
	<b><u>Target:</u></b>					<b><u>5.6</u></b>

**Figure F-1. Summary of Pre-, and Post-Lasagna™ Soil Samples for L2A-01 and L2A-02 Soil Cores Analyzed by ORNL/GJ (GC/EC).**



**Figure F-2. Summary of Pre-, Inter-, and Post-Lasagna™ Soil Samples for L2A-03, L2A-04 and L2A-05 Soil Cores Analyzed by ORNL/GJ (GC/EC).**





## Search for Chlorinated Degradation Products

While degradation of TCE below 5.6 ppmw ( $\mu\text{g}$  TCE/g soil) is the primary goal of this demonstration, it is also important to demonstrate that the high concentrations of TCE have not been replaced by equally high concentrations of other hazardous organic compounds such as *cis*-dichloroethylene or vinyl chloride. These two compounds are intermediate degradation products which are formed before the final products, acetylene, ethane, and ethylene, are formed. As discussed above, an alternate purpose for the purge-and-trap analysis of soil is to determine whether these two degradation products can be detected.

These analyses were conducted using purge-and-trap instrumentation since the electron capture detector used for TCE analyses does not have sensitivities large enough to detect these compounds at low concentrations. The search for these chlorinated compounds is further complicated since the soil samples contain significant (although low parts-per-million) concentrations of TCE which make low level analyses of other compounds problematic when using expensive and sensitive GC/MS instrumentation.

The initial set of split samples analyzed by LMUS showed only two samples which contained *cis*-

dichloroethylene. However, since the relatively large concentrations of TCE in the samples required these water samples be diluted many fold, the lack of detection coupled with the high detection limits for vinyl chloride and *cis*-dichloroethylene did not allow conclusions to be drawn on whether complete degradation of chlorinated products was occurring.

Therefore Weston Laboratories was asked to perform the split purge-and-trap soil analyses using a more robust GC/photoionization detector which had a larger dynamic range and good sensitivities for *cis*-dichloroethylene and vinyl chloride. The results of these analyses are shown in Table F-8. All soil concentrations were calculated from water concentrations, assuming a soil density of 2.0  $\text{g}/\text{cm}^3$  and a vial total volume of 40  $\text{cm}^3$ .

*cis*-Dichloroethylene was detected in only four of the twenty soil samples and at concentrations below 1 ppmw ( $\mu\text{g}$  DCE/g soil). Vinyl chloride was not detected in any of the soil samples above a limit of detection of 0.1 or 0.2 ppmw ( $\mu\text{g}$  VC/g soil). This clearly demonstrates that the TCE has not been converted to a higher concentration of either of the intermediate chlorinated compounds, and that the degradation of TCE to acetylene, ethane, and ethylene, is occurring as predicted. A later subsection of Section F will further discuss the iron dechlorination of TCE to these light  $\text{C}_2$  hydrocarbons and the design of the treatment zones to insure complete degradation to these products.

**Table F-8. Summary of TCE, DCE, and VC Concentrations Measured in Post-Lasagna™ Soil Samples Analyzed by Weston (GC/PID).**

Sample ID	Location	Water Conc. (ppm)			Soil Conc. (ppm)			Soil Wt. (g)	Water Volume (mL)
		TCE	DCE	VC	TCE	DCE	VC		
2015-PT	L2A-03FS-019	0.12	0.011	<0.01	1.60	0.15	<0.1	2.9	38.6
2020-PT	L2A-03FS-023	0.036	<0.01	<0.01	0.34	<0.1	<0.1	4.0	38.0
2030-PT	L2A-03FS-032	<0.01	<0.01	<0.01	<0.1	<0.1	<0.1	3.5	38.3
2037-PT	L2A-03FS-039	<0.01	<0.01	<0.01	<0.1	<0.1	<0.1	4.0	38.0
2050-PT	L2A-04FS-014	<0.01	<0.01	<0.01	<0.1	<0.1	<0.1	5.1	37.5
2063-PT	L2A-04FS-024	0.51	<0.02	<0.02	6.55	<0.2	<0.2	3.0	38.5
2079-PT	L2A-04AFS-015	<0.01	<0.01	<0.01	<0.1	<0.1	<0.1	4.8	37.6
2098-PT	L2A-04AFS-032	5.6	<0.02	<0.02	48.11	<0.2	<0.2	4.4	37.8
2107-PT	L2A-04AFS-039	0.052	<0.01	<0.01	0.49	<0.1	<0.1	4.0	38.0
2120-PT	L2A-05FS-015	0.28	0.085	<0.01	2.41	0.73	<0.1	4.4	37.8
2141-PT	L2A-05FS-024	2.3	<0.02	<0.02	21.85	<0.2	<0.2	4.0	38.0
2158-PT	L2A-05FS-040	0.031	<0.01	<0.01	0.16	<0.1	<0.1	7.1	36.5
2181-PT	L2A-01FS-024	0.24	<0.01	<0.01	1.05	<0.1	<0.1	8.2	35.9
2212-PT	L2A-10FS-015	0.095	<0.01	<0.01	0.62	<0.1	<0.1	5.7	37.2
2228-PT	L2A-10FS-028	3.8	<0.02	<0.02	27.90	<0.2	<0.2	5.1	37.5
2244-PT	L2A-02FS-016	0.54	0.064	<0.01	5.41	0.64	<0.1	3.8	38.1
2258-PT	L2A-02FS-028	5.4	<0.02	<0.02	42.30	<0.2	<0.2	4.8	37.6
2272-PT	L2A-02FS-040	0.033	<0.01	<0.01	0.22	<0.1	<0.1	5.5	37.3
2294-PT	L2A-02FS-020	7.8	0.022	<0.02	62.48	0.18	<0.2	4.7	37.7
2299-PT	L2A-11FS-024-D	14	<0.02	<0.02	120.27	<0.2	<0.2	4.4	37.8

## Water Sample Results

In the full scale remediation of a contaminated site, one parameter which can be used to evaluate the effectiveness of the process can be the concentration of TCE in water from wells which have been drilled into the site. However, these measurements can only be used if the only source of TCE is from the volume of soil which is being cleaned up. The present subsection describes water sample results from wells which were drilled in the Phase IIa site.

Figures F-3 and F-4 show all TCE concentrations measured for all wells near the soil cores. The vast majority of samples were taken at locations PZ-2A-03, PZ-2A-04, and PZ-2A-05. (Only partial data is available for the remaining wells due to inability to obtain water and the low priority of this data.) The data from the three different analytical laboratories are shown with different types of bars. The range of concentrations measured are shown as error bars for those samples which were taken in replicate. Tables Appendix D-9 and Appendix D-10 show two tables which summarize all of the data shown in these two figures.

Note that all wells show an initial increase in TCE concentrations, followed by a decrease. The water data shown in these figures is more variable than would normally be expected from the analysis of homogeneous water samples. This could be explained by having very large amounts of free liquid present in the form of droplets in the soil formation which results in highly variable TCE concentrations in the samples taken. Sampling technique may have also been an important issue, and analyzing water which had been collected at near 70 °C could also have led to the variable TCE concentrations.

### TCE Pore Water Concentrations

TCE adsorption has been measured for Paducah soil and was reported in Phase I topical report for Tasks #12 and 13<sup>13</sup>. The Langmuir adsorption

isotherm for TCE at room temperature was measured to be:

$$C_s = 4.53 C_w / (4.73 + C_w)$$

where:  $C_s$  is TCE concentration adsorbed on the soil, in mg TCE/kg soil, and  $C_w$  is TCE concentration in pore water in equilibrium with TCE adsorbed on soil, in mg TCE/L water.

Mass balance for TCE based on the Paducah soil properties of 2 g/cm<sup>3</sup> soil density and 0.4 soil porosity can be calculated from:

$$\text{Total TCE in soil} = 0.8 * C_s + 0.2 * C_w$$

Combining the mass balance equation with the adsorption isotherm, we can arrive at the following table showing the distribution of TCE in pore water and TCE adsorbed on soil for a given value of total TCE, measured as mg TCE per kg of soil (including both soil and pore water).

**Table F-9. Distribution of TCE in Water and on Soil, Using the Langmuir Isotherm Data from Phase I**

Total TCE (ppmw or µg/g soil)	TCE in water (ppmw or µg/mL water)	TCE on soil (ppmw or µg/g soil)
1	1.2	0.9
5.7	14.8	3.4
10	34.1	4.0
20	82.9	4.3
50	232.2	4.4
100	482.1	4.5
200	982	4.5
500	2482	4.5

Note that for a total TCE level of 5.6 ppmw (µg/g) in soil, the TCE concentration in water is 14.8 ppmw (µg/g), a factor of 2.6x instead of 5x if there were no adsorption onto the soil. However, when the total TCE level approaches 100 ppmw (µg/g), the approximation of 5x is a good assumption, since the actual value is 4.82.

### TCE Concentrations in Wells 03 - 05

The final TCE concentration measured in Well PZ-2A-03 was approximately 25 ppmw (µg/g), in

<sup>13</sup> Athmer, C. J., Ho, S. V., Hughes, B. M., Sheridan, P. W., Brodsky, P. H., Shapiro, A. P., Thornton, R. F., Salvo, J. J., Schultz, D. S., Landis, R. C., Griffith, R., and Shoemaker, S. H., **Topical Report for Tasks #12 and 13 entitled "Large Scale Field Test of the Lasagna™ Process"** (September 26, 1994 -

May 25, 1996), DOE Contract Number DE-AR21-94MC31185.

Well PZ-2A-04 approximately 50 ppmw ( $\mu\text{g/g}$ ) and in Well PZ-2A-05 approximately 150 ppmw ( $\mu\text{g/g}$ ). If one assumes that these well water samples are at the same concentrations as the water in the pores of the clay formation, and the water concentrations from the Langmuir isotherm shown in Table F-9, a total TCE concentration of 5.6 ppmw ( $\mu\text{g/g}$ ) which contains 20% pore water, would contain water with a TCE concentration of 15 ppmw ( $\mu\text{g/g}$ ). This concentration is approximately 30% of the final concentrations measured for PZ-2A-03 and PZ-2A-04. The PZ-2A-05 measured concentrations are even higher than would be predicted from the soil concentration data. An explanation for this discrepancy may be that TCE migrated from outside the Phase IIa site.

As mentioned earlier, the assumption that the water concentration can be used to monitor the cleanup of the clay is based upon the assumption that

the only source of TCE in the water is pore water from the clay. From the soil TCE concentration profiles shown in Figure C-5, carbon samples, and groundwater samples taken in this test area, it was determined that there is likely a very large source of TCE which could be transported into the Phase IIa test site wells from the surrounding contamination through a permeable sand layer located at a depth of 20 to 25 ft. (6.1 - 7.6 meter); therefore, the well water TCE concentrations may not be a good indicator of soil cleanup during this test if TCE is migrating from nearby sources of TCE. It is for this reason that the water concentrations are used in this study only as an indicator of soil cleanup, but are not the primary indicators. The actual soil TCE concentrations should be used to indicate success of the *Lasagna*<sup>TM</sup> technology as required by the Commonwealth of Kentucky and mentioned in the QAPP and an earlier section of this report.

**Figure F-3. Summary of TCE Concentrations Measured in Wells 03, and 04 Analyzed by (GC/EC).**

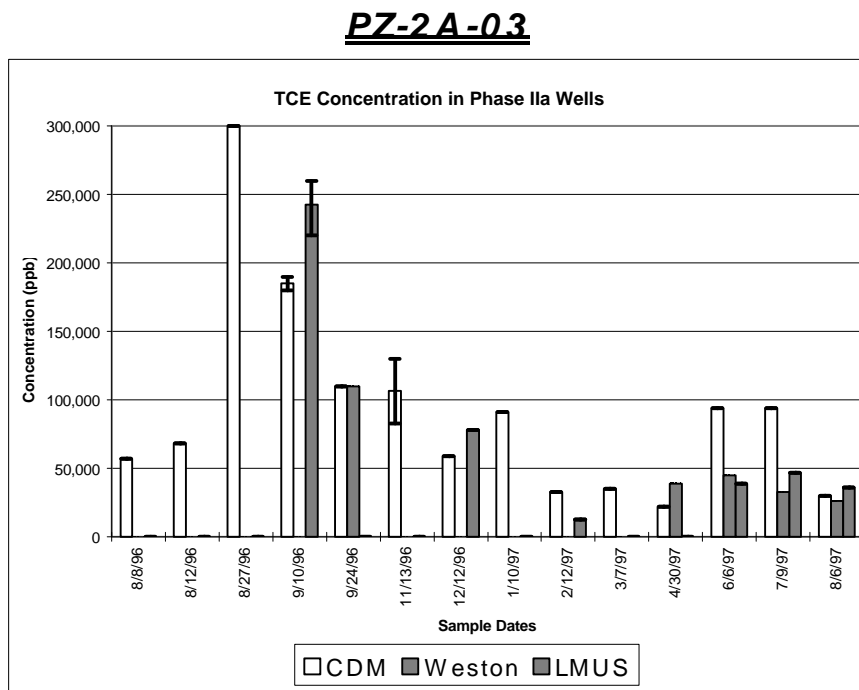


Figure F-3 (cont'd). Summary of TCE Concentrations Measured in Wells 03, and 04 Analyzed by (GC/EC).

**PZ-2A-04**

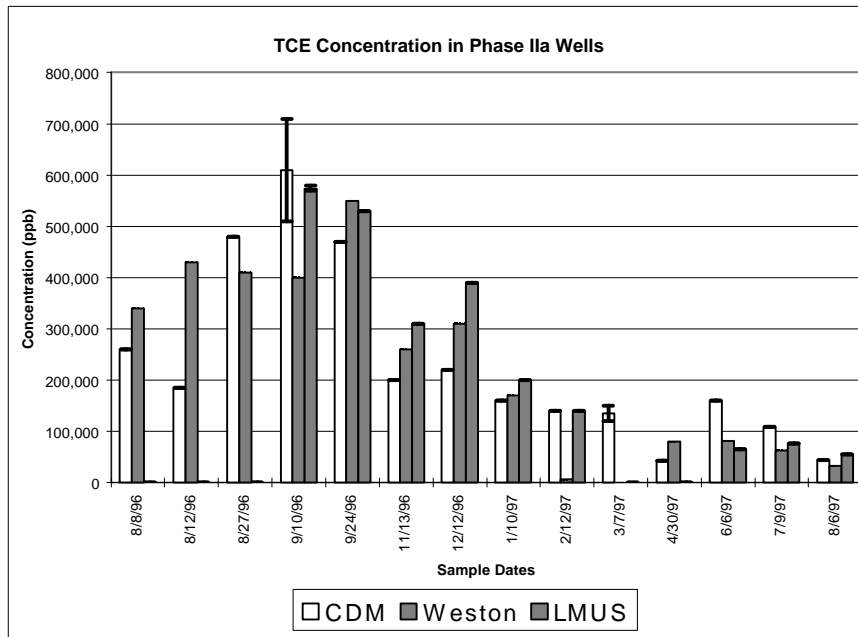
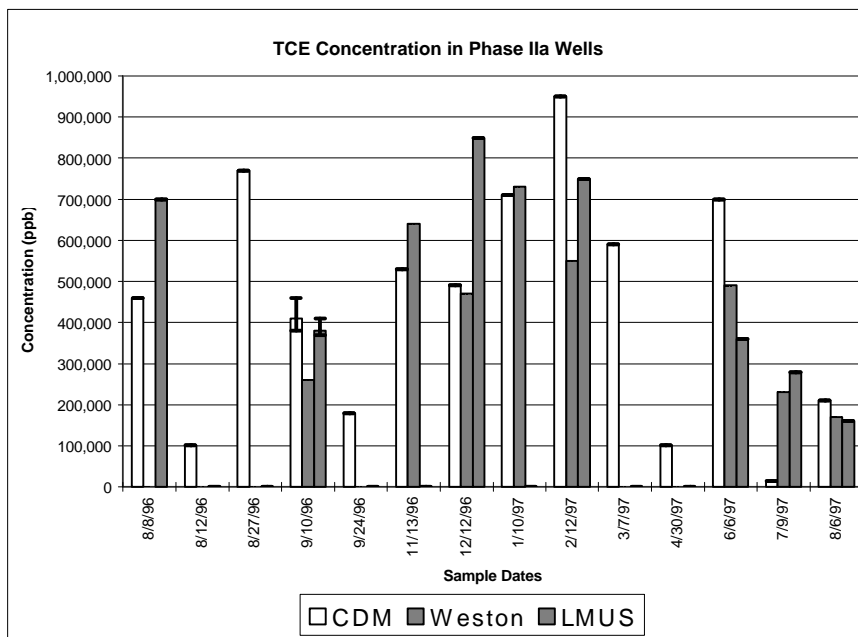


Figure F-4. Summary of TCE Concentrations Measured in Well 05 Analyzed by (GC/EC).

**PZ-2A-05**



## Carbon Sample Results

As shown in Table F-3, almost 500 discrete carbon samples were analyzed in conjunction with the Phase IIa test. These data were taken in an effort to determine whether the *Lasagna*<sup>TM</sup> team could use this type of sampling to monitor the progress of the field test. However, due to the very large amount of DNAPL the TCE concentrations in carbon were so large that they were only used in a qualitative manner.

Since the carbon extract concentrations were several orders of magnitude larger than those from the soil extracts, many more extract dilutions were required in order to accurately measure the TCE concentrations in the carbon extracts. These large numbers of dilutions may have led to confusion by the field laboratory when they were preparing to send duplicate extracts to Weston Laboratories for analysis. Diluted extracts may have inadvertently been sent to Weston. However, these small numbers of errors (if this is the proper explanation)

would not affect any of the measurements upon which the RCI Participants are basing their conclusions on the success of the *Lasagna*<sup>TM</sup> Phase IIa field study. (No carbon data is used in any of the conclusions used in this final report.)

## Flux Chamber Results

Flux chambers were placed over the Phase IIa site in an effort to detect and quantify TCE if it was being volatilized from the clay. Table F-10 summarizes the results obtained in December 1996. A second set of samples were also taken in January, but the QA/QC results indicated that no TCE could be detected in spiked tubes and therefore we could not validate the data obtained (see Subsection 3.2.2 in Section G). It is recommended that during the full cleanup of the site, additional flux chamber measurements be made to confirm that negligible amounts of TCE are being lost to the air during the *Lasagna*<sup>TM</sup> process.

**Table F-10. Summary of TCE Concentrations Measured December 1996 Flux Chamber Measurements.**

<b>Sample ID</b>	<b>Description</b>	<b>Micrograms/tube</b>	<b>% Recovery</b>
FC-1 Front	12 liters from FC	< 14	
FC-2 Front	12 liters from FC	< 14	
FC-3 Front	12 liters from FC	< 14	
FC-4 Front	12 liters from FC	< 14	
FC-5 Front	12 liters from FC	< 14	
FC-6 Front	12 liters from FC	< 14	
FC-7 Front	12 liters from FC	< 14	
FC-8 Front	12 liters from FC	< 14	
FC-9 Front	12 liters from FC	< 14	
FC-10 Front	12 liters from FC	< 14	
FC-11 Front	12 liters from FC	< 14	
FC-12 Front	12 liters from FC	< 14	
FC-13A	100 ug spiked tube	70	
FC-13B	100 ug spiked tube	75	
FC-13C Front	12 liters from FC	< 14	
FC-13D	Blank Tube	< 14	
BLK	Blank Tube	< 14	
BLK BS	Recovery Study		73%

## Acetylene, Ethane, and Ethylene Degradation Products

One of the important technical goals of the Phase IIa field study is to document that degradation of TCE, *cis*-DCE and VC are occurring and that the C<sub>2</sub> hydrocarbons

acetylene, ethane, and ethylene being formed in the field test area, while not being present in wells which are outside the Phase IIa test area. Figure C-4 shows the locations of these sampling locations. The data from these analyses are shown in Table F-11 and Figure F-5.

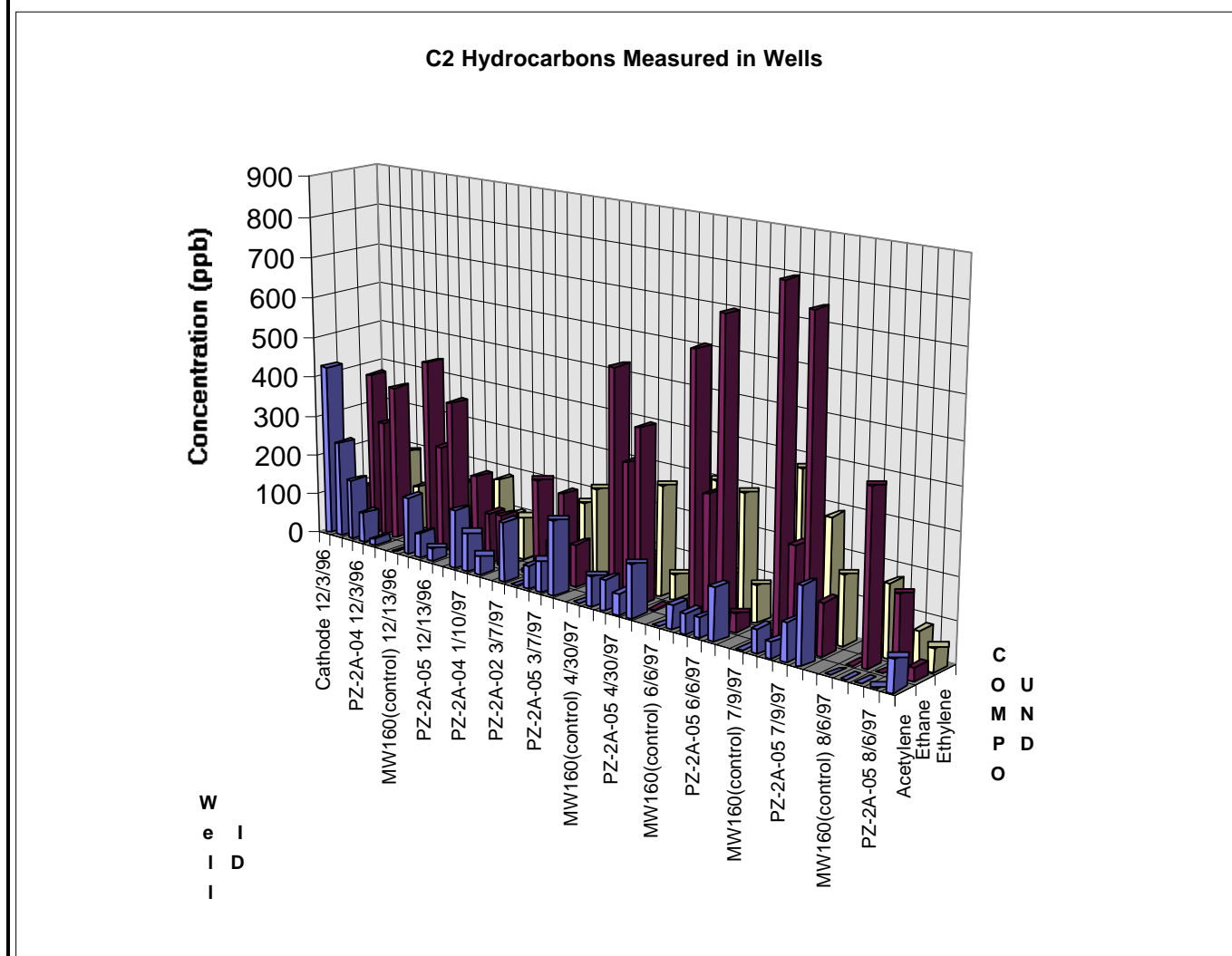
As can be seen, there is a rise in concentration over the time from December 1996 through July 1997, and a

decrease in concentration from July to August 1997. This is consistent with the formation of these products from the iron dechlorination of TCE. The next section

further documents how the treatment zones were designed to assure that complete in-situ degradation of TCE occurs.

**Table F-11. Summary of C<sub>2</sub> Hydrocarbon Degradation Products Measured in Water Samples.**

Date	Sample ID	Concentration (ppb)					C <sub>2</sub> Ratio
		Acetylene	Ethane	Ethylene	cis-DCE	TCE	
	<i>DNAPL TCE Degradation</i>	-	-	-	-	-	>10/0.2/1.0
	<i>Dissolved TCE Degradation</i>	-	-	-	-	-	<0.1/1.0/1.0
3-Dec	<i>Cathode 12/3/96</i>	426	38	168	NA	NA	2.5/0.2/1.0
3-Dec	<i>PZ-2A-02 12/3/96</i>	239	109	140	NA	NA	1.7/0.7/1.0
3-Dec	<i>PZ-2A-03 12/3/96</i>	149	405	177	NA	NA	0.8/2.3/1.0
3-Dec	<i>PZ-2A-04 12/3/96</i>	74	289	198	NA	NA	0.4/1.5/1.0
3-Dec	<i>PZ-2A-05 12/3/96</i>	15	385	113	NA	NA	0.1/3.4/1.0
13-Dec	<i>MW160(control) 12/13/96</i>	ND	ND	ND	ND	73,000	-
13-Dec	<i>PZ-2A-03 12/13/96</i>	144	466	223	ND	78,000	0.6/2.1/1.0
13-Dec	<i>PZ-2A-04 12/13/96</i>	61	262	153	ND	390,000	0.4/1.7/1.0
13-Dec	<i>PZ-2A-05 12/13/96</i>	33	379	165	ND	850,000	0.2/2.3/1.0
10-Jan	<i>PZ-2A-03 1/10/97</i>	143	211	184	NA	NA	0.8/1.1/1.0
10-Jan	<i>PZ-2A-04 1/10/97</i>	94	124	102	ND	200,000	0.9/1.2/1.0
10-Jan	<i>PZ-2A-05 1/10/97</i>	45	127	103	NA	NA	0.4/1.2/1.0
7-Mar	<i>PZ-2A-02 3/7/97</i>	147	ND	108	NA	NA	1.4/0.0/1.0
7-Mar	<i>PZ-2A-03 3/7/97</i>	ND	239	119	NA	NA	0.0/2.0/1.0
7-Mar	<i>PZ-2A-04 3/7/97</i>	57	85	70	NA	NA	0.8/1.2/1.0
7-Mar	<i>PZ-2A-05 3/7/97</i>	75	221	179	NA	NA	0.4/1.2/1.0
7-Mar	<i>Cathode 3/7/97</i>	184	105	221	NA	NA	0.8/0.5/1.0
30-Apr	<i>MW160(control) 4/30/97</i>	ND	ND	ND	NA	NA	-
30-Apr	<i>PZ-2A-03 4/30/97</i>	72	551	252	NA	NA	0.3/2.2/1.0
30-Apr	<i>PZ-2A-04 4/30/97</i>	74	334	92	NA	NA	0.8/3.6/1.0
30-Apr	<i>PZ-2A-05 4/30/97</i>	50	424	268	NA	NA	0.2/1.6/1.0
30-Apr	<i>Cathode 4/30/97</i>	127	ND	62	NA	NA	2.0/0.0/1.0
6-Jun	<i>MW160(control) 6/6/97</i>	ND	ND	ND		61,000	-
6-Jun	<i>PZ-2A-03 6/6/97</i>	59	629	311		45,000	0.2/2.0/1.0
6-Jun	<i>PZ-2A-04 6/6/97</i>	49	306	94		81,000	0.5/3.3/1.0
6-Jun	<i>PZ-2A-05 6/6/97</i>	49	720	297		490,000	0.2/2.4/1.0
6-Jun	<i>Cathode 6/6/97</i>	126	43	89		2,500	1.4/0.5/1.0
9-Jul	<i>MW160(control) 7/9/97</i>	ND	ND	ND		61,000	-
9-Jul	<i>PZ-2A-03 7/9/97</i>	58	816	385		47,000	0.2/2.1/1.0
9-Jul	<i>PZ-2A-04 7/9/97</i>	37	236	66		76,000	0.7/3.6/1.0
9-Jul	<i>PZ-2A-05 7/9/97</i>	91	763	290		280,000	0.3/2.6/1.0
9-Jul	<i>Cathode 7/9/97</i>	183	123	164		2,300	1.1/0.8/1.0
6-Aug	<i>MW160(control) 8/6/97</i>	ND	ND	ND		53,000	-
6-Aug	<i>PZ-2A-03 8/6/97</i>	ND	407	172		36,000	-/2.4/1.0
6-Aug	<i>PZ-2A-04 8/6/97</i>	ND	ND	ND		55,000	-
6-Aug	<i>PZ-2A-05 8/6/97</i>	ND	186	85		160,000	-/2.2/1.0
6-Aug	<i>Cathode 8/6/97</i>	76	34	55		850	1.4/0.6/1.0

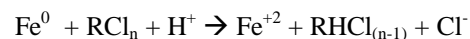
Figure F-5. Summary of C<sub>2</sub> Hydrocarbon Degradation Products Measured in Water Samples.

## Dechlorination of TCE in the Phase IIa Field Test

### Introduction

The dechlorination of trichloroethylene (TCE) using zero-valent iron has been studied extensively. A portion of the Phase IIa study involved laboratory studies of the iron dechlorination reaction<sup>14, 15</sup>. The effectiveness of

this approach has been demonstrated either as permeable reactive walls or packed bed reactors at many field sites. Laboratory studies on the reaction pathways and the rates of reactions for TCE and other chlorinated ethylenes have been studied by many groups. It is believed that the dechlorination goes through a step-wise process in which one chlorine atom is removed each time:



where R stands for an aliphatic hydrocarbon.

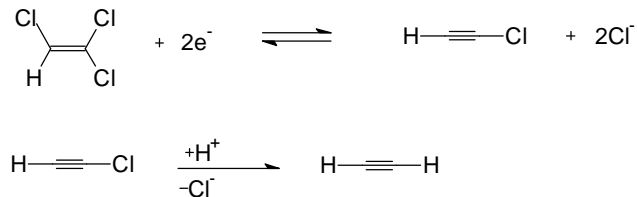
The mechanism of the dechlorination process is thought to be a surface reaction and to depend on the surface area. This surface dependence shows the type of

<sup>14</sup> Shapiro, A. P., Sivavec, T. M., and Principe, J. M., Topical Report for Task #3.2 Entitled, "Modeling and Iron Dechlorination Studies"(September 26, 1994 - August 31, 1997), Final Phase IIa Topical Report in preparation.

<sup>15</sup> Orth, R. G., Dauda, T. and McKenzie, D. E., Topical Report for Task #3.3 entitled, "Iron Dechlorination Studies"(September 26, 1994 - August 31, 1997), Final Phase IIa Topical Report in preparation.

results that would be expected for site limited reaction kinetics<sup>16, 17, 18</sup>.

Studies on carbon tetrachloride dechlorination showed formation of chloroform and methylene chloride sequentially<sup>19</sup>. The mechanism was suggested to be a direct electron transfer from the iron. The process is considered by Matheson<sup>19</sup> to be a corrosion process with the Fe forming Fe (II). Roberts et al<sup>20</sup> and Campbell et al<sup>21</sup> have put forth reaction pathways for the formation of the observed volatile hydrocarbons from dechlorination of TCE and PCE which combine  $\beta$  elimination and hydrogenolysis. The  $\beta$  elimination path would lead to acetylene. This would be followed by hydrogenolysis. This reaction path, which is summarized below, would go through an intermediate of chloroacetylene.



The pathways are supported by the observed products and calculations. It has been observed by many

researchers<sup>21, 22, 23</sup> that the observed products are ethylene, ethane and acetylene with minor amounts of higher C<sub>3</sub> to C<sub>6</sub> hydrocarbons. Along with these hydrocarbons minor amounts of vinyl chloride and isomers of dichloroethylenes have been observed.

### Rate of Dechlorination

The rate of TCE dechlorination was measured by GE researchers using the Paducah groundwater both in batch (stirred pot) and continuous (packed bed) mode. The basic rate constant determined from these measurement is  $k = 0.08 \text{ ccH}_2\text{O/hr-g Fe}$ , which for a packing density of  $2.41 \text{ g Fe/cm}^3$  and 0.614 packing porosity translated into a first order rate constant of  $0.31 \text{ hr}^{-1}$  or a half life of 2.2 hr at room temperature. The GE study also looked at the dechlorination of TCE daughter products dichloroethylene (DCE) and vinyl chloride (VC), which actually degrade at lower rates than TCE. The 25 °C half lives of DCE and VC are 5.2 and 3.3 hr, respectively. The following paragraphs show how these data were used in the Phase IIa design to ensure complete destruction of TCE as well as these daughter products, especially VC.

### Design of treatment zones

Key considerations in the design of the treatment zones for Phase IIa were:

- sufficient residence time for complete destruction of TCE and its daughter products, especially VC.
- optimum level of iron, allowing for ample amount of iron to degrade all the TCE present in the soil and last for the duration of the test, but at minimum cost.
- ease of installation and minimization of TCE loss through volatilization in the treatment zones.

At the targeted voltage gradient of 0.25 volt/cm, the velocity of water moving through the Paducah clay soil is approximately 0.32 cm/day. Calculations based on the GE rate data showed that a treatment zone thickness of 2 inches containing 100% iron was much in excess for

<sup>16</sup> Burris, D.R.; Campbell, T.J.; Manoranjan, V.S. 1995) Sorption of trichloroethylene and tetrachloroethylene in a batch reactive iron water system. *Environ. Sci. Technol.* 29(11): 2850-2855.

<sup>17</sup> Johnson, T.L.; Scherer, M.M.; Tratnyek, P.G. 1996. Kinetics of halogenated organic compound degradation by iron metal. *Environ. Sci. Technol.* 30(8): 2634-2640.

<sup>18</sup> Scherer, M.M.; Tratnyek, P.G. 1995. Dechlorination of carbon tetrachloride by iron metal: Effect of reactant concentration. 209th National Meeting, Anaheim, Ca, American Chemical Society, Vol. 35, No1. pp 805-806.

<sup>19</sup> Matheson, L. J. ; Tratnyek, P. G. 1994. Reductive dehalogenation of chlorinated methanes by iron metal.. *Environ. Sci. Technol.* 28(12), 2045-2053.

<sup>20</sup> Roberts, A.L.; Totten, A.; Arnold, W.A.; Burris, D.R.; Campbell, T.J. 1996. Reductive elimination of chlorinated ethylenes by zero valent metals. *Environ. Sci. Technol.* 30(8), 2654-2659.

<sup>21</sup> Campbell, T.J.; Burris, D.R.; Roberts, A.L.; Wells, J.R. 1997. Trichloroethylene and tetrachloroethylene reduction in metallic iron-water-vapor batch system. *Environ. Toxicol. Chem.* 16(4), 625-630.

<sup>22</sup> Senzaki, T., AND Y. Kumagai 1989. Removal of chlorinated organic compounds from wastewater by reduction process: II. Treatment of trichloroethylene with powder. *Kogyo Yosui.* 369, 19-25.

<sup>23</sup> Orth, W.S.; Gillham, R.W. 1996. Dechlorination of trichloroethene in aqueous solution using Fe<sup>0</sup>. *Environ. Sci. Technol.*, 30:60-71.



complete destruction of TCE and its daughter products. It was therefore decided to mix iron filings with an inert material. Sand and clay were the two obvious options considered. Clay was chosen because it offers a number of advantages. It is more stable in an electroosmotic environment since it has better water retention than sand. It should also minimize the amount of TCE volatilization. Clay easily forms a slurry, and is an inert diluent which will hold the iron particles in place throughout the 45-ft (13.7 meter) depth.

Tables F-16 through F-18 summarize the TCE, DCE, and VC half-lives, % Conversion, and Amount Remaining using a number of assumptions.

Table F-12 shows the estimated half life values for TCE, *cis*-DCE and VC at 25, 40 and 60°C for an 8 volume % iron packing. These rate calculations were based on the iron concentration (g Fe per cm<sup>3</sup> of the reaction zone). For 100% Fe, the Fe concentration is 2.41 g/cm<sup>3</sup>; for 8% volume Fe in clay, the Fe

concentration is 0.5 g/cm<sup>3</sup>. The conversion ratio is thus 2.41/0.5 = 4.82.

Based on these half lives, the amount of TCE, *cis*-DCE and VC converted due to passing through each treatment zone, assuming plug flow for the liquid, is shown in Table F-13.

And, finally, for pore water entering a treatment zone containing 1,000 ppmw (µg/g) TCE, 10 ppmw (µg/g) each *cis*-DCE and VC, the amount of these chemicals in the pore water exiting the treatment zone are summarized in Table F-14. These tables show that the amount of iron present is theoretically more than sufficient to destroy TCE and its daughter products to very low levels in a single pass.

Note that the average soil temperature was to be about 40 °C or greater for most of the test. Additionally, the remediation design calls for operation of at least 2 pore volumes, i.e. each fluid element will pass through treatment zones twice.

**Table F-12. Summary of Half-Lives of Chlorinated C<sub>2</sub> Degradation Products Measured in Water Samples.**

Temperature, °C	Half Life in Hr		
	TCE	<i>cis</i> -DCE	VC
25	10.7	25.3	16.2
40	3.5	8.2	5.2
60	2.4	5.5	3.6

**Table F-13. Summary of % Conversion of Chlorinated C<sub>2</sub> Degradation Products Measured in Water Samples.**

Temperature, °C	% Conversion per Pass		
	TCE	<i>cis</i> -DCE	VC
25	100	99.83	100
40	100	100	100
60	100	100	100

**Table F-14. Summary of ppmw (µg/g) of Chlorinated C<sub>2</sub> Degradation Products Measured in Water Samples.**

Temperature, °C	ppmw (µg/g) per Pass		
	TCE	<i>cis</i> -DCE	VC
25	0.0003	0.0168	0.0005
40	0	0	0
60	0	0	0

## Installation & Operation

The treatment zone materials were installed using the hollow mandrel method to be described in detail in the Topical Report of Task 7.4. Peerless iron filings were mixed with wet kaolin clay to form a slurry that was poured down the center of the hollow mandrel 45 ft (13.7 meter) deep. Three treatment zones were installed between the two electrodes, which were 21 feet apart, 30 feet wide and 45 feet deep. The first treatment zone was 7 feet from the anode, the second one 5 feet away from the first one, and the last one was located two feet from the second one. The original design called for iron content of 8 volume % in clay, as discussed above. However, due to the excess water needed to allow the clay slurry to pour down the mandrel, additional solid was required to increase the consistency and density of the slurry to prevent it from spewing out of the treatment zone once the mandrel was pulled out of the soil. The actual amount of iron filings in the clay slurry was increased to 26 volume % (or 1.61 g Fe/cm<sup>3</sup>), which was more than three times the design level. Also, the clay slurry contained 50 weight % water, much higher than the saturation level of approximately 40% for kaolin clay. It was anticipated that the treatment zones would shrink during the operation due to electroosmotic flow removing the excess water. However, the five feet of gravel on the ground surface was not contaminated; therefore, no treatment zone was needed for this section.

The field test was operated for almost 1 year, except for 3 weeks down time for soil sampling. The detailed schedule for operation is shown in Table F-1. A total electroosmotic flow equivalent to 2.5 pore volumes of the soil between the 2 foot-spaced treatment zones was obtained. The average soil temperature increased from 18 °C to a peak of 60 °C after 5 to 6 months of operation and was reduced to approximately 50 °C until the end of the test. The dechlorination of TCE and its daughter products would therefore be occurring between 40 and 60 °C for the majority of the operation.

## Dechlorination Results

Due to the presence of DNAPLs at various locations at the test site, laboratory experiments were conducted to investigate the dechlorination of TCE. It was found that as long as some water was present, the rate of TCE destruction of DNAPL would proceed at a rate comparable to that with TCE-saturated water. An unexpected outcome was the product distribution. For dissolved TCE, the products observed were primarily ethylene and ethane in equal amounts, with very little

acetylene (see Figure Appendix F-1). A simulated DNAPL situation, however, led to acetylene as the major dechlorination product, followed by ethylene, with very little ethane (see Figure Appendix F-2). This difference in product distribution provides an excellent marker for determining TCE degradation in the field. The two reaction paths are shown schematically in Figure Appendix F-3.

Water samples from various wells within the test site and from a control well (MW 160) were taken eight times during the test. Figure C-4 shows the location of these wells. The results are shown in Table F-11 and plotted in Figure F-5. It can be seen immediately that, throughout the test, samples from the control well showed high levels of TCE but non-detectable levels of the product gases ethylene, ethane and acetylene. By contrast, all the samples from wells in the test zone showed high levels of the three product gases. For example, on December 13, 1996, the control sample showed 73 ppmw (µg/g) TCE and none of the other gases, whereas well PZ-2A-03 had comparable TCE level (78 ppmw (µg/g)) and 144 ppbw (µg/kg) acetylene, 466 ppbw (µg/kg) ethane, and 223 ppbw (µg/kg) ethylene. The same is true for intermediate samples as well as for the final samples taken on August 6, 1997. It is thus clear that dechlorination of TCE was definitely occurring in the test zone, but not in the control area.

Additionally, the product distribution, while highly variable, seemed very consistent with the combination of the two pathways depicted above for TCE dechlorination. High levels of acetylene, which suggest that dechlorination of DNAPL was dominating, accompanied intermediate levels of ethylene and much lower levels of ethane. On the other hand, low levels of acetylene, which suggests dechlorination of dissolved TCE was dominating, accompanied much higher levels of ethane and intermediate to comparable levels of ethylene.

Notice that acetylene concentrations were measured in most early and intermediate well data, while the last sampling date showed measurable acetylene in only the cathode well. This trend is consistent with the complete absence of DNAPL by the end of the field test on August 6, 1997, further strengthening the argument that the *Lasagna*<sup>TM</sup> process may be used to remediate DNAPLs. It should be noted that high levels of the three product gases were also detected in the cathode water samples, indicating TCE dechlorination was occurring at the cathode as well as the treatment zones. This result is consistent with results from laboratory studies reported earlier.

## G. Treatment Effectiveness Verification

The results of analysis of a large number of water, soil, and carbon samples will be used to evaluate the effectiveness of the *Lasagna*<sup>TM</sup> process. Standard statistical evaluation procedures will be used to determine the accuracy of the obtained data.

### 1.0 Introduction

#### 1.1 Objective

The objective of this report is to assess the usability of the analytical data collected in support of the *Lasagna*<sup>TM</sup> Technology Demonstration Phase IIa. This assessment is needed to ensure that decisions on TCE treatment effectiveness are technically correct. The section will also analyze time trends in TCE concentrations to determine if sufficient data have been collected to determine treatment effectiveness with adequate statistical confidence.

#### 1.2 Quality Assurance Project Plan

A Quality Assurance Plan was prepared in the early phases of *Lasagna*<sup>TM</sup> Phase IIa which provided for *Quality Assurance* and *Quality Control* (QA/QC) activities to ensure data quality.

*Quality Assurance* activities included:

- (1) Routine field and laboratory audits
- (2) Use of standard field and laboratory procedures
- (3) Complete and detailed documentation of project results
- (4) Data review and validation

*Quality Control* included:

Field Sample Collection:

- (1) Field duplicates
- (2) Field equipment rinse blanks
- (3) Field trip blanks

Laboratory Operations:

- (1) Split analyses between independent laboratories with Roy F. Weston as the Reference laboratory
- (2) Intralaboratory replicates
- (3) Blind performance evaluation samples
- (4) Analytical method quality control (calibration, calibration verification, recovery evaluation, etc.)

This report provides an assessment as to the effectiveness of the above QA/QC activities in providing usable data for evaluation of *Lasagna*<sup>TM</sup> technology remedial effectiveness.

### 2.0 Assessment Approach

The data quality assessment was accomplished in three review phases: (1) Field Sample Collection Assessment, (2) Analytical Laboratory Operations Assessment, and (3) Rollup Assessment and Statistical Evaluation of DQOs.

The goal of the *Field Sample Collection Assessment* is to ensure that samples were collected in a manner that is representative of the media being sampled and that minimizes cross-contamination during sample handling and transport. Information reviewed included:

- (1) Field sample collection audit reports
- (2) Field sample duplicate results
- (3) Field trip blank data
- (4) Field equipment rinse blank data

The goal of the *Analytical Laboratory Operations Assessment* is to ensure that analytical data are of sufficient accuracy and precision for making treatment effectiveness decisions. Information reviewed includes:

- (1) Laboratory audit reports
- (2) Split samples results between independent analytical laboratories
- (3) Laboratory duplicate analyses results
- (4) Validation results for the reference laboratory data
- (5) Method QC data for critical decision data (i.e. soil analysis data)

The goal of the *Rollup Assessment and Statistical Evaluation* of DQOs is to look holistically at all weaknesses identified during the previous two assessments and determine if these result in any limitations on data usability for rendering decisions on the effectiveness of the *Lasagna*<sup>TM</sup> process. An interpretation of soil data is also presented to determine what statistical inference can be made concerning treatment effectiveness.

### 3.0 Results

The *Lasagna*<sup>TM</sup> Phase IIa quality assurance/quality control program was fully implemented by August 1996. The following sections describe the results of this QA/QC program and the impacts that these results could have on

data quality. (It should be noted that the first soil sampling event was conducted in March 1996 before the establishment of many of the QA/QC program elements. This sampling, however, was performed by the same staff (ORNL/GJ) using the same methods as the March 1997 and August 1997 soil sampling events.)

The focus of the following assessment is on TCE and not any of the other contaminants that were also measured (e.g. vinyl chloride, ethane, and dichloroethene). This was done since TCE is the basis of remedial effectiveness decisions. Any quality implications derived from the analysis of TCE can be conservatively extended to these other contaminants of concern.

### 3.1 Field Sample Collection Assessment

#### 3.1.1 Sampling Technique Evaluation

The ability to obtain accurate and representative samples was assured through use of trained field sampling teams that follow documented standard operating procedures. The representativeness, and hence acceptability, of the sampling approach was verified by a series of seven onsite field audits and corrective action follow-up reviews. These audits were performed for each type of field sampling including:

- (1) Groundwater sampling conducted by Camp, Dresser, and McKee (CDMFPC),
- (2) Soil core sampling (onsite observations of intermediate-, and post-*Lasagna*<sup>TM</sup>) conducted by ORNL/GJ,
- (3) Air flux chamber sampling conducted by Monsanto, and
- (4) Carbon cassette sampling conducted by ORNL/GJ.

A summary of field audits, key issues observed, and corrective action response is included in Table Appendix E-1. Key issues noted included the use of an improper groundwater sampling technique at the start of the project, the lack of adequate cross contamination control during carbon cassette sampling, and lack of adequate depth measurements during carbon cassette sampling. Each of these problems were corrected early in the sampling effort, as noted in Table Appendix E-1, thus minimizing any potential impact to data quality.

#### 3.1.2 Control of Cross Contamination

Cross contamination of samples was controlled through careful sample handling and packaging to prevent commingling of discrete samples and introduction of nonrepresentative artifacts. Field blank samples were analyzed at an approximate 5% frequency to assess control of cross-contamination; these included

trip blanks and equipment rinse blanks for groundwater, soil, and air flux sampling events.

Field blanks were of four types: (1) trip blanks for groundwater samples consisting of sample vials filled with deionized water, (2) trip blanks for soil boring samples consisting of sample containers filled with hexane only, (3) blank carbon tubes for air flux chamber analysis and (4) equipment rinse blanks for groundwater bailers and soil coring samplers.

Results for groundwater sampling trip blanks, soil sampling trip blanks, blank carbon tubes (for air flux), and equipment rinse blanks (bailers and soil coring equipment) are presented in Table Appendix E-2. These data indicate that cross contamination was adequately prevented during sample handling and processing with all but one result containing less than detectable quantities of TCE. A TCE "hit" of 26 ppb was detected in one trip blank sent to the LMUS laboratory with field groundwater samples. TCE cross contamination at this low level, however, will have no effect on the decision making value of the data.

#### 3.1.3 Field Precision

Field precision is the measure of variability in the results of two field samples collected sequentially from the same field location using the same technique. Variability will result from differences in field sample handling and heterogeneity of the sampled media. Variability will also result from laboratory analysis imprecision. Thus field precision is actually a measure of the total precision of the measurement.

While it is the goal to minimize variability due to field sample handling, significant differences in analyte concentration within the same immediate sampling location and even within the same sample can occur. Thus differences in the results of field duplicates may represent natural heterogeneity of the media and should not necessarily be interpreted as a data quality limitation.

For *Lasagna*<sup>TM</sup> Phase IIa sampling events, field duplicates were collected for soil borings and carbon cassette sampling at a 5% frequency. Replicate volatile organic samples were collected and analyzed during groundwater sampling; however, these were typically sent for interlaboratory comparisons or were used to make differing laboratory dilutions. For this reason, groundwater replicate data are not discussed in this section but will be presented in section 3.2.4. Also, field duplicates were not collected for the air flux study.

Soil field duplicate results are presented in Figure G-1 for the pre, intermediate, and post sampling events. This table indicates that relative percent difference was less than 30% on 60% percent of the duplicate pairs.

**NOTE:** RPDs of less than 30% are generally considered to represent relatively homogenous conditions at sampling location and good field sampling/laboratory analysis technique. For the remaining duplicate pairs, large and varied differences were often noted with relative percent differences as high as 200%. The 200% points are artifacts where one of the two replicates showed no detected amount of TCE. Large variability in replicates occurred for samples that have very low concentrations of TCE. This is not

unusual as analytical imprecision becomes greater as the concentrations approach the instrument limit of detection.

Figure G-2 presents field duplicate results for extracts from carbon cassette sampling. This figure indicates excellent agreement between replicate pairs with only 3 of 28 replicate pairs having relative percent differences greater than 30%.

**Figure G-1a. Field Duplicates - ORNL/GJ Soil Sampling - March 1996**

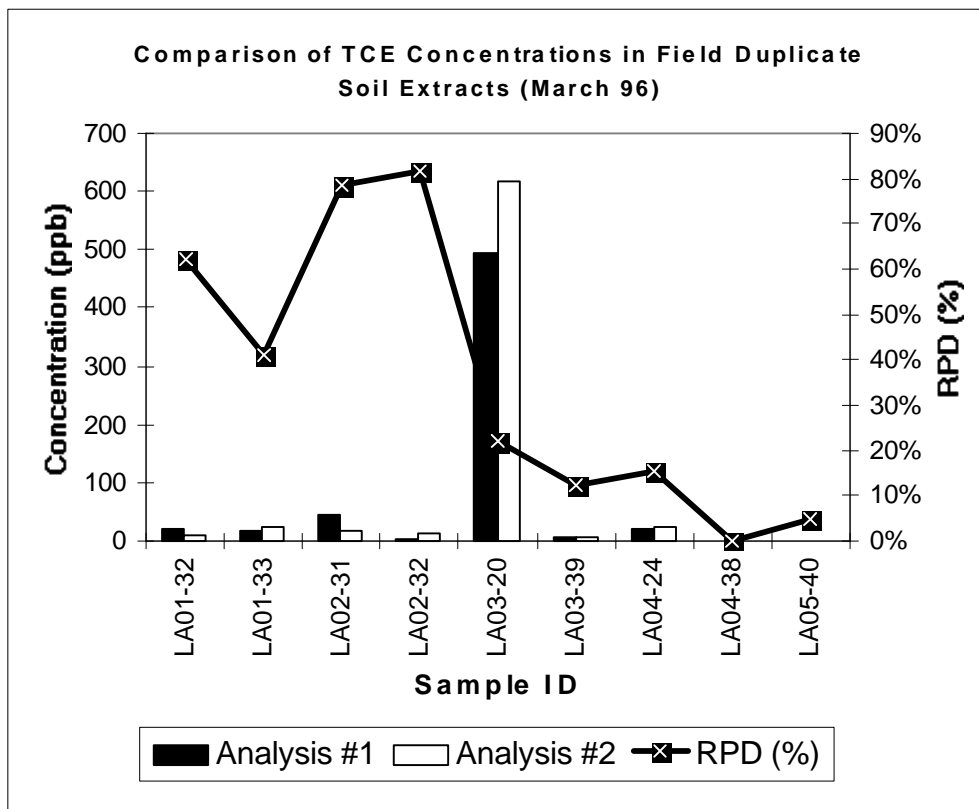


Figure G-1b. Field Duplicates - ORNL/GJ Soil Sampling - March 1997

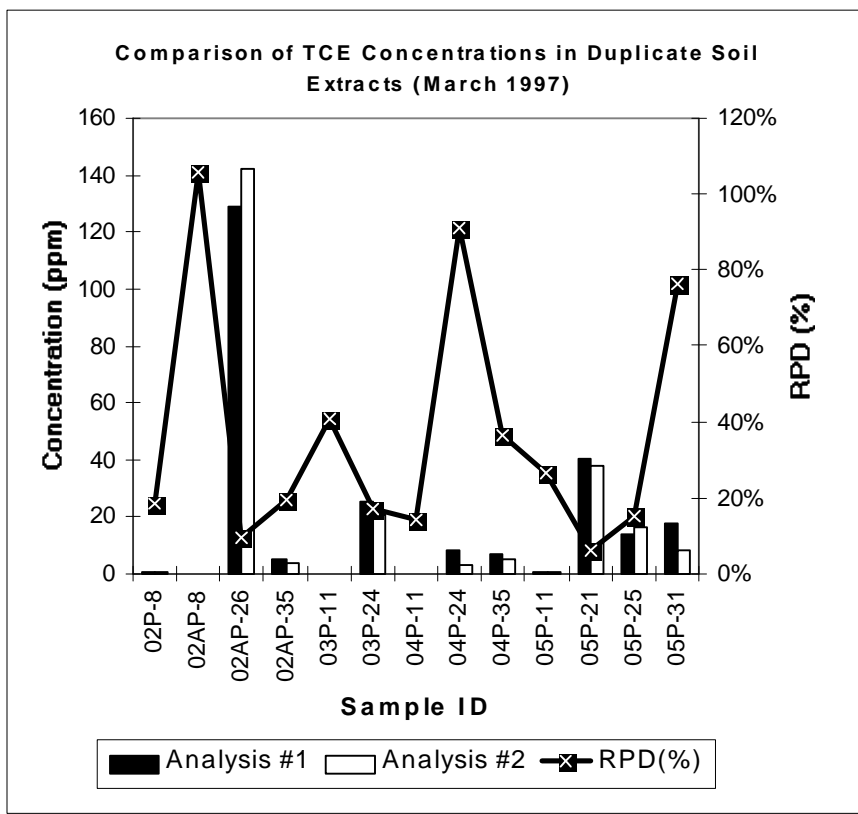


Figure G-1c. Field Duplicates - ORNL/GJ Soil Sampling - August 1997

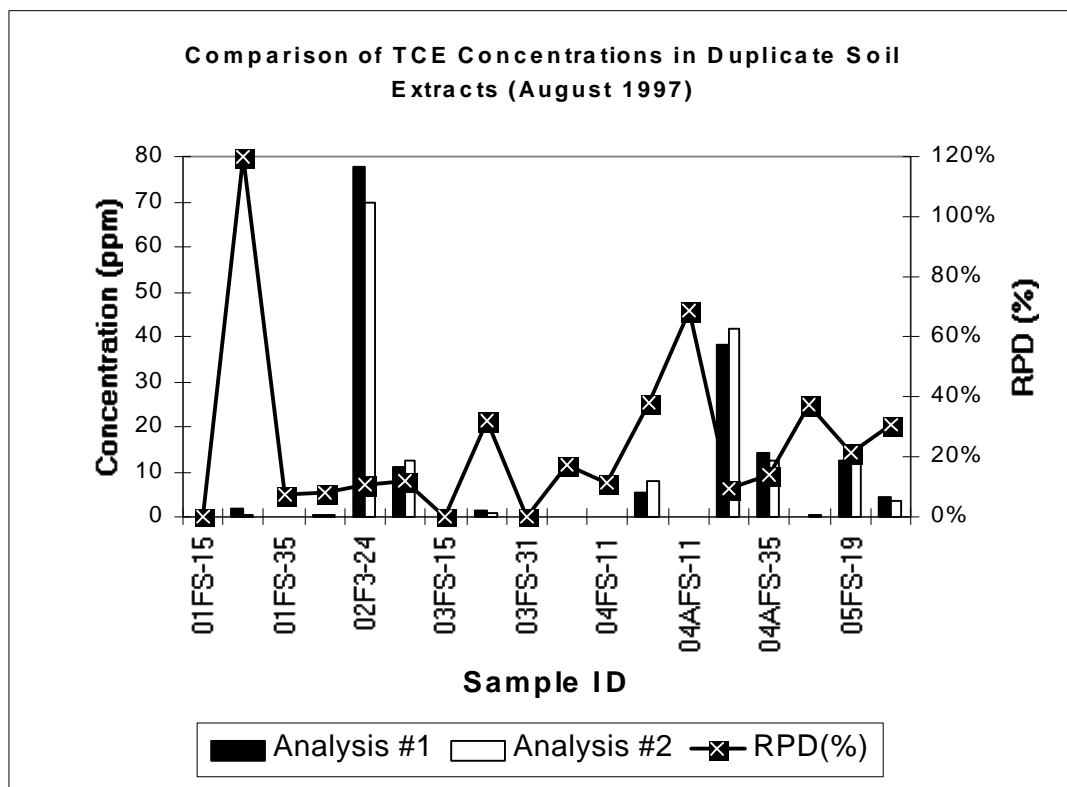
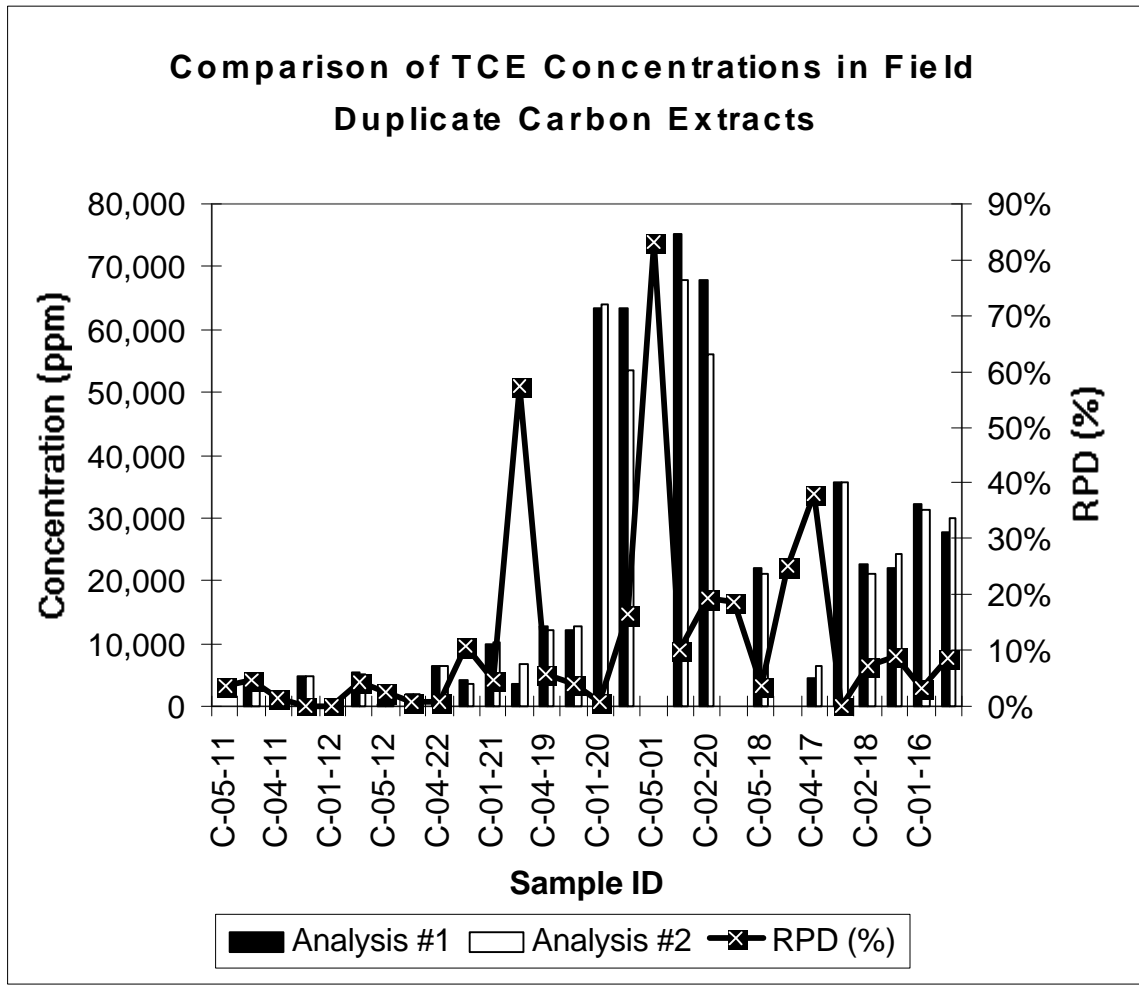


Figure G-2. Field Duplicates - Carbon Cassettes



### 3.2 Analytical Laboratory Operations Assessment

The *Lasagna*<sup>TM</sup> Phase IIa demonstration was conducted using four different laboratories:

(1) CDMFPC Field Laboratory: Served as the primary laboratory for analysis of groundwater samples using a purge and trap gas chromatography method with a capillary column and an electrolytic conductivity detector.

(2) ORNL Grand Junction (ORNL/GJ) Field Laboratory: Served as the primary laboratory for analysis of soil and carbon cassette samples using hexane extraction followed by direct injection gas chromatography with a capillary column and an electron capture detector.

(3) Lockheed Martin Utility Services (LMUS) Analytical Laboratory: Conducted analysis of CDMFPC split groundwater samples using a purge and trap gas chromatographic method with a capillary column and

mass spectrometric detector. Also conducted analysis of ORNL/GJ split water extracts of soil samples using GC/MS techniques.

(4) Roy F. Weston Analytical Laboratory (now called Recra Labnet Laboratory): Served as independent reference laboratory for analysis of groundwater, soil samples, and air flux carbon tubes. Groundwater analysis and a limited number of soil samples were analyzed using purge and trap gas chromatography with a capillary column and an electrolytic conductivity detector. Soil analysis was conducted on a field hexane extract (i.e. extracted by ORNL/GJ with extract shipped to Weston) using direct injection gas chromatography with a capillary column and an electron capture detector. A set of duplicate soil samples was also extracted with water and the water analyzed using a purge and trap/photoionization detector.

[Note: The terms Field Laboratory and Analytical Laboratory are used to differentiate laboratories that

are set up in non-permanent trailers (i. e. Field Laboratories) and those established at a fixed base location.]

Due to its independence from the project and its proven reputation on numerous other DOE projects, the Roy F. Weston Laboratory was selected as the reference laboratory for the *Lasagna*<sup>TM</sup> project.

Each laboratory used methods equivalent to the applicable SW-846 gas chromatography method.

### 3.2.1 Analytical Laboratory Technique Evaluation

The adequacy of the analytical techniques utilized by each of the four laboratories specified above was evaluated by onsite laboratory audits. At least two audits each were conducted of the three onsite laboratories (ORNL/GJ Field Laboratory, CDMFPC Field Laboratory, LMUS Analytical Laboratory) and the reference laboratory. The dates and results of these audits are provided in Table Appendix E-1. Key issues identified include:

- (1) Inadequate dilution techniques for the CDMFPC Field Laboratory,
- (2) Inadequate heating and air conditioning control of the CDMFPC Field Laboratory resulting in difficulty in maintaining calibration constancy,
- (3) Method deviations in organic quantitation by the ORNL/GJ Field Laboratory, and
- (4) Lack of a written procedure for ethane analysis by the LMUS Analytical Laboratory.

With the exception of (1) and (2) above, the issues identified should be minimal impact of the quality of the data produced. As noted in section 3.2.2 below, CDMFPC had the greatest differences in split sample analysis results. There has been no definitive conclusion as to whether these differences were due to analytical error, small scale heterogeneity, or both. However, issues such as (1) and (2) could potentially have significant influence on sample results. No issues impacting data quality were identified for the reference laboratory.

### 3.2.2 Laboratory Proficiency Evaluation

The ability of a laboratory to accurately quantitate TCE in water and soil extracts was evaluated through use of a single blind performance evaluation (PE) sample for the various media. Five replicate samples were submitted during August 1997 in a blind manner (i.e. the samples were identified as PE samples but were at concentrations unknown to the laboratories) to each of the four laboratories. The PE samples consisted of a Supelco standard solution at a concentration of 991 ppmw ( $\mu\text{g}$  TCE/mL methanol). Since the Roy F. Weston laboratory

was required to use two methods (purge and trap GC with electrolytic conductivity detector and direct injection GC of a hexane extract using electron capture detector), it analyzed one set of replicates for each of these methods.

Each laboratory was requested to make appropriate dilutions and analyze the PE samples using the methods that they would utilize for field samples during the study (see section 3.2). The PE sample results for each laboratory are presented in Figure G-3. This figure shows bars representing the average of the five replicate sample results. These results indicate good agreement with the true value with all laboratories within 15% of the actual concentration for all laboratories and all methods. This meets the 80 to 120% accuracy goals specified in the Quality Assurance Project Plan.

The ability of the Weston laboratory to adequately quantitate TCE in air flux carbon tubes was assessed by sending TCE spiked air flux tubes with each of the two batches of samples shipped. This data is shown in Table F-10. During the December 1996 air flux sampling event, two tubes spiked with 100 micrograms of TCE per tube were sent to Roy F. Weston with acceptable recoveries of 70 and 75% (mean of 73%). During the February air flux sampling event, two tubes were sent in with the same level of spike as the previous event (100 micrograms). Recovery, however, was inadequate with less than detectable results. Due to this low spike recovery, the data from the February sampling event were deemed unusable.

### 3.2.3 Reference Laboratory Data Validation

Roy F. Weston Laboratory was selected as the reference laboratory for the *Lasagna*<sup>TM</sup> Phase IIa demonstration. As the reference laboratory, Weston data would serve as the *basis for comparison* for the field laboratories (CDMFPC and ORNL/GJ) and the other analytical laboratory (LMUS). For this reason, it was determined that a 100% independent validation of the data was required. Laboratory Data Consultants, Inc. served as the data validator for the split samples analyzed by Weston. The validation was conducted using a modified outline of the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (February 1994). Some minor modifications were made to these validation guidelines to method specific differences between SW-846 and CLP. The DOE Verification Entity provided a review of the validation packages received by Laboratory Data Consultants, Inc.

A total of eighteen sample delivery groups (SDGs) was received at the Weston Laboratories for analysis. Table Appendix E-2 shows validation findings associated



with each sample identification number in 13 of the 18 SDGs. Key validation issues included exceedances of the EPA recommended preservation temperature of  $4 \pm 2$  °C. during shipment and holding time exceedances.

Temperature exceedances were not large for water samples with a maximum exceedance of 3.4 °C. for SDG #9608L576. Temperature exceedances were greater for some hexane extracts. However, TCE is much more stable in hexane than in water and the results should not be impacted by the temperatures noted. The independent validators also noted several exceedances of holding times for hexane extracts that were shipped to the Weston Laboratory. The validators, however, were applying water based holding times for TCE that do not apply to hexane extracts of TCE. In hexane, TCE is stable beyond the water based holding time of 14 days for preserved water samples. Holding time exceedances for hexane extracts were thus not considered to represent a data quality issue. One groundwater sample (111070302-586) missed its holding time by five days. None of the data were qualified as unusable due to data quality problems.

#### 3.2.4 Accuracy and Precision of Analytical Data

Differences in analytical results of split samples analyzed at two independent laboratories can result due to differences in the accuracy and precision of the respective methods at each laboratory and/or heterogeneity differences in the paired replicates of the split sample. Since the rationale for differences can be complex and often cannot be isolated and defined fully, the best recourse is to document the numerical differences and ensure that they stay within a pre-defined data quality objective. The *Lasagna*<sup>TM</sup> Phase IIa Quality Assurance Project Plan set these limits at 30% for replicated sample aliquots. The following sections discuss intercomparisons for samples split and analyzed in independent laboratories (i.e. interlaboratory

comparisons) and samples split and analyzed in the same laboratory (intralaboratory comparisons).

##### 3.2.4.1 Interlaboratory Comparisons

Numerous samples of groundwater, carbon cassettes, and soil were split and analyzed at different laboratories to provide intercomparisons among sample results and to evaluate comparability of different analytical methods. Several interlaboratory comparative combinations and permutations are possible; however, presentation of all of these would be lengthy and would not be of added benefit. The discussion that follows will mostly use the Weston laboratory data as the reference for comparison.

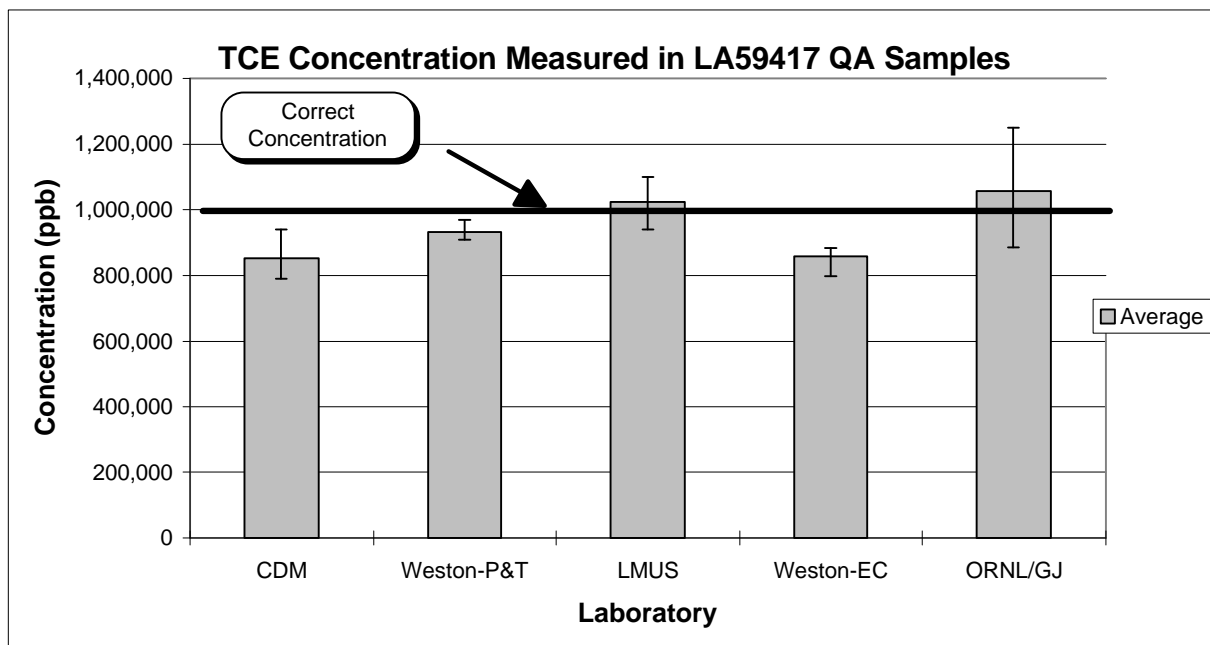
#### Groundwater

Figure G-4 presents interlaboratory comparisons between CDMFPC and Weston (Figure G-4a) and LMUS and Weston (Figure G-4b). These figures represent split samples that were analyzed for TCE. While both laboratories displayed a substantial variability from the reference laboratory, these figures show that the range of RPD percentages tend to be higher and much more variable for the CDMFPC laboratory.

#### Soil

Split soil extracts were analyzed by Weston and the LMUS laboratory for comparison with ORNL/GJ data. Interlaboratory comparisons are presented in Figures G-5a and G-5b for ORNL/GJ vs. Weston during March 1997 and August 1997, respectively. These figures describe analysis results for split soil extracts expressed as concentration of TCE in the extract (i.e. not converted to a soil concentration basis). It is evident that good comparability was present for most split pairs with RPDs less than 30% for 11 of 13 sample pairs for the March 1997 sampling event and 17 of 21 pairs for the August 1997 sampling event.

**Figure G-3. Summary of TCE Concentrations Measured by Various Laboratories for a Blind QA Sample LA59417**



Sample ID	Analysis	Measured Concentration (ppb)				
		TCE-CDM	TCE-Weston	TCE-LMUS	TCE-Weston-EC	ORNL/GJ
LA59417	rep1	790,000	970,000	940,000	861,000	990,000
LA59417	rep2	940,000	930,000	980,000	884,000	885,000
LA59417	rep3	890,000	940,000	1,100,000	873,000	1,113,000
LA59417	rep4	860,000	910,000	1,100,000	798,000	1,250,000
LA59417	rep5	830,000	910,000	1,000,000	877,000	1,054,000
LA59417	rep6	800,000	--	--	--	--
Averages:		851,667	932,000	1,024,000	858,600	1,058,400
SDs:		57,067	24,900	72,664	34,890	136,434
% RSDs:		6.70%	2.67%	7.10%	4.06%	12.89%
Correct Conc:		991,000	991,000	991,000	991,000	991,000
% of Correct:		85.9%	94.0%	103.3%	86.6%	106.8%

Figure G-4a. Laboratory Intercomparison - TCE in Groundwater Samples - CDMFPC vs. Weston

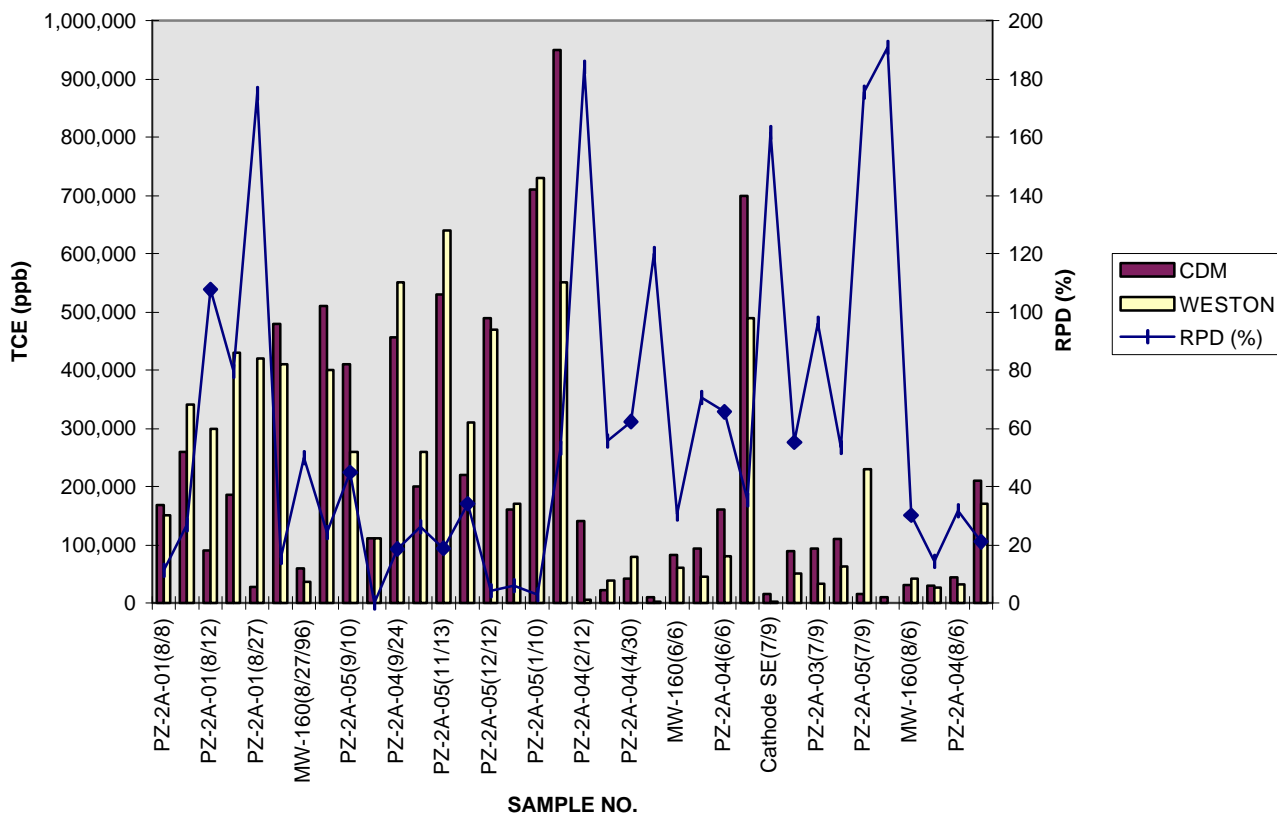


Figure G-4b. Laboratory Intercomparison - TCE in Groundwater Samples - LMUS vs. Weston

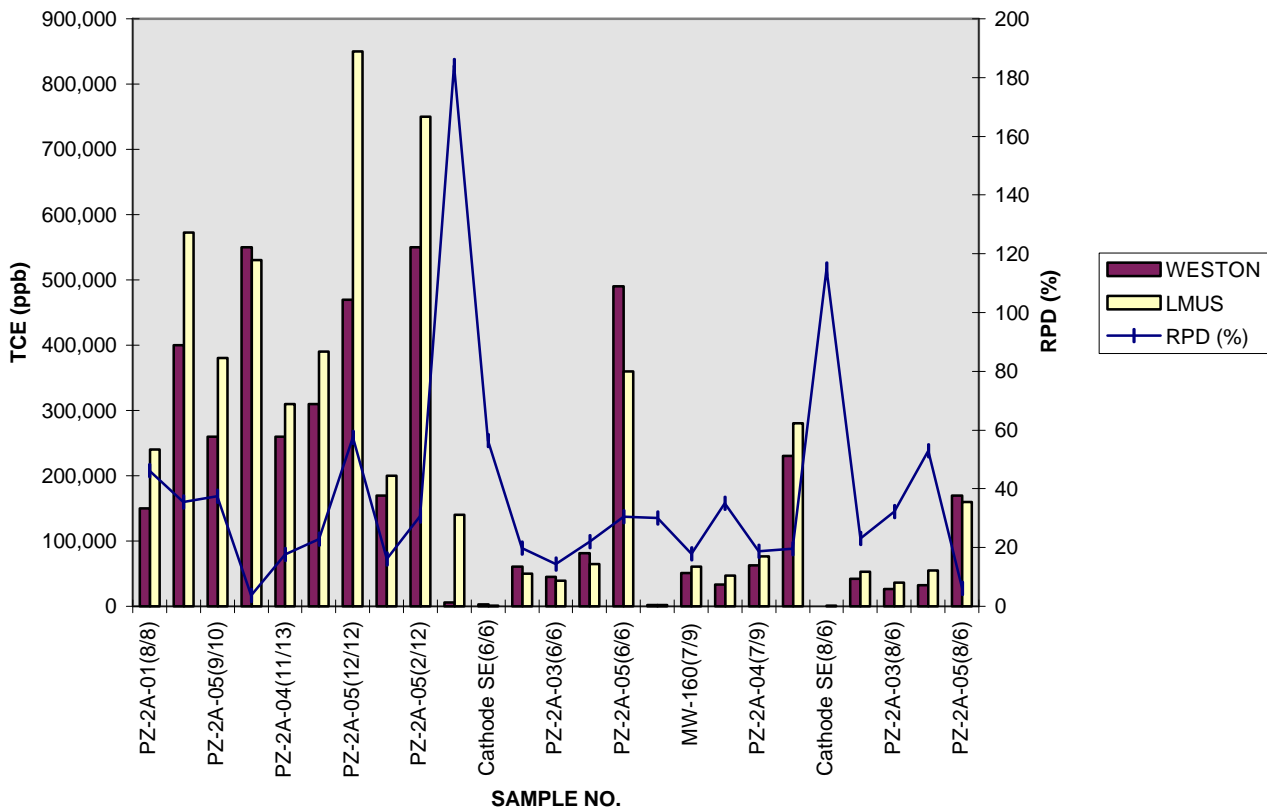
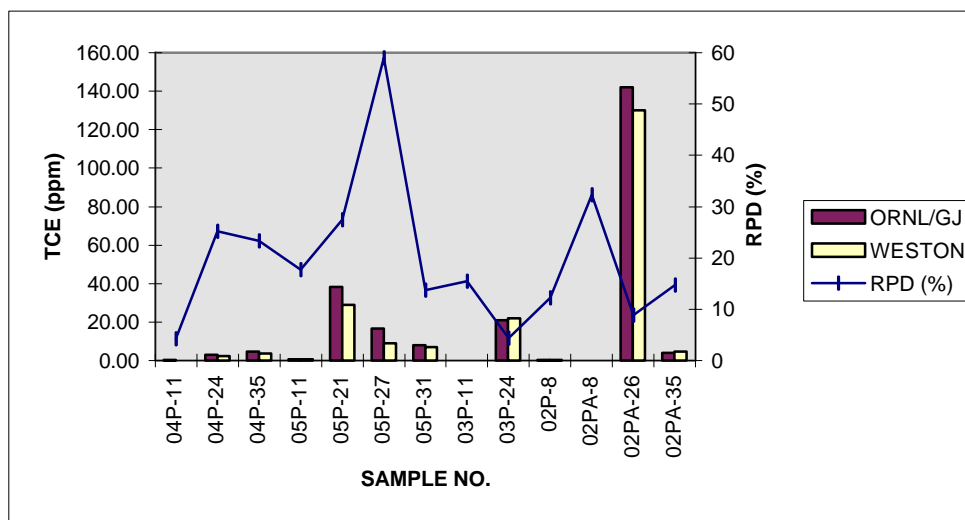
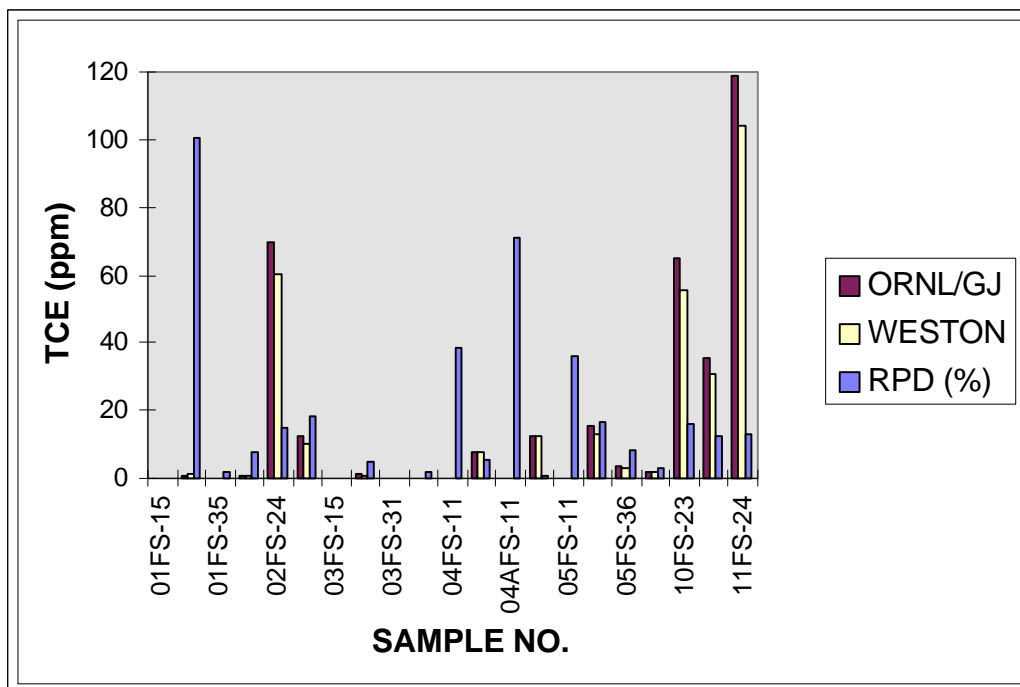


Figure G-5a. Interlaboratory Comparison - Soil Analysis by GC-ECD - March 1997 - ORNL/GJ vs. Weston



**Figure G-5b - Interlaboratory Comparison - Soil Extract Analysis by ORNL/GJ and Weston (EC Detector) - August 1997**

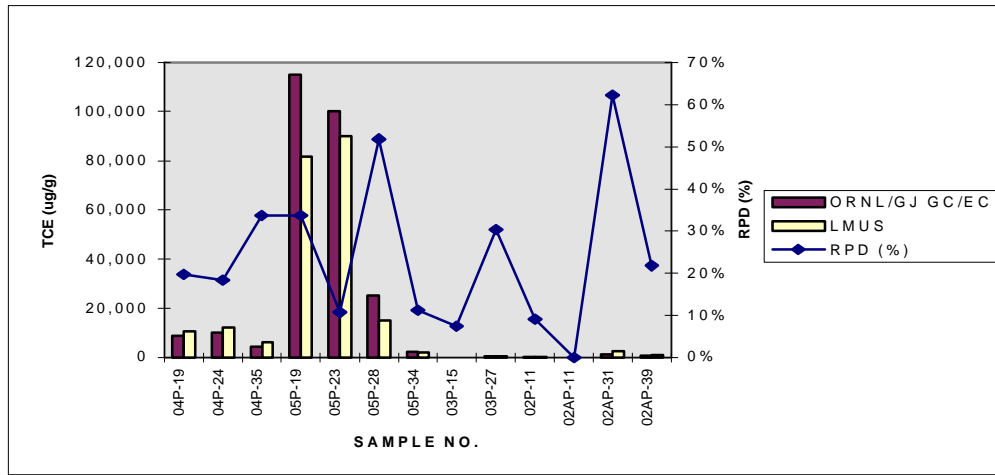


To ensure that the analytical method employed by ORNL/GJ yields results that are comparable to standard EPA volatile organic techniques (e. g. EPA Method 624 Purge-and-Trap GC/MS), several field samples were split and analyzed by Purge-and Trap GC/MS. These splits were prepared by taking a small aliquot of the ORNL/GJ sample before hexane extraction, placing the aliquot in a vial, and filling the vial headspace with organic free deionized water. Figure G-6 presents interlaboratory comparisons for ORNL/GJ vs. LMUS for the March 1997 sampling event. This comparison was made so that results from the GC/EC method used in the field could be compared to GC/MS data obtained at an onsite laboratory. It was hoped that these samples could also be used to quantify the minor *cis*-DCE and VC impurities. However, the extent of sample dilution caused all minor

components to be at concentrations below the instrument level of detection. Data are expressed as actual wet weight soil concentration. The comparability between sample pairs was not as good as that for ORNL/GJ vs. Weston split extract data with five of 13 pairs having RPDs at or greater than 30%. No pattern can be seen in the paired comparisons to explain these differences. Neither laboratory had a low or a high bias consistently.

Figure G-7 presents interlaboratory comparisons between ORNL/GJ and Weston with Weston using a purge and trap GC/PID analysis method rather than the extract injection method shown previously in Figures G-5a and G-5b. This comparison shows good agreement for most sample pairs with 13 of 20 paired comparisons having less than 30% RPD.

**Figure G-6. Interlaboratory Comparison - Soil Analysis by ORNL/GJ (EC Detector) and LMUS (P&T) - March 1997**



**Figure G-7. Interlaboratory Comparisons - Soil Analysis by ORNL/GJ (EC Detector) and Weston (P&T) - August 1997**

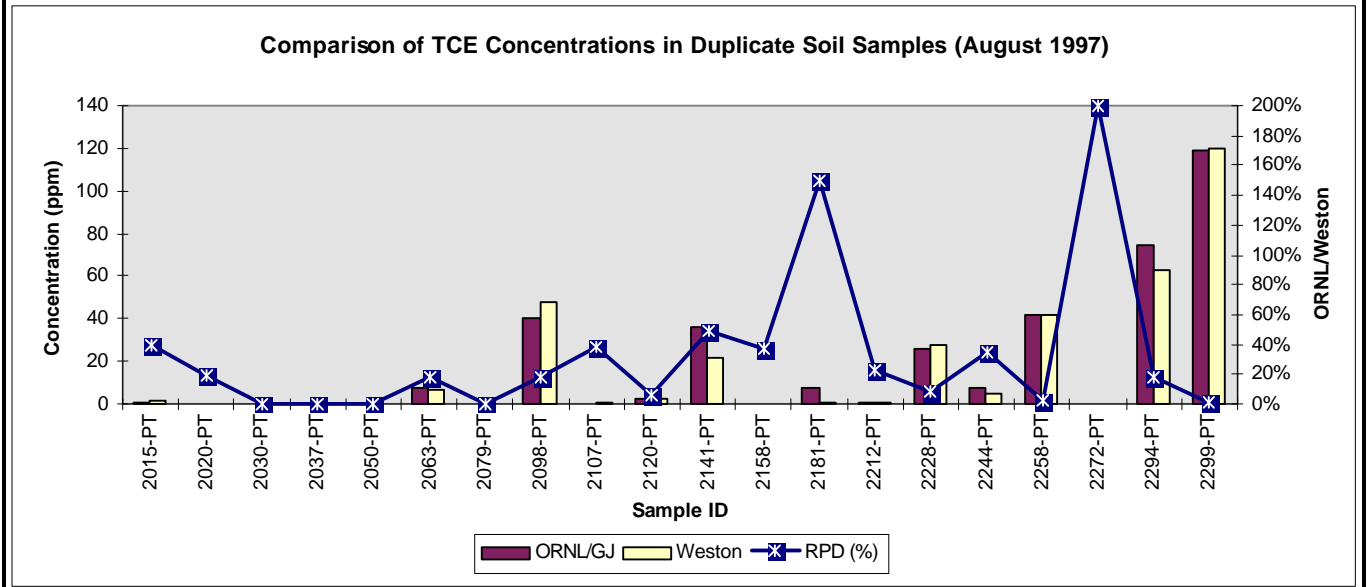
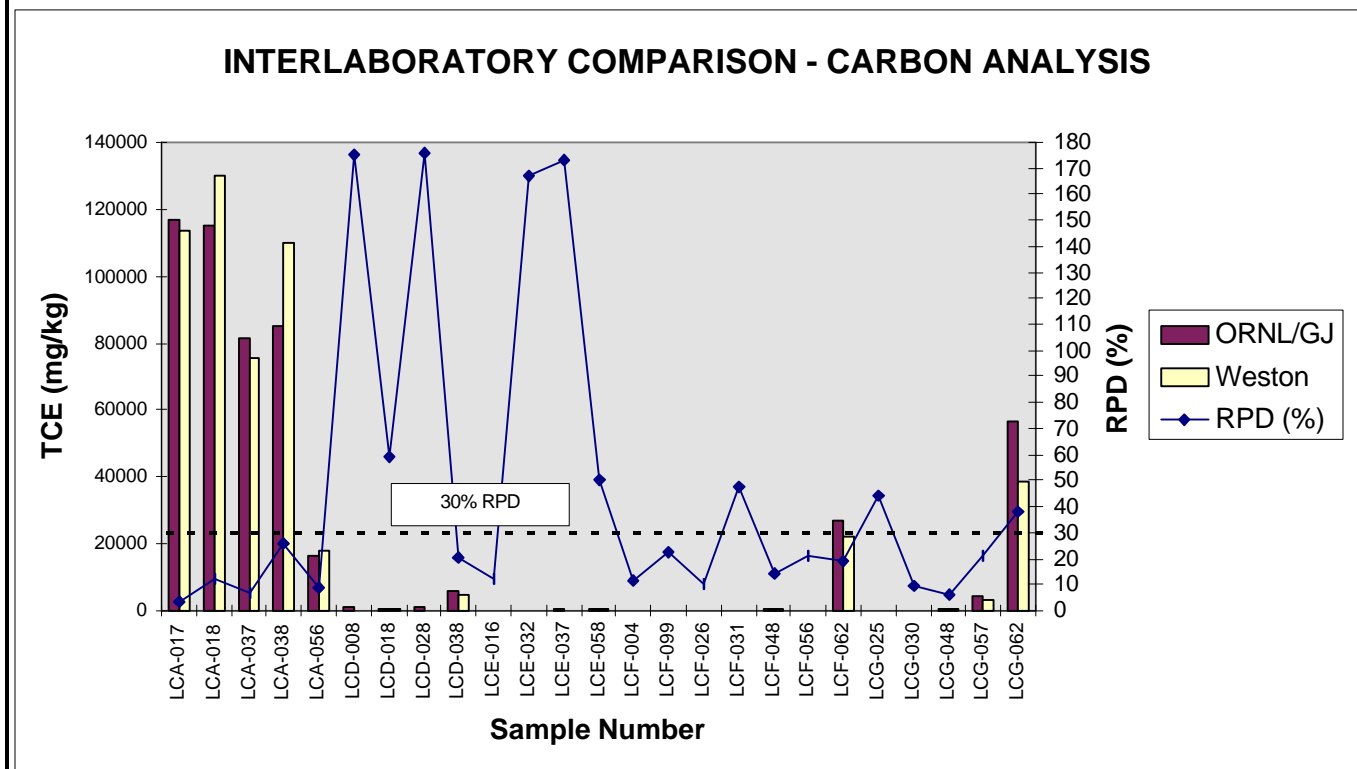


Figure G-8. Interlaboratory Comparisons - Carbon Cassette Analysis by ORNL/GJ and Weston



Carbon Cassettes

Split hexane extracts were analyzed for TCE on 25 occasions by the ORNL Grand Junction field laboratory and by the Weston reference laboratory. It should be noted that sample splitting was performed AFTER the sample was extracted by sonication at the ORNL Grand Junction field laboratory. Figure G-8 shows the intercomparison data for these split extract analyses. Sixteen of the 25 split analyses had acceptable comparability with results less than 30% RPD. Nine of the split pairs showed results ranging from 38% RPD to an extremely poor precision of 176%. It has been speculated that the extremely high differences in some sample pairs may be due to the field laboratory diluting these extracts before they were sent to the Weston Lab without making a record of this dilution for later data interpretation. Another explanation could be due to analytical uncertainty since, all %RPDs greater than 100% were observed for extracts with very low TCE concentrations. When using these suspect data pairs, one should conservatively use the higher of the two values.

The comparisons of the soil and carbon data shown above as measured by RPD% for ORNL/GJ vs. Weston-ECD, ORNL/GJ vs. LMUS, and ORNL/GJ vs. Weston-P&T, indicate much better comparability among

laboratories for soil than those for groundwater split samples.

Air Flux Tubes

There were no interlaboratory comparisons for the air flux tubes samples. All samples were analyzed by Weston laboratory.

3.2.4.2. Intralaboratory Comparisons

At the CDMFPC, LMUS, and ORNL/GJ laboratories, numerous samples were analyzed in duplicate (i.e. laboratory duplicates) to provide an estimate of intralaboratory variability.

Groundwater

Figures G-9 and G-10 display the means and value ranges for replicate pairs of groundwater samples collected and analyzed by CDMFPC and LMUS, respectively. Replicates included two to four analyses of the same well sample either by analysis of different vials from the same well or by repeat analysis of one vial. The mean of the replicates is represented by the bar and the vertical lines represent the maximum and minimum replicate values. The graphed line represent the difference between the minimum and maximum replicate. Figure G-9 indicates that large variations occurred in many of the replicates analyzed by CDMFPC. These variations were noted by independent data

reviewers early in the project and were the source of special investigation. Although, no definitive rationale could be determined for the source of the variance, possible explanations include uncontrolled temperature fluctuations in the CDMFPC field laboratory and propagation of error due to inaccuracies when making large dilutions.

Soil

To ensure that instrumental analysis precision objectives (<20% RPD) were being met, ORNL/GJ staff routinely analyzed soil extracts in replicate. The results of these laboratory duplicate analysis are shown in Figure G-11. This figure shows that most duplicate analysis had RPD values of less than 5% with all having less than 10% RPD. EPA typically specifies RPD requirements of less than 20% for TCE analysis (e.g. the Contract Laboratory Program requires less than 14% RPD for TCE duplicates).

Figure G-9. Intralaboratory Comparison - TCE in Groundwater - CDMFPC Field Laboratory

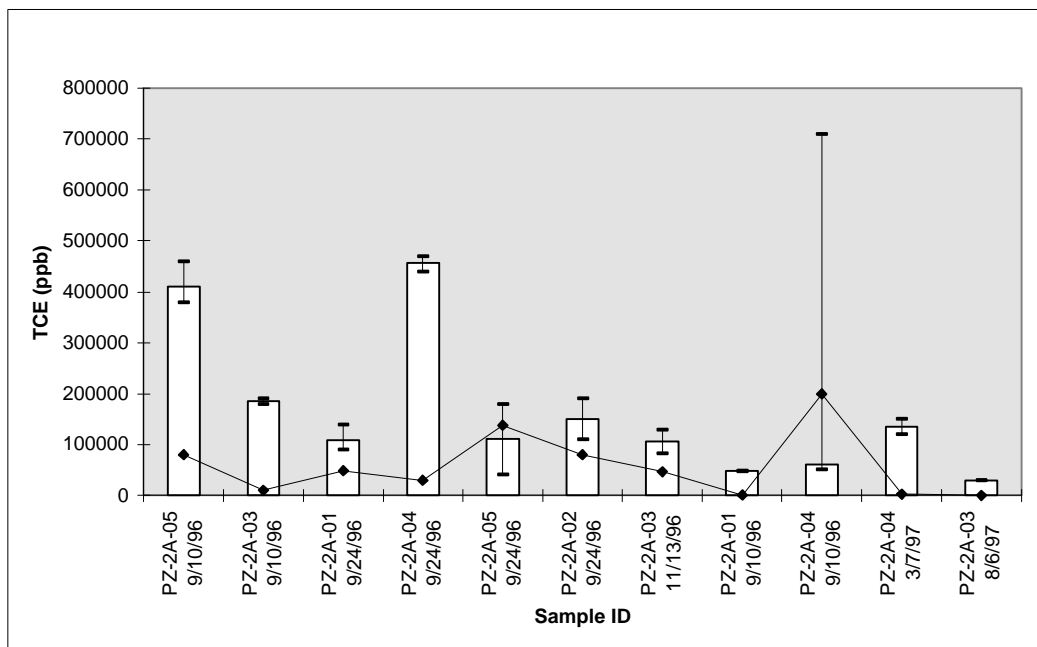




Figure G-10. Intralaboratory Comparison - TCE in Groundwater - LMUS Laboratory

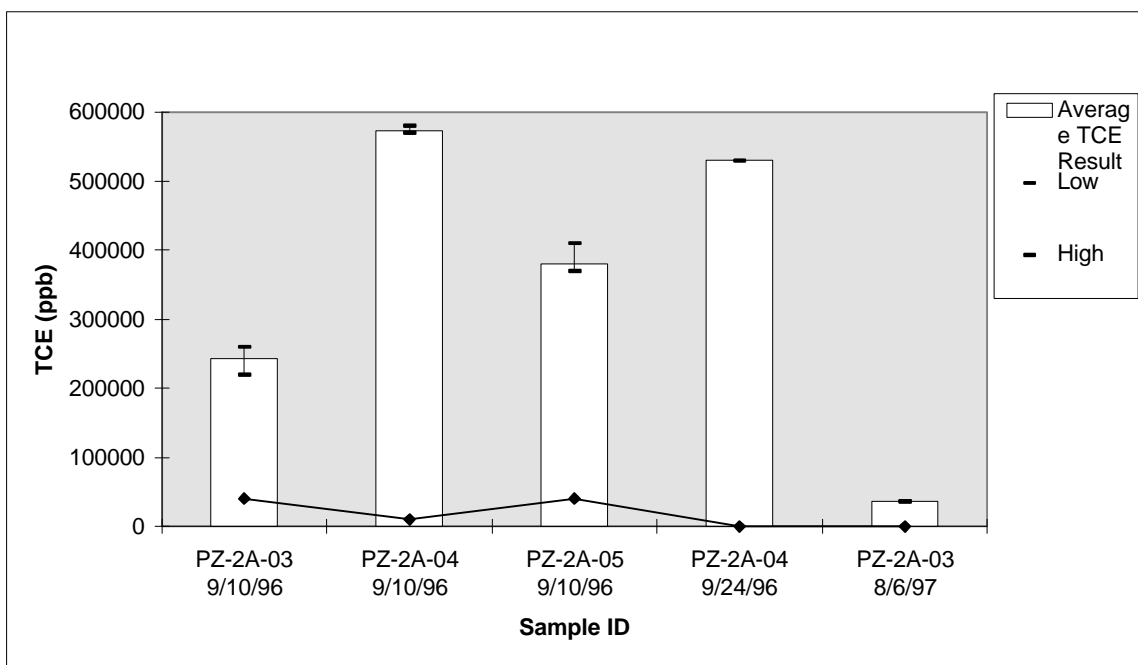
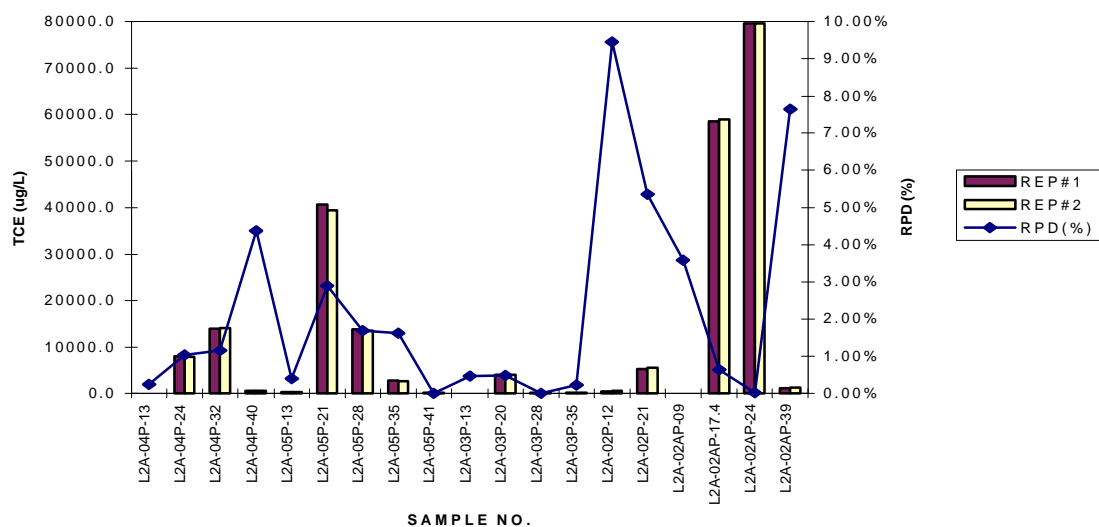


Figure G-11. Duplicate Analysis of the Same Soil Extract - ORNL/GJ Field Laboratory



Carbon Cassettes

Laboratory duplicates were analyzed by splitting the carbon extracts and analyzing the replicate pair. Figure G-2 illustrates the results of the analysis of these split extracts. Excellent agreement was obtained between laboratory duplicate pairs with all but three duplicates with RPD less than 30%.

Air Flux Tubes

Laboratory duplicates were not requested for the Weston laboratory analysis of air flux tubes.

3.2.5 Analytical Method Quality Control for Soil Analysis

Given the critical importance of the soil sampling data in validating the efficiency (and hence the success) of the *Lasagna*<sup>TM</sup> technology, it is necessary to ensure that the laboratory operations that produced these data were

under control when these data were generated. The discussion that follows presents laboratory blank data, calibration verification data, and the results of a special extraction efficiency study for a soil matrix.

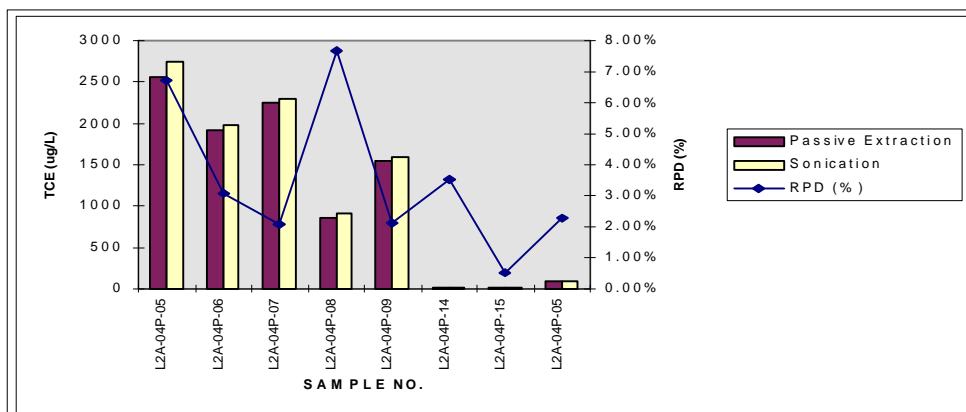
To ensure that the hexane used in the extractions of soil and carbon was free of contamination and that contaminant carryover in the chromatographic column was absent, hexane blanks were run frequently during the period of analytical operations. Table Appendix E-4 presents a summary of the TCE results for analysis of hexane laboratory blanks. All results were less than the detection limit of 5 ppb indicating no cross-contamination.

At the beginning of each set of analyses, a five-point calibration curve was generated. To ensure that the instrument accuracy was acceptable on each day of operation an instrument calibration verification standard was analyzed before the instrument was used each day and again after every 10 samples. Verification standards used included both a 100 ppb and 250 ppb TCE standard representing the mid and upper region of the calibration range. The specific standard concentration used at any given time depended on the expected range of TCE in the samples that were to follow. Table Appendix E-5 presents the results of calibration verification standard analysis for the periods of analytical operation. Most

results indicated less than a 10% difference from the initial calibration with all results less than the 15% requirement stipulated by EPA in SW-846 (Method 8000) for proper chromatographic operation. In the event that calibration standard analysis was measured to be greater than  $\pm 15\%$ , the calibration curve would be re-generated.

The analytical method for TCE extraction from the soil matrix involved simply placing a small aliquot of soil into a weighed vial of hexane in the field, shaking to transfer the TCE into the hexane, reweighing the vial to obtain the weight of the soil, and GC analysis of the hexane supernatant. To ensure that this analytical method was aggressive for the extraction of TCE, comparisons were made by splitting samples and extracting one replicate portion using the ORNL/GJ method and the other using sonication (an EPA approved method for soil sample extraction). The results of this analysis are presented in Figure G-12. This shows that sonication was essentially the same as TCE extraction using the ORNL/GJ method since approximately 3.5% greater average %RPD was measured for the analyte. With such a small difference in extraction efficiency, the added time and effort required for sonication would not be justified for a project, such as *Lasagna*<sup>TM</sup>, where other sources of data variability are much more pronounced.

**Figure G-12. Soil Sample Extraction Technique Evaluation for TCE ORNL/GJ Method vs. Sonication Method**



Location	TCE (ppb)		RPD (%)
	Passive Extraction	Sonication	
L2A-04P-05	2568	2747	6.74%
L2A-04P-06	1913	1973	3.09%
L2A-04P-07	2253	2300	2.06%
L2A-04P-08	850	918	7.69%
L2A-04P-09	1551	1584	2.11%
L2A-04P-14	11.2	11.6	3.51%
L2A-04P-15	19.2	19.1	0.52%
L2A-04P-05	86	88	2.30%

### 3.3 Rollup Assessment and Statistical Evaluation of Data Quality Objectives

#### 3.3.1 Data Quality Issues and Limitations

The above assessment indicated some data quality issues and limitations that must be considered when using the *Lasagna*<sup>TM</sup> Phase IIa data for remedial decisions. These issues and their impacts are summarized in Table G-1.

#### 3.3.2 Evaluation of Trends

The goal of the *Lasagna* Phase IIa demonstration is to demonstrate the effectiveness of the *Lasagna*<sup>TM</sup> process in treating TCE contaminated low-permeability soils to a regulator set criterion of 5.6 ppmw (µg/g). The success in meeting this goal can be assessed by evaluating pre-treatment and post-treatment soil sample results obtained in March 1996 and August 1997, respectively. This evaluation of effectiveness will be presented in two manners: (1) by looking at the efficiency of TCE removal in the primary contamination zone where TCE is significantly elevated above background, and (2) by comparing the overall mean of TCE for the depth of sampling to the regulator criterion of 5.6 ppmw (µg/g) (NOTE: the mean of the soil TCE concentration integrated over depth is the most appropriate value to use for comparison since the regulator criterion is based on a

goal for a site average TCE level). The following paragraphs present the results of these analyses.

##### 3.3.2.1 Treatment Effectiveness

The treatment effectiveness assessment was conducted by plotting depth profiles of TCE contamination at each of the five *Lasagna*<sup>TM</sup> soil boring locations in the treatment zone. These plots are shown in Figures F-1 and F-2 and in the figures in Appendix B. For duplicate pairs, the average concentration was obtained and plotted.

Once depth profiles were obtained, the “zone of TCE contamination” was obtained from the initial pre-treatment TCE profile. This was done by locating the depth interval for which TCE concentrations exceeded 2.5 ppmw (µg/g). This concentration was chosen as a value that, from an analytical chemistry perspective, can be well differentiated from background. This pre-treatment “zone of contamination” was then compared with the TCE concentrations in the same depth zone for the post treatment soil sampling. Statistics were computed for the pre and post treatment conditions in the “zone of contamination” including the mean TCE concentration and the 95% confidence bound of the mean. These statistics as well as comparisons of pre and post conditions are shown in Table G-2. It should be noted that the 95% confidence bounds represent the region within which one can be 95% certain the true mean of the TCE contamination falls.

***Table G-1. Data Quality Issues and Impacts Upon the *Lasagna*<sup>TM</sup> Phase IIa Technology Demonstration***

<b><u>ISSUE</u></b>	<b><u>IMPACT</u></b>
The QA/QC program was not fully implemented during the initial soil sampling event of March 1996 (e. g. interlaboratory replicates were not analyzed and QA audits were not performed). However, all analytical and sampling procedures were the same as those described in the present final report.	Reduces knowledge of any measurement variability present during the first soil sampling event. It can be reasonably expected, however, that the variability and hence data quality is similar to that of the other two soil sampling events (March and August 1997) since the sampling and analysis was performed using the same staff (ORNL/GJ) and procedures.
Intralaboratory and interlaboratory precision of analysis for groundwater was poor for many sample replicates analyzed by the CDMFPC field laboratory.	This impacts the usability of CDMFPC groundwater data which is about 80% or more of the total groundwater database. The error introduced by imprecision can, however, be estimated and used to mitigate decision errors. The impact that this would have on data use would then depend on the sensitivity of the decision to concentration.
Air flux chamber data are unusable for the February 1997 sampling event due to lack of ability to analytically recover matrix spikes	Only one set of usable air flux chamber data exists for this project. It is not advisable to make remedial action decisions based on one sampling event. It is recommended that additional air flux chamber data be collected during future <i>Lasagna</i> <sup>TM</sup> work to confirm that air release of TCE is not significant.

This table indicates that substantial treatment occurred at each location with treatment effectiveness ranging from 33% at location L2A-02 to 99.7% at L2A-03. At two locations, L2A-01 and L2A-03, the upper 95% confidence bound of the mean was below the treatment goal of 5.6 ppmw ( $\mu\text{g TCE/g clay}$ ). At each other location, the treatment efficiency indicates that *Lasagna*<sup>TM</sup> was effective but that more treatment time is needed.

#### 3.3.2.2 Progress in Meeting Action Levels

The effectiveness of the *Lasagna*<sup>TM</sup> technology as a remedial approach for meeting Paducah site action levels can be evaluated by comparing the average concentration in the soil column to the Commonwealth of Kentucky accepted site action level of 5.6 ppmw ( $\mu\text{g/g}$ ). Table G-3 presents a summary of the mean concentrations of TCE at each of the five sampling locations and the 95% statistical confidence bounds for the mean. At locations L2A-01 and L2A-03, the data indicate that *Lasagna*<sup>TM</sup> cleanup met cleanup goals with the upper 95% confidence bound of the mean being less than the 5.6 ppmw ( $\mu\text{g/g}$ ) action level. At locations, L2A-04 and L2A-05, significant cleanup has occurred but it is uncertain whether the cleanup goal has been met (i. e. only the confidence bound was below the 5.6 ppmw ( $\mu\text{g/g}$ ) limit).

**Table G-2. *Lasagna*<sup>TM</sup> Treatment Effectiveness -Statistical Data Summary for TCE in Soil for the Zone of Contamination<sup>24</sup>**

<b>Location</b>	<b>BEFORE Mean TCE (ppmw (<math>\mu\text{g/g}</math>))</b>	<b>BEFORE 95% Confidence Bounds (ppmw (<math>\mu\text{g/g}</math>))</b>	<b>AFTER Mean TCE (ppmw (<math>\mu\text{g/g}</math>))</b>	<b>AFTER 95% Confidence Bounds (ppmw (<math>\mu\text{g/g}</math>))</b>	<b>Mean Efficiency (%)</b>
L2A01	51	19 to 83	1.9	0 to 3.9	96%
L2A02	45	31 to 59	30	7 to 43	33%
L2A03	76	31 to 121	0.24	0.03 to 0.45	99.7%
L2A04	67	42 to 92	20	12 to 28	70%
L2A05	58	35 to 81	14	7.7 to 21	76%

## 4.0 Conclusions

The preceding assessment indicated that technology demonstration data are of sufficient quality for making informed decisions regarding *Lasagna*<sup>TM</sup> treatment effectiveness. Limitations exist on the usability of the CDMFPC groundwater due to high replicate variability. However, these data combined with independent groundwater data from the LMUS and Weston laboratories should allow a user of the data to obtain a reasonably reliable representation of groundwater contamination and trends over time. The soil and carbon data were of good quality with minimal limitations for usage. One should always, however, consider the data variability in making decisions rather than using the straight mean of the data. Due to the availability of only one usable dataset for air flux measurements, any decisions of air flux from the *Lasagna*<sup>TM</sup> site should be provisional only with confirmation needed during other operations before final decisions are reached.

The analysis of trends in TCE contamination of soil before and after *Lasagna*<sup>TM</sup> treatment clearly indicate that substantial decreases did occur and that the technology can be used to meet regulator accepted action levels. Refer to Section F for further discussion of treatment including chemical evidence of treatment effectiveness.

<sup>24</sup> The Zone of Contamination was the depths where the pre-treatment TCE concentrations were greater than 2.5 ppmw ( $\mu\text{g/g}$ ).

**Table G-3. *Lasagna*<sup>TM</sup> Process - Compliance with Cleanup Target - Statistical Data Summary for TCE in Soil**

<b>Location</b>	<b>BEFORE Mean TCE (ppmw (µg/g))</b>	<b>BEFORE 95% Confidence Bounds (ppmw (µg/g))</b>	<b>AFTER Mean TCE (ppmw (µg/g))</b>	<b>AFTER 95% Confidence Bounds (ppmw (µg/g))</b>	<b>Met KY Action Level?</b>
L2A01	18	4.4 to 32	0.87	0.13 to 1.6	yes
L2A02	42	28 to 56	24	13 to 35	no
L2A03	52	20 to 85	0.16	0.018 to 0.31	yes
L2A04	34	17 to 50	11	5.3 to 16	maybe
L2A05	34	17 to 51	9.2	4.7 to 14	maybe

## H. Summary of Installation Costs, Lasagna™ Phase IIa Test

A detailed cost analysis was conducted by MSE-TA. Their report is shown in this section.

### Summary

*Lasagna*™ is an *in-situ* method for remediating soils that have low hydraulic conductivity. The Phase IIa test was designed to provide final data to the consortium for design purposes and to demonstrate to DOE EM-40 that full-scale cleanup (Phase IIb) should proceed. For the Phase IIa test, 6,750 ft<sup>2</sup> of electrode and treatment zones were emplaced to a depth of 45 feet and were configured to remediate 1,050 yards of TCE contaminated soils to less than 5.6 ppm. This test was planned to operate for six months, but due to the presence of DNAPL, the duration was extended to a total of 11 months.

Total costs incurred during the Phase IIa test were \$1,375,206, or \$1,309.71/yard<sup>3</sup>. Of this, \$578.76/yard<sup>3</sup> were core (direct) costs and \$730.95/yard<sup>3</sup> were non-core costs, mainly sampling and oversight (core and non-core costs are discussed further in the narrative following this summary). The observed costs are much higher than anticipated for an actual, full-scale, remediation because of the level of support given to the Phase IIa test, which was conducted on a relatively small volume of soil. The Phase IIa test indicated that the costing formula, developed by the consortium during initial evaluation of

the *Lasagna*™ technology, can be used to estimate remediation core costs for this technology to within 20% of actual costs.

As part of this cost verification for the Phase IIa test, the formula developed by the consortium was used to simulate core costs in a paper study of a full-scale clean up. This study indicated that *Lasagna*™ remediation core costs can range from approximately \$120/yard<sup>3</sup> of soil remediated in optimal conditions to nearly \$260/yard<sup>3</sup> in adverse conditions. This study was based on a treatment zone that was 60 feet wide and 100 feet long and that was either 15 feet or 45 feet deep. Clean-up time was varied from one to five years and the movement of either 2 or 4 pore volumes of contaminated soil water was used as the required treatment parameter. All other factors were held constant. These cost estimates are for core costs only, as non-core (indirect) costs such as oversight, health and safety, sampling, and QA/QC can vary significantly from one site to the next. A typical cost breakdown for one of these cost studies is summarized in the following table (Table H-1). Also, as an example of the impact some of these factors have on cost, if the required pore volumes in this example were reduced to 2, then the core remedial cost reduces to \$137/yard<sup>3</sup>.

***Table H-1. Example of an estimated cost breakdown for a full-scale Lasagna™ remediation, 45 feet deep, with 4 pore volumes required. Time to remediation is 3 years. Total volume is 10,000 yd<sup>3</sup>.***

Cost Element	Estimated Cost	Cost/yard <sup>3</sup>	% of Total
Pretreatment Sampling and Design	\$20,000	\$2.00	1.2%
Site Preparation	\$25,000	\$2.50	1.5%
Electrode, Treatment zones, and equipment installation	\$1,050,000	\$105.00	61.0%
Operation and Maintenance	\$502,000	\$50.20	29.1%
Project Management	\$100,000	\$10.00	5.8%
Site Restoration	\$25,000	\$2.50	1.5%
<b>Totals</b>	<b>\$1,722,000</b>	<b>\$172.20</b>	<b>100.1%</b>

## Introduction

As discussed in Section C of this Final Report of the RCI Participants, a consortium was formed to pursue Lasagna™ design and testing. This consortium consists of Monsanto, DuPont, and General Electric with the US EPA and DOE participating and with Monsanto acting as lead for the consortium. The consortium chose the DOE Paducah Gaseous Diffusion Plant in Paducah, Kentucky, where trichloroethylene (TCE) has contaminated soils with low hydraulic conductivity, as the Phase I and IIa test site. Basically, the Lasagna™ process, also discussed in Section C, involves emplacement of treatment zones between electrode zones and passing direct current through the electrode zones. The electric field thus established moves soil pore water through the treatment zones, which remove the targeted contaminants from the pore water. Soil samples are used to measure soil parameters necessary to estimate the amenability of these soils to Lasagna™ technology and to design a remediation strategy.

The consortium contracted with many different companies to conduct the Phase IIa test. CDM Federal Programs Corporation (CDMFPC) in Paducah was contracted to provide on-site support and management of the test. Nilex Corporation, Denver, Colorado, was contracted through CDMFPC to install the electrode and treatment zones. Asphalt Paving, Inc., also in Paducah, was contracted through CDMFPC to provide on-site labor. LMES in Paducah was contracted by DOE to provide oversight for the test, to manage soil, water, and carbon cassette sampling, and to analyze soil and cassette samples at the GJO soils laboratory in Grand Junction, Colorado. Water samples were analyzed at the CDMFPC laboratories in Paducah. MSE Technology Applications, Butte, Montana, was contracted by DOE to provide cost verification.

Remediation Technology costs can be divided into two broad categories, basic remediation costs or “core costs,” and ancillary costs, or “non-core costs.” Core costs are those that are unique to and directly attributable to the technology itself, and can be broken down into four main cost elements:

- 1) pretreatment sampling and design,
- 2) site preparation and technology installation,
- 3) operation and maintenance and,
- 4) technology removal and site cleanup.

Ancillary or non-core costs are those more generic to all remediations and they are commonly site specific. They include such cost elements as management and oversight, health and safety, sampling, and quality assurance or quality control (QA/QC). Because they are

commonly site specific, they are highly variable and difficult to estimate without input from site managers.

Pretreatment sampling and design costs for the Lasagna™ Phase IIa test were not reported by the consortium. Also, only minimal post-installation cleanup costs and no removal costs have been incurred as yet. Thus, this review of Phase IIa costs will focus mainly on installation and operation and maintenance costs, with a brief discussion of pretreatment sampling and design costs. Sampling, management, and oversight costs for Phase IIa will also be reviewed. However, since Phase IIa was a test of Lasagna™ technology, most of these costs are far greater than would be experienced in a full-scale remediation.

As discussed in Sections E and F of this report, Phase IIa was initially designed to operate for 3 to 6 months, depending upon results. However, the test was extended to nearly a year because free phase TCE was found to exist in a sandy soil horizon at a depth of approximately 20 feet. As shown in Table F-1, page F-2, the Phase IIa test ran for 331 days, or nearly 8,000 hours.

## Phase IIa Installation Costs

Installation costs for Phase IIa can be subdivided into site and equipment preparation, installation of electrode and treatment zones, installation of monitoring and sampling points, and equipment and site cleanup prior to startup of treatment. Equipment for installation of Phase IIa arrived on-site on June 12, 1996, and was released off-site on July 22, 1996. Installation of both electrode and treatment zones and also sampling and monitoring points began on June 25, 1996 and was completed by July 19, 1996. Site and equipment preparation took approximately 9 working days, electrode and treatment zone installation took approximately 13 working days, installation of sample points and monitoring wells took 5 working days, and site cleanup and equipment decontamination took 3 working days.

Nilex provided equipment, a supervisor, and a vibrator operator; API provided 4 laborers and 2 equipment operators; CDMFPC provided 2 people for escort and documentation services; DuPont provided 2 technicians; and LMES provided 1 oversight person. Unforeseen delays during installation resulted from a DOE safety review, modifications of equipment, and the normal learning curve for driving mandrels into this type of ground followed by the emplacement of electrode and treatment zone materials.

Details of the layout of the Phase IIa test are depicted in Figure C-4. The test area was 21 feet wide by 30 feet long by 45 feet deep, yielding a total volume of 28,350 cubic feet (ft<sup>3</sup>), or approximately 1,050 cubic yards (yd<sup>3</sup>).

A total of 2,700 square feet (ft<sup>2</sup>) of electrode zone and 4,050 ft<sup>2</sup> of treatment zone was emplaced, for a total zone emplacement of 6,750 ft<sup>2</sup>. Finally, 24 measurement and sampling points and 5 monitoring wells were installed using a smaller mandrel.

Installation costs were obtained mainly by accounting reports provided by CDMFPC and LMES. Invoices submitted by Nilex and API were also reviewed. The Nilex cost was \$133,677, which equates to approximately \$5,000 per day for the 27 days Nilex personnel were on site. Included in the Nilex cost is the equipment and mobilization and demobilization costs. API personnel were on site for 30 working days. Total charges for API were \$169,939, which equates to approximately \$5,500 per day. Management and oversight charges for the installation phase were \$84,500

for CDMFPC (\$3,500 per day) and \$40,000 (\$1,500 per day) for LMES.

Total cost for all materials was approximately \$70,000, which includes a 30% oversupply for contingencies. Material cost for electrode and treatment zones was \$27,700. Material cost for the monitoring wells was \$5,000 per well or \$25,000 total. Cost of personal protective equipment, fencing, and other miscellaneous supplies was \$11,000, leaving approximately \$6,300 in unused materials on site. Thus, cost of materials actually used was \$63,700.

Costs incurred by contractors, prorated for the various installation cost elements, are summarized in Table H-2. Table H-3 summarizes these costs as unit costs per cubic yard of soil and per square foot of both electrode and treatment zone emplaced.

**Table H-2. Cost Summary for Phase IIa Installation by Contractor and Cost Element.**

	<b>Site Prep. And Cleanup</b>	<b>Zone Installation</b>	<b>Sample Points. And Monit. Wells</b>	<b>Totals</b>
Nilex	\$8,667	\$100,000	\$25,000	\$133,667
API	\$70,939	\$71,500	\$27,500	\$169,939
Materials	\$11,000	\$27,700	\$25,000	\$63,700
CDMFPC	\$21,500	\$45,500	\$17,500	\$84,500
LMES	\$13,000	\$19,500	\$7,500	\$40,000
<b>Totals</b>	<b>\$125,106</b>	<b>\$264,200</b>	<b>\$102,500</b>	<b>\$491,806</b>

**Table H-3. Cost Summary for Phase IIa Installation by Cost Per Unit.**

	<b>Total Cost</b>	<b>Cost/yd<sup>3</sup> of Soil <sup>1</sup></b>	<b>Cost/ft<sup>2</sup> of Zone <sup>2</sup></b>
Site Prep. And Cleanup	\$125,106	\$119.15	na.
Zone Installation	\$264,200	\$251.62	\$39.14
Sample Pts. And Monitor. Wells.	\$102,500	\$97.62	na.
<b>Totals</b>	<b>\$491,806</b>	<b>\$468.39</b>	<b>\$39.14</b>

- 1: Based on 1,050 yd<sup>3</sup> of soil
- 2: Based on 6,750 ft<sup>2</sup> of Electrode and Treatment zones
- na: These costs are not applicable to zone installation



**Table H-4. Core and Non-Core Costs for Electrode and Treatment Zones**

	<b>Core Costs</b>	<b>Non-Core Costs</b>	<b>Costs/ft<sup>2</sup> of Zone</b>
Nilex	\$100,000		\$14.81
API	\$71,500		\$10.59
Materials	\$27,700		\$4.10
<b>Subtotal</b>	<b>\$199,200</b>	<b>\$0.00</b>	<b>\$29.50</b>
CDMFPC		\$45,500	\$6.74
LMES		\$19,500	\$2.89
<b>Subtotal</b>	<b>\$0.00</b>	<b>\$65,000</b>	<b>\$9.63</b>
<b>Totals</b>	<b>\$199,200</b>	<b>\$65,000</b>	<b>\$39.13</b>

**Table H-5. An Estimate of Installation Costs for a Full-Scale Lasagna™ Remediation.** <sup>1</sup>

	<b>Total Cost</b>	<b>Cost/ft<sup>2</sup></b>
Mobilization	\$20,000	\$1.60
Emplacement	\$28,000	\$2.24
Labor & Supervision	\$27,200	\$2.18
Materials	\$62,500	\$5.00
<b>Totals</b>	<b>\$137,700</b>	<b>\$11.02</b>

1. Based on 10 - 8 hr. days. Total zones emplaced = 12,500 ft<sup>2</sup>.

In the Draft Topical Report for Task #5, Cost Analysis (no date given), it was estimated that electrode zone emplacement costs, including materials, would be approximately \$16.00/ft<sup>2</sup> and that treatment zone emplacement costs would be roughly \$10.00/ft<sup>2</sup>. Materials used in the Task 5 study were iron and carbon filler with an iridium oxide coated titanium electrode in the electrode zones and a clay and iron mixture in the treatment zones. Physical emplacement of the zone segments themselves, without materials considerations, was estimated to cost approximately \$8.00/ft<sup>2</sup> using the mandrel tremie tube system. Thus, materials costs in the Task 5 study amounted to \$8.00/ft<sup>2</sup> for the electrode zones and \$2.00/ft<sup>2</sup> for treatment zones. As shown in Table H-3, however, emplacement costs for both electrode and treatment zones during the Phase IIa test totaled slightly over \$39/ft<sup>2</sup>. In order to obtain a better understanding of which of these costs are core and which are not, they are further broken out in Table H-4.

Note in Table H-4 that the core cost (without management and oversight cost) is \$29.50/ft<sup>2</sup> of electrode and treatment zone installed and that materials cost is

slightly more than \$4.00/ft<sup>2</sup>. Phase IIa used a 50/50 mixture of Peerless Iron and Loresco Coke in the electrode zones with a 3/4 inch carbon steel rod inserted to ensure electrical contact. The treatment zones were comprised of 60% Peerless Iron and 40% kaolin clay. Thus, the overall cost of \$4.00/ft<sup>2</sup> for both treatment and electrode zone material seems reasonable. Total emplacement cost is high (\$29.50 vs. \$15 estimated initially by the consortium) because the Nilex bid was a fixed cost bid, and API labor costs, over \$10/ft<sup>2</sup>, are also higher than anticipated in the consortium's initial studies.

Because Phase IIa was a demonstration test, some costs attributable to a demonstration test would not be incurred during a full-scale cleanup. For example, management and oversight and also sampling requirements would probably be significantly reduced. Mr. James Cramer with Nilex was contacted regarding the cost for physically emplacing the electrode and treatment zones. For a typical site in the United States, he stated that mobilization and demobilization would be \$20,000, and that operating costs would be approximately \$350 per hour, including Nilex labor

costs. He estimated that 100 drives (zone segment emplacements) to 50 feet deep could be completed per 8 hour day. This equates to roughly one drive every four minutes and seems somewhat optimistic. Phase IIa production was a little more than 13 drives per day. Therefore, if 20 drives per day is used as a reasonable production rate, (15 " {38.1 cm} zone width per drive to a depth of 50 feet {15.2 meters}) Nilex costs would be approximately \$4.00/ft<sup>2</sup> of zone for a 10 working day installation period. If 3 laborers are required at \$70 per hour each for mixing materials and filling the mandrel, one overall supervisor at \$130 per hour, and material costs of \$5.00/ft<sup>2</sup>, then total zone emplacement cost for a 10 day working period, in which approximately 250 linear feet of zone would be emplaced to a depth of 50 feet, would be slightly over \$11/ft<sup>2</sup>, as delineated in Table H-5. Increasing square footage of zone emplaced will decrease the cost per ft<sup>2</sup> of mobilization only. All other costs will remain fixed.

## Phase IIa Test Operating and Maintenance Costs

Installation of Phase IIa was essentially complete by the end of July, 1996 and the test began operation at noon on August 14. It was scheduled to terminate in February 1997, but received an extension to continue for another 6 months. Final shutdown of the power was on August 6, 1997. A major shutdown occurred from February 26, 1997 to March 17, 1997, while the decision to continue was under consideration, for a total of 19 days. Minor shutdowns for sampling and normal maintenance occurred on five separate occasions. These lasted no more than 8.5 hours and averaged 4 hours. Four days were lost due to a malfunction in the generator and one day was lost to refurbishing the anodes. Thus, for purposes of this cost verification, the Lasagna™ Phase IIa ran approximately 8,000 hours during 357 calendar days for an operational availability of 93%, which is quite good for a test situation.

As previously mentioned, CDMFPC was the on-site Lasagna™ project manager for the consortium for the Phase IIa test and accumulated nearly all of the cost data, including costs incurred by Nilex and API. API was able to separate their labor and generator (electrical) costs, which are the major core cost elements during the operation and maintenance of Lasagna™. LMES provided oversight for the DOE, managed the water and soil sampling program, and provided cost information for these functions. Costs reported by CDMFPC and LMES for operational and maintenance of Phase IIa are depicted in Table H-6 and are summarized with installation costs in Table H-7.

The operating and maintenance costs (core costs) are \$228.95 per yd<sup>3</sup>, significantly higher than anticipated in the consortium's estimates. This appears to result in large part from the level of support given to this demonstration, from the use of generators instead of local power, and from relatively high labor costs, all applied to a relatively small test volume of soil. Sampling and oversight costs are also quite high, again reflecting the support given to this test. These high costs probably would not be encountered in a full-scale cleanup.

In Table H-6, CDMFPC Project Management consisted primarily of one manager with secretarial help, quality assurance (QA), and health and safety support. LMES project oversight was provided by one manager and all administrative support. Operation and maintenance was provided by API and included rental on the generator, servicing of the generator, fuel, maintenance of any ancillary equipment, and all labor required during the operational phase. Also, included in API's operation and maintenance cost is a minor amount of CDMFPC supplied labor during the sampling periods. Finally, a limited amount of site restoration was completed by API during the operational phase.

As delineated in Tables F-2, F-3, and F-4, 197 water samples, 470 carbon cassette samples, and 307 soil samples were collected during the Phase IIa test. Soil and carbon cassette sampling and analysis was completed by LMES in Grand Junction, Colorado, for a cost of nearly \$200,000. The majority of this cost was labor and analysis, but it also included travel (4 round-trips for the crew) material, overhead, and the use of a GeoProbe™ for soil sampling. Some well abandonment at the end of the operational phase, and the removal of the LMES field office at the Paducah test site is also included in LMES costs.

The CDMFPC lab in Paducah did on-site water sampling and analysis to save on costs. These costs were carried by the LMES Paducah office who reported the water sampling cost at \$30,000 and the QA lab work at \$17,000, for a total of \$46,000. Thus, the total for all sampling appears to be \$246,000. This does not include third party QA analysis by the Roy Weston Lab. These costs were carried by Monsanto. Total Phase IIa costs are delineated in Table H-7.

**Table H-6. Cost Summary for Phase IIa Operations and Maintenance.**

	<b>Cost Function</b>	<b>Core Cost</b>	<b>Non-Core Cost</b>	<b>Core cost/yd<sup>3</sup>.<sup>1</sup></b>	<b>Non-core cost/yd<sup>3</sup>.<sup>1</sup></b>
API & CDMFPC	Operation	\$59,900		\$57.05	
Generators	Electrical	\$180,500		\$171.90	
<b>Subtotals</b>		<b>\$240,400</b>		<b>\$228.95</b>	
API & CDMFPC	Site Cleanup		\$27,000		\$25.71
LMES	Samp/Analy.		\$246,000		\$234.29
CDMFPC	Proj. Mgmt.		\$76,000		\$72.38
LMES	Oversight		\$294,000		\$280.00
<b>Subtotals</b>		<b>\$0.00</b>	<b>\$643,000</b>		<b>\$612.38</b>
<b>Totals</b>		<b>\$240,400</b>	<b>\$643,000</b>	<b>\$228.95</b>	<b>\$612.38</b>

1. Based on 1,050 yd<sup>3</sup>.

**Table H-7. Cost Summary for Phase IIa Test.**

	<b>Total Cost</b>	<b>Core Cost</b>	<b>Non-Core Cost</b>	<b>Core Cost/yd<sup>3</sup>.<sup>1</sup></b>	<b>Non-Core Cost/yd<sup>3</sup>.<sup>1</sup></b>	<b>Total Costs/yd<sup>3</sup>.<sup>1</sup></b>
Pretreat. Sampling and Design	Not Reported	----- -	-----	-----	-----	-----
Site Preparation	\$125,106	\$90,606	\$34,500	\$86.29 (14%)	\$32.86	\$119.15
Electrode/Treatment Zone Installation	\$264,200	\$199,200	\$65,000	\$189.71 (33%)	\$61.90	\$251.61
Install Sample Pts. & Monitoring Wells	\$102,500	\$77,500	\$25,000	\$73.81 (13%)	\$23.81	\$97.62
Project Management & Oversight	\$370,000	-----	\$370,000	-----	\$352.38	\$352.38
Operation and Maintenance	\$240,400	\$240,400		\$228.95 (40%)	-----	\$228.95
Sampling	\$246,000	-----	\$246,000	-----	\$234.29	\$234.29
Site Restoration	\$27,000	-----	\$27,000	-----	\$25.71	\$25.71
<b>Totals</b>	<b>\$1,375,206</b>	<b>\$607,706</b>	<b>\$767,500</b>	<b>\$578.76 (100%)</b>	<b>\$730.95</b>	<b>\$1,309.71</b>

1. Based on 1,050 yd<sup>3</sup>.

## Discussion of Phase IIa Costs

The total cost of \$1,310/yd<sup>3</sup> in Table H-7 is very high and again reflects the high level of support given to this test. The test volume is small, 1,050 yd<sup>3</sup>s, and economy of scale factors should rapidly lower per yd<sup>3</sup> costs as larger volumes of soil are remediated. In a full-scale cleanup, a much larger volume would

probably be treated. Also, in a full-scale cleanup instead of a test situation, sampling and oversight requirements would probably not be as high, further reducing costs.

Prior to conducting the Phase IIa demonstration at Paducah, the consortium completed cost estimates based on costing formulas they developed for zone installation and electricity needed to pass a certain

number of pore volumes of water through the treatment zones (Draft Topical Report for Task #5: Cost Analysis). They then developed 12 test scenarios to measure cost effect due to different parameter changes. These tests showed that the significant cost elements are installation of electrode and treatment zones, and operation and maintenance costs. The scenarios also showed that a significant factor in electrical usage is soil conductivity.

The Jacobs Engineering Group, Inc. (Jacobs) estimated core costs for various in-situ and ex-situ remediation technologies for the Paducah TCE contaminated site. For the Jacobs study, the Lasagna™ consortium estimated the core cost to be \$175/yd<sup>3</sup> for a 4 year cleanup period. It is assumed that this was based to some degree on experience gained from the Phase IIa test.

The costing formulas derived by the consortium can be expressed as:

$$C = C_e + C_t + C_p + C_l + C_r + C_f,$$

where:

$C_e$  = cost of installing electrode zones,

$C_t$  = cost of installing treatment zones,

$C_p$  = electrical cost,

$C_l$  = labor cost,

$C_r$  = rectifier cost, and

$C_f$  = fixed costs.

Included in the fixed costs estimate are costs for the data acquisition system, the electrical distribution system, mobilization and demobilization of all equipment, the fluid handling system, maintenance costs, and contingencies at 35%.

Using the above formula to estimate Phase IIa costs yields a value of \$471.24/yd<sup>3</sup>, which is within 20% of the total core cost shown in Table H-7 of \$578.76. Also, the consortium estimated that electrode and treatment zone construction would average between 20% and 40% of the total core cost and the cost data in Table H-7 verifies that prediction.

Two generators were used in the Phase IIa test. The first, a 50 kW unit, reportedly caused higher than expected electrical losses and thus was replaced by a 100 kW generator on October 1, 1996. According to API records, the 50 kW generator was used for 1,070 hours and the 100 kW generator was used for 6,600 hours. Total generator time is therefore 7,670 hours which checks reasonably well with the 8,000 hours of operation for Phase IIa reported by the consortium.

The consortium reported that a total of 176,000 kilowatt hours (kwh) of electricity was used during the Phase IIa test. Power settings varied from 120 volts to 200 volts but stabilized at approximately 175 volts, DC. Amperage varied from 90 to 220 amps DC. If the 176,000 kwh is DC, then using 0.9 as the rectifier efficiency, this equates to nearly 200,000 kwh AC. Total generator costs were \$180,500. Thus, electrical cost was nearly \$0.90 per kwh, far above local power costs, which are normally in the range of \$0.05 to \$0.09 per kwh.

Pretreatment sampling and design costs are not listed in Table H-7 as costs for these studies apparently were not tracked. These sampling and design costs are not for delineating the extent of contamination, only for the cost of sampling and analyzing soils for assessing Lasagna™ specific parameters, i.e., conductivity and electro-osmotic permeability of soils, and for the design of the zones and the remediation in general. It is assumed that all characterization studies have already been completed by site management to the extent necessary to analyze all treatment options. Pretreatment sampling and design costs for Lasagna™ requirements have been estimated by Dr. Sa Ho, with Monsanto, as probably no more than \$10,000 per average site. The US EPA commonly uses \$20,000 to \$80,000 per site but how much of this cost is general characterization and how much is technology specific characterization is not certain.

## Simple Core Cost Estimates For A Full Scale Lasagna™ Remediation Based on Depth, Time, and Pore Volumes Required

Because the costing formula derived by the consortium appears to be within 20% of costs incurred during the Phase IIa test, it was used to undertake a simple study on the effects of:

- 1.) depth of zone emplacement,
- 2.) the number of years allowed for remediation, and
- 3.) the number of pore volumes required,

on the core cost per cubic yard. Remediation times of from one to five years were studied for 2 and 4 pore volumes, with zone emplacement depths of 15 and 45 feet. It must be remembered that this is only a hypothetical study, and that each site will have its own unique treatment cost based on criteria pertaining to that site.

Other parameters used in this study, which were not varied, were:

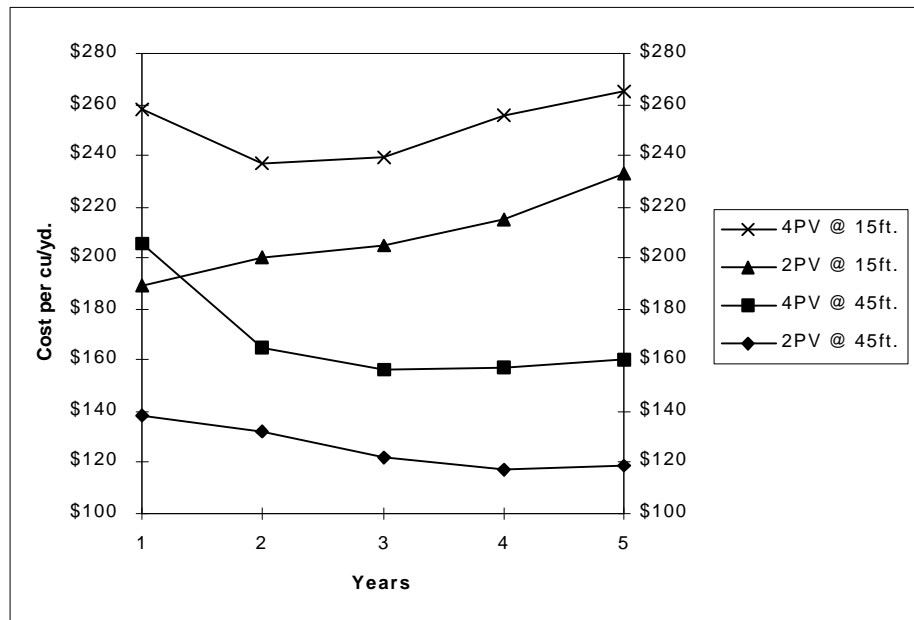
- Pretreatment sampling and design costs = \$20,000
- Area = 60 feet wide by 100 feet long,
- (Electrode and Treatment zones were placed parallel to length of area (each 100 feet wide)
- Cost of installing electrode and treatment zones = \$15.00/ft<sup>2</sup>.
- Soil electrical conductivity of 0.3,
- Electro-osmotic permeability of 0.000015
- Soil Porosity of 0.4
- Voltage optimized to 200 vts, DC
- Labor costs (1 man, half-time, @ \$70/hr.) = \$70,000
- Maintenance costs = \$20,000
- Fixed costs, \$150,000

- Electricity, \$0.05 per kwh

The results of this study on core costs are given in Table H-8 and depicted in Figure H-1. As to be expected, a deeper emplacement effects a larger soil volume, thus costs are lower due to the economy of scale. Also, it will be more costly to move 4 pore volumes of contaminated ground water through the treatment zones than 2 pore volumes. However, three of the curves in Figure 1 suggest that there is an optimum time period for the remediation conditions depicted by that curve, and that there is no monetary advantage to extending treatment time longer, thus emplacing fewer treatment and electrode zones and using a lower voltage.

**Table H-8. Estimated Lasagna™ Remediation Core Costs/yr<sup>3</sup> as a Function of Depth, Pore Volumes Required, and Years Allowed for Remediation**

Year	2PV @ 15ft.	4PV @ 15ft.	2PV @ 45ft.	4PV @ 45ft.
1	\$189	\$258	\$138	\$206
2	\$200	\$237	\$132	\$165
3	\$205	\$239	\$122	\$156
4	\$215	\$256	\$117	\$157
5	\$213	\$265	\$119	\$160



**Figure H-1. Plotting of the Study Values Listed in Table H-8.**

The voltage in the Phase IIa test was stabilized at approximately 175 vts. DC. However, this required a very close spacing of treatment and electrode zones in the

above study. Therefore, voltage was optimized at 200 vts. DC for the study.

## Summary

In summary, the Lasagna™ Phase IIa test indicated that the costing formula developed by the consortium can be used to estimate remediation core costs to within 20% of actual core costs. The above study indicates, that under optimum conditions, Lasagna™ remediation core costs can be as low as approximately \$120/yd<sup>3</sup> of soil remediated to nearly \$260/yd<sup>3</sup> under adverse conditions. These cost estimates are for core costs only, as non-core costs such as oversight, health and safety constraints, sampling, and QA/QC can and will vary significantly from site to site.

## I. *References, Merit of the Technology, Abbreviations and Definitions*

---

More information may be found in Topical Reports written from the Phase I studies funded under DOE contract number DE-AR21-94MC31185.

---

The following 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] or from:

National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161

Phone: (703)487-4600 (Regular Service)

Phone: (703)487-4700 (Rush Service, \$10.00 Additional Per Item)

- **Topical Report for Task No. 1 entitled "Evaluation of Treatment Zone Formation Options" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5436, DE97002165**  
Stephen H. Shoemaker, Richard C. Landis, Ronald J. Griffith, Dale S. Schultz, and Gary E. Quinton (DuPont Company)
- **Topical Report for Tasks No. 2-4 entitled "Electrokinetic Modeling" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5391, DE97002135**  
Andrew P. Shapiro (General Electric Company)
- **Topical Report for Task No. 5 entitled "Cost Analysis" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5389, DE97002134**  
Gary Quinton, Dale Schultz, Richard Landis, Ronald Griffith, and Stephen Shoemaker (DuPont Company)
- **Topical Report for Task No. 6 entitled "Lab-Scale Development of Microbial Degradation Process" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5388, DE97002130**  
J. Martin Odom (DuPont Company)
- **Topical Report for Task #7 entitled "Development of Degradation Processes" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5495, DE97002165**  
M. J. Brackin, M. H. Heitkamp and S. V. Ho (Monsanto Company)
- **Topical Report for Task No. 9-Part I entitled "TCE Degradation Using Non-Biological Methods" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5392, DE97002133**  
Andrew P. Shapiro, Timothy M. Sivavec, and Sunita S. Baghel (General Electric Company)
- **Topical Report for Task #9 - Part II entitled "TCE Degradation Using Non-Biological Methods" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5393, DE97002131**  
Robert G. Orth and David E. McKenzie (Monsanto Company)
- **Topical Report for Tasks #10 entitled "Laboratory and Pilot Scale Experiments of the *Lasagna*<sup>TM</sup> Process" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5375, DE97002150**  
Sa V. Ho, Christopher J. Athmer, and P. Wayne Sheridan (Monsanto Company)  
Andrew P. Shapiro (General Electric Company)
- **Topical Report for Task #11 entitled "Evaluation of TCE Contamination Before and After the Field Experiment" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5496, DE97002166**  
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)
- **Topical Report for Tasks #12 and 13 entitled "Large Scale Field Test of the *Lasagna*<sup>TM</sup> Process" (September 26, 1994 - May 25, 1996), DOE/METC/31185-5390, DE97002156**  
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)  
Dale S. Schultz, Richard C. Landis, Ron Griffith, and Stephen H. Shoemaker (DuPont)

## Merit of the Technology

### **Lasagna™ Technology**

This technology (*Lasagna™*) is an integrated *in-situ* treatment in which degradation zones are installed directly in the contaminated soil and electro-osmosis is utilized to move the contaminants back and forth through those zones until treatment is completed. The technology, once developed, will have tremendous benefits over existing technologies in many aspects including safety, environmental impacts, treatment time, cost, process flexibility, waste generation, breadth of applications, and synergistic effects to other important remedial technologies. The technology would

- greatly reduce public and occupational health risks since the whole treatment is done *in-situ* in the subsurface soil, there will be no excavation and waste handling above ground;
- have minimal environmental impacts since the treatment is carried out under existing natural soil conditions. Injected materials for treatment are benign, e.g. graphite, non-pathogenic bacteria, iron filings;
- be able to treat contamination in low-permeability soils or in heterogeneous soil matrices with possible significant reduction in treatment time. With electro-osmosis the *Lasagna™* technology can move liquid through the low-permeability zones for effective flushing of the contaminants. Other technologies that rely on hydraulic pressure driving force suffer from liquid bypassing that could result in very long treatment time, e.g. one hundred years or so by some estimates for classic pump-and-treat methods;
- enable *in-situ* remediation that is currently not possible due to physical constraints, such as targeted treatment of very deep contamination; *in-situ* remediation under existing structures or residential areas;
- not generate secondary wastes since degradation occurs in place in the subsurface soil. The ground water flowing through the treated soil is totally recycled in place to the other electrode region;
- likely have very favorable cost advantages. The approximate cost estimates carried out show that the cost could be in the range of \$50 to \$100/yd<sup>3</sup> of treated soil (for volumes in excess of 50,000 yd<sup>3</sup>), which is very attractive considering that the treatment is done *in-situ* and for difficult low-permeability soils (where incineration treatment costs could be as high as 10 times this amount);
- have excellent prospect of meeting regulatory requirements with the built-in ability of repeating the *in-situ* treatment simply through the technology's cyclic operation of electrical polarity/flow reversal;
- conceptually be able to handle mixed wastes of organic and inorganic (metals, radionuclides) contaminants, which currently represents a very challenging remediation problem due to treatment incompatibility of these two types of contaminants. The treatment zones in the *Lasagna™* technology could contain sorbents for trapping/immobilizing the inorganics as well as microbes for degrading the organics;
- have the synergistic effects of promoting the development of other innovative remedial technologies such as engineering methods for delivering or recovering materials in subsurface soil (e.g. hydrofracturing), and degradation processes for chlorinated solvents (e.g. aerobic/anaerobic biodegradation, metal-enhanced reduction). These key technologies are components of the *Lasagna™* technology.

## Abbreviations

**CDMFPC** - CDM Federal Programs Corporation's Analytical Laboratory

**DOE** - Department of Energy

**DOE - HQ** - DOE-Headquarters

**DOE - ORO** - DOE-Oak Ridge Operations Office

**DRE** - Destruction and Removal Efficiency

**ECD** - Electron Capture Detector

**ELCD** - Electrolytic Conductivity Detector (formerly called Hall Detector)

**EM** - Environmental Management

**EO** - Electro-Osmosis

**EK** - Electro-Kinetics



**EPA** - Environmental Protection Agency

**EPA/RREL** - EPA's Risk Reduction Engineering Laboratory

**EPA/RTDF** - EPA's Remediation Technologies Development Forum

**EPA/TIO** - EPA's Technology Innovation Office

**ft** - feet

**GC** - Gas Chromatography

**GC/EC** - Gas Chromatography/Electron Capture detector

**GC/ELCD** - Gas Chromatography/Electrolytic Conductivity Detector

**GC/FID** - Gas Chromatography/Flame Ionization Detector

**GC/MS** - Gas Chromatography/Mass Spectrometry

**GC/HD** - Gas Chromatography/Hall Detector

**IEAP** - Integrated Economic Analysis Project implemented by MSE for cost evaluation

**LMES** - Lockheed Martin Energy Systems

**LMUS** - Lockheed Martin Utility Services analytical laboratory at Paducah, Kentucky

**ORNL-GJ** - Oak Ridge National Laboratory located at Grand Junction, Colorado

**P&T** - Purge-and-Trap GC/MS, GC/HD, or GC/ELCD analyses

**PGDP** - Paducah Gaseous Diffusion Plant

**PID** - Photoionization Detector

**PNL (ISIP)** - Pacific Northwest Laboratories *In-Situ* Remediation Integrated Program

**ppbw** - parts-per-billion by weight or µg/L or µg/kg

**ppmw** - parts-per-million by weight or µg/g or mg/kg or mg/L

**ppmv** - parts-per-million by volume or µg/L

**RCI** - Rapid Commercialization Initiative

**RCI Participants** - State, Federal, and Private Industry personnel involved in the verification of the operation and cost of the *Lasagna*<sup>TM</sup> process. A list of those involved is shown in the Verification Statement located in Appendix A.

**RCIWG** - RCI Working Group consisting of representatives of a number of government agencies

**RPD** - Relative Percent Difference =  $100 * (|value1 - value2|) * 2 / (value1 + value2)$

**TCE** - Trichloroethylene

**Weston** - Roy F. Weston Laboratories, Lionville, Pa.

## Electro-Kinetics

Electro-kinetics is a general term used to describe a number of phenomena that occur due to coupling between electrical and hydraulic flows and gradients in the soil-water-electrolyte system where there are charged particles balanced by mobile counter charges (1) (Note: All references are located at the end of this section.). Within the context of remediation, two relevant electro-kinetic phenomena are **electro-osmosis** and **electro-phoresis**. Electro-osmosis is the movement of ion-containing liquid (e.g. water) relative to a stationary charged surface (e.g. soil pores) due to the applied electric field; electro-phoresis, the opposite of electro-osmosis, refers to the movement of charged particles through a stationary liquid. The transport of soluble ions in solution in the presence of an electric field has also been called **electro-migration** (2). The following subsection describes in more details electro-osmosis and its potential applications in waste site remediation.

## Electro-Osmosis

When a dc electric field is applied between two electrodes immersed in a wet soil mass, water in the soil matrix will move from the one electrode to the other electrode. This occurs because of the movement of a thin layer of charged fluid near the pore wall that drags the bulk fluid in the pore along with it. Most soils are negatively charged, which results in cations accumulated near the surface of the soil particles. The thin layer of fluid (called double layer) near the pore wall is thus positively charged, which moves towards the negative electrode (cathode), resulting in a net water flow from the anode (positive electrode) to the cathode. The bulk electro-osmotic flow through soil is normally described empirically by:

$$Q = k_e i_e A$$

where: Q : volumetric flow rate by electro-osmosis (cm<sup>3</sup>/sec)

k<sub>e</sub> : coefficient of electro-osmotic conductivity (cm<sup>2</sup>/volt-sec)

i<sub>e</sub> : voltage gradient applied across the soil mass (volt/cm)

A : cross-sectional area perpendicular to flow (cm<sup>2</sup>)

Note that the electro-osmotic flow is proportional to the applied voltage gradient, and that the electro-osmotic

conductivity has the units of velocity over field strength (cm/sec over volt/cm).

Based on the Hemholtz-Smoluchowski theory, which assumes thin double layer with respect to pore size,  $k_e$  is given by the following relationship (1):

$$k_e = \epsilon \zeta \theta / \mu$$

- where
- $\epsilon$  : permittivity of solution;  $\epsilon = \epsilon_0 D$
  - $\epsilon_0$  : permittivity of vacuum ( $\epsilon_0 = 8.854 \times 10^{-12}$  farad/m)
  - $D$  : dielectric constant of solution (no units)
  - $\zeta$  : zeta potential of the soil (volt)
  - $\theta$  : soil porosity
  - $\mu$  : liquid viscosity (Newton-sec/m<sup>2</sup>), and  $k_e$  in units of m<sup>2</sup>/V-sec (multiply by  $10^{-4}$  to convert to cm<sup>2</sup>/V-sec)

The model thus predicts that the electro-osmotic flow is independent of pore size of the soil matrix. Experimental data obtained for various soil types (Table I-1) present a consistent picture with the model. For soils with hydraulic conductivity values ranging over five orders of magnitude due to the large differences in particle size, the electro-osmotic conductivities obtained vary only a few-fold (1). Electro-osmotic conductivity typically ranges from  $10^{-5}$  to  $10^{-4}$  cm<sup>2</sup>/V-sec and is relatively independent of soil type. It is because of this characteristic that electro-osmosis is effective for moving liquid through fine-grained soils such as clayey, silty soils.

Electro-osmosis has been used since the 1930s for removing water from clays, silts, and fine sands (3). In the late 60s, Monsanto had scaled up the technology and successfully implemented it at a Monsanto Tennessee mine operation for accelerating the settling of fine clay particles in a pond of several acres(4). More recently, electro-osmosis is being investigated as an *in-situ* method for soil remediation (5-10). Water injected into the soil at the anode region flows under electro-osmosis through the contaminated soil, removing the contaminants (metals, organics), then collected at the cathode for further treatment or disposal. Major advantages of electro-osmosis as an *in-situ* remediation method for low-permeability soils include uniform flow distribution in a heterogeneous soil because the flow is independent of pore size; controllable flow direction by the placement of the electrodes and the voltages applied; and low power consumption (9, 11). Table I-2 lists the practical parameter ranges for using

electro-osmosis in remediation, adapted from references 13-14.

During electro-osmosis, however, there are a large number of chemical and electrochemical processes occurring that can negatively affect the electro-osmotic process and the effectiveness of the intended remediation. These include ion diffusion, ion exchange, development of osmotic and pH gradients, soil drying, mineral decomposition, precipitation of salts and secondary minerals, electrolysis, physical and chemical adsorption, soil fabric changes, etc. (12). At the current state of knowledge, the electro-osmosis method appears to suffer from two key limitations that make it somewhat impractical for actual field remediation of organics-contaminated soils. First, the liquid flow induced by electro-osmosis is very slow, about 1 inch per day for clayey soils, which could result in a cumbersome and very long-term operation for large-scale applications. Second, it has been found in several laboratory studies (7-10) that extended operation of electro-osmosis could lead to soil drying and cracking, pH change in the soil bed, and precipitation of metals and minerals near the cathode, which resulted in high electrical resistance and reduced flow, and could eventually stop the process.

**Table I-1. Comparison of Electro-Osmotic Permeability and Hydraulic Conductivity for Various Soils (Adapted from J. Mitchell, (1))**

<u>Material</u>	<u>Water Content (%)</u>	$k_e$ in $10^{-5}$ cm/sec volt/cm	Approximate $k_h$ (cm/sec)
Na-Montmorillonite	170	2.0	$10^{-9}$
Boston blue clay	50.8	5.1	$10^{-8}$
Kaolin	67.7	5.7	$10^{-7}$
Rock Flour	27.2	4.5	$10^{-7}$
Clayey silt	31.7	5.0	$10^{-6}$
Mica powder	49.7	6.9	$10^{-5}$
Fine sand	26.0	4.1	$10^{-4}$
Quartz Powder	23.5	4.3	$10^{-4}$

**Table I-2. Practical Ranges for Electro-osmotic Flushing in Fine-Grained Soils (adapted from Mitchell (13) & Probstein (14)).**

Parameter	Ranges
Soil Porosity	0.1 to 0.7
Hydraulic Conductivity	$10^{-9}$ to $10^{-4}$ cm/sec
Effective Diffusion Coefficient	$2 \times 10^{-6}$ to $2 \times 10^{-5}$ cm <sup>2</sup> /sec
Applied Electric Field	20 to 200 V/m
Electrode Spacing	2 to 10 m
Current Density	0.5 to 5 A/m <sup>2</sup>
Electro-osmotic Conductivity	$10^{-5}$ to $10^{-4}$ cm <sup>2</sup> /V-sec
Electrical Conductivity	100 to 5,000 μS/cm
Effective Ionic Mobility	$3 \times 10^{-5}$ to $10^{-4}$ cm <sup>2</sup> /V-sec

**References**

1. J. K. Mitchell, *Fundamental of Soil Behavior* , Chapter 15 (Wiley, New York, 1976).
2. R. F. Probstein, *Physicochemical Hydrodynamics. An Introduction* (Butterworth-Heinemann, Boston, 1989).
3. Casagrande, L. “*Electro-osmotic Stabilization of Soils*” J. BSCE, 39:51-83 (1952).
4. “Electrical Separation Tested in Phosphate Settling Ponds,” C&EN, pg. 23, Jan. 30, 1984.
5. Shapiro, A.P., Renauld, P., and Probstein, R. “*Preliminary Studies on the Removal of Chemical Species from Saturated Porous Media by Electro-osmosis*” Physicochemical Hydrodynamics, Vol. 11, No. 5/6, pp. 785-802 (1989).
6. Hamed, J., Acar, Y.B., and Gale, R.J. “*Pb(II) Removal from Kaolinite Using Electro-kinetics*” J. Geotech. Eng., ASCE, Vol. 112, pp. 241-271, February (1991).
7. Bruell, C. J. and Segall, B. A. “*Electro-osmotic Removal of Gasoline Hydrocarbons and TCE from Clay*” J. Environ. Eng., Vol. 118, No. 1, pp 68-83, Jan/Feb 1992.
8. Segall, B. A. and Bruell, C. J “*Electro-osmotic Contaminant Removal Processes*” J. Environ. Eng., Vol. 118, No. 1, pp 84-100, Jan/Feb 1992.
9. Acar, Y.B., Li, H., and Gale, R.J “*Phenol Removal from Kaolinite by Electro-kinetics*” J. Geotech. Eng., ASCE, Vol. 118, No. 11, pp. 1837-1852, November 1992.
10. Shapiro, A.P., and Probstein, R.F. “*Removal of Contaminants from Saturated Clay by Electro-Osmosis*” Environ. Sci. Technol. 27, pp. 283-291, 1993.
11. Shapiro, A.P. 1990. “Electro-osmotic Purging of Contaminants from Saturated Soils.” Ph.D. Thesis, Department of Mechanical Engineering, M.I.T., Cambridge, Mass.
12. Mitchell J.K.. *Potential Uses of Electro-kinetics for Hazardous Waste Site Remediation*, Position Paper prepared for USEPA-University of Washington. *Workshop on Electro-kinetic Treatment and its Application in Environmental-Geotechnical Engineering for Hazardous Waste Site Remediation*, Seattle, WA, August 4-5 1986.
13. Mitchell, J.K. (1991). *Conduction Phenomena: From Theory to Geotechnical Practice*, Geotechnique, 41, No. 3, pp. 299-340.
14. Probstein, R.F. and Hicks, R.E. *Removal of Contaminants from Soils by Electric Fields*, Science, Vol. 260, pp. 498-503, April 23, 1993.

## J. Conclusions

The objective of the *Lasagna*<sup>TM</sup> Participants of the Rapid Commercialization Initiative was to demonstrate that the *Lasagna*<sup>TM</sup> Soil Remediation Technology could reduce TCE concentrations in soil to concentrations below 5.6 ppmw ( $\mu\text{g/g}$ ). The demonstration occurred in a treatment cell measuring 21 feet wide by 30 feet long, by 45 feet deep within a larger TCE contaminated volume. The initial soil average concentrations at five locations were 18 (L2A-01), 42 (L2A-02), 52 (L2A-03), 34 (L2A-04), and 34 (L2A-05) ppmw ( $\mu\text{g TCE/g soil}$ ), respectively. After a treatment period of 6 months, concentrations at locations L2A-03, L2A-04, and L2A-05 were reduced to average levels of 2.3, 12.8, and 16.2 ppmw. After a period of 11 months, average concentrations of 0.87, 24, 0.16, 11, and 9.2 ppmw, respectively were measured at the five corresponding locations. At locations L2A-01 and L2A-03, the data indicate that *Lasagna*<sup>TM</sup> cleanup met cleanup goals with the upper 95% confidence bound of the mean being less than the 5.6 ppmw ( $\mu\text{g/g}$ ) action level. At locations, L2A-04 and L2A-05, significant cleanup has occurred but it is uncertain whether the cleanup goal has been met (i. e. only the confidence bound was below the 5.6 ppmw ( $\mu\text{g/g}$ ) limit.). No TCE was measured ( $<1$  ppmv or  $\mu\text{g TCE/liter air}$ ) in air above the site.

An additional goal of the field study was to provide validated cost information which can be used to determine whether this innovative technology is cost competitive with other existing technologies which may also be used for remediating TCE-contaminated clays. Total cost for the installation, operation, and maintenance of the *Lasagna*<sup>TM</sup> Phase IIa test was \$1,375,200, or \$1,310/ $\text{yd}^3$  of remediated soil. Total cost for installation of electrode and treatment zones only was \$491,800, or \$39.14/ $\text{ft}^2$  of zone emplaced. Core technology costs, which can also be considered direct costs, include equipment, labor, and materials used in site preparation, electrode and treatment zone installation, installation of instrumentation and sampling points and monitoring wells, and operation and maintenance, totaled \$579/ $\text{yd}^3$  and \$29.50/ $\text{ft}^2$  of treatment zone and electrode zone emplaced. These costs are higher than estimated for a full-scale cleanup and reflect the high level of support given to this test, which was of a relatively small volume (1,050  $\text{yd}^3$ ). Unit costs for full-scale remediations should be less than those for the Phase IIa test because they will be distributed over larger volumes of soil treated and larger surface areas of electrode and treatment zones installed. Phase IIa cost data, applied to the formula derived by the consortium to estimate remediation costs using *Lasagna*<sup>TM</sup> technology, yielded an estimated total core cost of \$470/ $\text{yd}^3$ , which is within 20% of the actual cost of \$579/ $\text{yd}^3$ . Using this formula for hypothetical remediations to 15 foot and 45 foot depths, for time periods of one to five years, and for treating 2 and 4 pore volumes of vadose water, yielded results that varied from \$190 to \$260/ $\text{yd}^3$  for depths to 15 feet and from \$120 to \$200/ $\text{yd}^3$  for depths to 45 feet. In a Jacobs Engineering Group, Inc study to remediate the Paducah TCE site, the core costs were estimated to be approximately \$175/ $\text{yd}^3$  for a 4 year cleanup period.

Non-core costs such as oversight, sampling, health and safety, QA/QC, etc., cannot be estimated as they are site specific and depend upon, to a great extent, the management objectives at each site.

The analysis of trends in TCE contamination of soil before and after *Lasagna*<sup>TM</sup> treatment clearly indicate that substantial decreases did occur and that the technology can be used to meet regulator accepted action levels. Refer to Sections F and G for further discussion of the *Lasagna*<sup>TM</sup> treatment including chemical evidence of treatment effectiveness and for discussions of the usability of the data.

## Appendix A. Verification Statement



February 18, 1998

### RAPID COMMERCIALIZATION INITIATIVE VERIFICATION STATEMENT FOR *Lasagna*<sup>TM</sup>

**TECHNOLOGY NAME:** *Lasagna*<sup>TM</sup> *in-situ* Soil Remediation Technology

**TECHNOLOGY TYPE:** *In-situ* Soil Remediation

**APPLICATION:** Low-permeability soils and clays and sand mixed with these materials.

**COMPANY:** Monsanto Company

**ADDRESS:** 800 North Lindbergh Boulevard  
St. Louis, MO 63167

**PHONE:** (314) 694-1466/FAX: (314) 694-8080

**REPORT TITLE:** Rapid Commercialization Initiative (RCI) Final Report for an Integrated *in-situ* Remediation Technology (*Lasagna*<sup>TM</sup>)

**REPORT NUMBER:** DOE/OR/22459-1

#### ENVIRONMENTAL PROBLEM

This technology addresses the contamination of low permeability soils with trichloroethylene (TCE). During the 1960s - 1980s TCE was used as a degreasing agent for cleaning aircraft, circuit boards, and as a general low-cost inert solvent at DOD and DOE facilities. Currently TCE is thought to be carcinogenic and has a federal drinking water standard of 5 ppbw (or  $\mu\text{g}$  TCE/L). This technology addresses TCE contamination of soils which is a major source of TCE groundwater contamination at many government and private facilities.

#### TECHNOLOGY DESCRIPTION

The *Lasagna*<sup>TM</sup> Soil Remediation Technology is a soil treatment technology for *in-situ* removal of TCE and other chlorinated solvents from contaminated low-permeability soils, thereby reducing or eliminating the generation of hazardous waste and/or toxic air emissions associated with soil removal and treatment. The technology uses electro-osmosis to move contaminated water (the present verification applies only to TCE contamination) through specially-designed treatment zones that degrade the waste *in-situ*.

#### VERIFICATION SUMMARY

The objective was to demonstrate that the *Lasagna*<sup>TM</sup> Soil Remediation Technology could reduce TCE concentrations in soil to concentrations below 5.6 ppmw ( $\mu\text{g}$  TCE/g soil). The demonstration occurred in a treatment cell measuring 21 feet wide by 30 feet long, by 45 feet deep within a larger TCE contaminated volume. The initial soil average concentrations at five locations were 18 (L2A-01), 42 (L2A-02), 52 (L2A-03), 34 (L2A-04), and 34 (L2A-05) ppmw, respectively. After a treatment period of 6 months, concentrations at locations L2A-03, L2A-04, and L2A-05 were reduced to average levels of 2.3, 12.8, and 16.2 ppmw. After a total treatment period of 11 months, average concentrations of 0.87, 24, 0.16, 11, and 9.2 ppmw, respectively were measured at the five corresponding locations. At locations L2A-01 and L2A-03, the data indicate that *Lasagna*<sup>TM</sup> cleanup met cleanup goals with the upper 95%

confidence bound of the mean being less than the 5.6 ppmw action level. At locations, L2A-04 and L2A-05, significant cleanup occurred but it is uncertain whether the cleanup goal was met (i. e. only the confidence bound was below the 5.6 ppmw limit.). No TCE air emissions were detected (<1 ppmv or µg TCE/liter air) above the treatment cell.

#### DEMONSTRATION DESCRIPTION

- Test Site: This demonstration was conducted at the DOE Gaseous Diffusion Plant in Paducah, KY at a site which was contaminated with trichloroethylene (TCE). The specific site was a former storage cylinder drop test area designated Solid Waste Management Unit (SWMU) 91, comprising approximately 1/2 acre. SWMU 91 has relatively shallow TCE penetration, detailed soil characterization data, and low soil permeability.
- Test Plan Objectives: The objective was to show that the technology could reduce TCE concentrations in low permeability soil to below 5.6 ppmw (µg TCE/g soil). This concentration was the cleanup level established by the Commonwealth of Kentucky for SWMU 91. A secondary objective was to confirm that no significant emissions to air or water occur.
- Technology Description (Technology Elements): The *Lasagna*<sup>TM</sup> Soil Remediation Technology is an *in-situ* technology for treatment of chlorinated organic solvent contamination in moderate- to low-permeability soils (<10<sup>-4</sup> cm/sec), which reduces or eliminates the generation of hazardous waste and/or toxic air emissions associated with soil removal. The technology uses electro-osmosis to move water contaminated with TCE and other contaminants (the present verification applies only to TCE) through specially-designed treatment zones that degrade the waste *in-situ*. Electro-osmosis is an electro-kinetic process that causes water to move through low-permeability soils. A consortium (consisting of Monsanto, Dupont, and General Electric) was formed to develop this technology with participation by the Department of Energy and Environmental Protection Agency. Enviro-Chem Systems, Inc. (Enviro-Chem), a wholly-owned subsidiary of Monsanto Company, licenses and provides services for the technology.
- Technology Description (Technology Elements): The *Lasagna*<sup>TM</sup> Soil Remediation Technology is an *in-situ* technology for treatment of chlorinated organic solvent contamination in moderate- to low-permeability soils (<10<sup>-4</sup> cm/sec), which reduces or eliminates the generation of hazardous waste and/or toxic air emissions associated with soil removal. The technology uses electro-osmosis to move water contaminated with TCE and other contaminants (the present verification applies only to TCE) through specially-designed treatment zones that degrade the waste *in-situ*. Electro-osmosis is an electro-kinetic process that causes water to move through low-permeability soils. A consortium (consisting of Monsanto, Dupont, and General Electric) was formed to develop this technology with participation by the Department of Energy and Environmental Protection Agency. Enviro-Chem Systems, Inc. (Enviro-Chem), a wholly-owned subsidiary of Monsanto Company, licenses and provides services for the technology.

The *Lasagna*<sup>TM</sup> system includes at least one pair of electrodes (anode and cathode) and at least one treatment zone. The treatment zones are sandwiched between the electrodes forming treatment cells of numerous layers from which the "*Lasagna*<sup>TM</sup>" technology derives its name. These layers can be installed in the horizontal or vertical configuration. For the current study, a specially designed mandrel/tremie tube system was used for introducing electrode and treatment zone materials. The length of the mandrel allowed it to be driven to a depth of 45 feet. A crane and a vibratory hammer were used to position and drive the mandrel into the ground. The electrode material consisted of a dry mixture of 50/50 by volume Peerless iron filings and Loresco coke. Six steel rods (3/4" diameter, hot rolled) were inserted into the electrode materials of each electrode, approximately 5 feet apart, to a depth of 40 feet to effect uniform current distribution. A source of alternating current voltage and a rectifier provided direct current voltage to the anode and cathode (steel rods imbedded into iron and carbon zones), thus producing an electric field that causes the water to move away from the anode and toward the cathode. TCE, moving with the water, passes through the treatment zones. The treatment material consisted of Peerless iron filings (60% wt) suspended in wet kaolin clay. When iron filings are used in the treatment zones, the by-products of TCE degradation are primarily acetylene, ethane, and ethylene and chloride and ferric ions. The acetylene, ethane, and ethylene either volatilize, or are transported to the anode(s) or cathode(s). Solvents other than TCE may require the use of materials other than iron filings for treatment. Water

recovered from the cathode gravity drains back via subsurface piping to the anode so the soil will not dry out and clean water will flush contaminants into the treatment zone while electro-osmosis occurs. Typical water flow rates result in estimated cleanup times on the order of months or years. Process water flow velocities vary depending on the specific system (soil type, applied voltage, treatment zone spacing, degree of cleanup, etc.) and range from less than 5 to approximately 30 inches/month. The soil temperature at the center of the treatment soil may reach 80°C or higher while the soil temperature near the surface remains at near ambient temperature.

- **Technology Demonstration/Operating Parameters:** The demonstration occurred in a treatment cell installed in the vertical configuration measuring 21 feet wide by 30 feet long, by 45 feet deep (1,050 yd<sup>3</sup>) within a larger TCE contaminated area. The power delivery reached a maximum of 200 volts and 218 amps until the center (core) temperature reached 83 °C at which point the power was reduced to approximately 180 volts and 180 amps. At the maximum voltage, the water moved through the soil at a rate of 11 inches/month and produced a volumetric flow rate at the cathode of 18 liters/hr.
- **Data Results (Verification of Performance):** Under the authority of the Rapid Commercialization Initiative's Memorandum of Understanding, the Participants in this RCI Project verified the performance of *Lasagna*<sup>TM</sup> *in-situ* Treatment Technology System licensed by Monsanto's Enviro-Chem subsidiary for treating low-permeability soils contaminated with TCE when the technology was installed, operated, monitored, and maintained according to Monsanto Enviro-Chem's standards and specifications. The Participants reviewed plans, data, and reports generated during a demonstration at U.S. DOE's Paducah Gaseous Diffusion Plant and concluded that there was a reasonable basis for rendering a verification decision. The following table compares the soil TCE concentrations measured at five locations in this study to the Commonwealth of Kentucky's accepted site action level:

*Lasagna*<sup>TM</sup> Process - Compliance with Cleanup Target - Statistical Data Summary for TCE in Soil

Location	BEFORE Mean TCE (ppmw (µg/g))	BEFORE 95% Confidence Bounds (ppmw (µg/g))	AFTER Mean TCE (ppmw (µg/g))	AFTER 95% Confidence Bounds (ppmw (µg/g))	Met KY Action Level?
L2A01	18	4.4 to 32	0.87	0.13 to 1.6	yes
L2A02	42	28 to 56	24	13 to 35	no
L2A03	52	20 to 85	0.16	0.018 to 0.31	yes
L2A04	34	17 to 50	11	5.3 to 16	maybe
L2A05	34	17 to 51	9.2	4.7 to 14	maybe

Since the cleanup objective was attained at two of the five locations and the reductions of the remaining three locations were considered sufficient by the DOE site office and its prime contractors, the *Lasagna*<sup>TM</sup> Technology was selected as a treatment option for the site's Proposed Plan. After proper approvals, it may become the preferred remedy for the Record of Decision (ROD) for SWMU 91. The Participants verified that the *Lasagna*<sup>TM</sup> *in-situ* Treatment Technology System may be an acceptable alternative to excavation and above ground treatment that eliminates or reduces the generation of hazardous wastes.

The Participants also evaluated the hazard associated with the air emissions from this system at the Paducah site. During the month of December 1996, 12 flux chambers were placed randomly over the test site. No TCE was observed at concentrations above the detection limit of 1 ppmv (µg/liter). These flux chamber tests addressed only TCE emissions. It is unknown at this time whether other toxic gases may be produced and may need to be controlled.

- **Cost Data:** Total cost for the installation, operation, and maintenance of the *Lasagna*<sup>TM</sup> Phase IIa test was \$1,375,200, or \$1,310/yd<sup>3</sup> of remediated soil. Total cost for installation of electrode and treatment zones only was \$491,800, or \$39.14/ft<sup>2</sup> of zone emplaced. Core technology costs, which can also be considered direct costs, include equipment, labor, and materials used in site preparation, electrode and treatment zone installation, installation of instrumentation and sampling points and monitoring wells, and operation and maintenance, totaled \$579/yd<sup>3</sup> of treatment volume and \$29.50/ft<sup>2</sup> of treatment zone and electrode zone emplaced. These costs are higher than estimated for a full-scale cleanup and reflect the high level of support given to this test, which was of a relatively small volume (1,050 yd<sup>3</sup>). Unit costs for full-scale

remediations should be less than those for the Phase IIa test because they will be distributed over larger volumes of soil treated and larger surface areas of electrode and treatment zones installed. Phase IIa cost data, applied to the formula derived by the consortium to estimate remediation costs using Lasagna™ technology, yielded an estimated total core cost of \$470/yd<sup>3</sup>, which is within 20% of the actual cost of \$579/yd<sup>3</sup>. Using this formula for hypothetical remediations from surface to 15 foot or from surface to 45 foot depths, for time periods of one to five years, and for treating 2 and 4 pore volumes of vadose water, yielded results that varied from \$190 to \$260/yd<sup>3</sup> for depths to 15 feet and from \$120 to \$200/yd<sup>3</sup> for depths to 45 feet. In a Jacobs Engineering Group, Inc study to remediate the Paducah TCE site, the core costs were estimated to be approximately \$175/yd<sup>3</sup> for a 4 year cleanup period.

Non-core costs, which can also be considered indirect costs, include oversight management, health and safety, QA/QC requirements, and sampling and analysis requirements for the Phase IIa test, cannot be estimated for other sites as they are site specific and depend upon, to a great extent, the management objectives at these sites.

- Technical Limitations of the Technology and the Demonstration:

The verification is strictly limited to the demonstration tests of Lasagna™ *in-situ* Treatment Technology System for treating TCE in low permeability soils using iron filings in the vertical configuration as described above. The verification makes no claims concerning the performance or effectiveness of Lasagna™ *in-situ* Treatment Technology System to remove chlorinated solvents from low permeability soils at other sites. The Participants do not know all the possible combinations of solvents and soils and other potential contaminants to which the technology may be applied, nor do the Participants know all of the performance specifications required by end-users. Achieving performance specifications involves many variables including the soil porosity; soil hydraulic and electrical conductivity; the type and amount of contamination; the required cleanup levels; the size, depth, and shape of the site to be cleaned; etc. These factors all affect the benefit that may be realized from use of the Lasagna™ *in-situ* Treatment Technology System.

Additional care must be considered in the construction materials of the anodes. The Phase IIa tests experienced anode corrosion which required replacement of the steel rods after six months of operation. An improved anode design will be used to assure that this does not occur in the full scale cleanup.

Another problem involved migration of TCE from a nearby source into the Phase IIa treatment volume. Highly variable TCE water data, and the lack of complete reduction of TCE concentrations in sampling wells and soil cores, were attributed by the technology holder to DNAPL mobilized from outside the Phase IIa treatment volume. Migration through a porous sand lens at 20 feet below the surface is thought to have caused this phenomenon. However, the technology holder expects that once the whole site is subjected to remediation, this condition will not occur, since all of the contamination will be within the treatment area. Potential end-users must examine their individual processes and product specifications, and work with Monsanto's Enviro-Chem to evaluate and determine whether the Lasagna™ *in-situ* Treatment Technology System can meet the end-user's performance specifications and, if so, what emission controls may be required.

The end-user is ultimately responsible for determining the suitability of the Lasagna™ *in-situ* Treatment Technology System for his specific applications and for complying with the applicable Federal, State, Air Quality Management District (AQMD) and local regulatory requirements. For each specific application, the end-user must ensure compliance with all applicable worker health and safety standards established by OSHA, other federal agencies, and other state and local agencies. Due to the highly varied applications and the wide variety of contaminants, soil types, hydraulic and electrical conductivity, hydraulic permeability, and levels of contamination, the Participants in this RCI Project make no specific recommendations regarding the application of the Lasagna™ *in-situ* Treatment Technology System. The Participants recommend potential end-users contact the manufacturer for suitability for their specific application. The Participants' verification is based on the technology's performance and by itself does not change the regulatory status of the *in-situ* treatment system. Instead, the verification is meant to facilitate and encourage the acceptance of this technology for *in-situ* remediation and to reduce or eliminate the generation of hazardous waste and/or toxic emissions associated with the excavation and above ground treatment of contaminated soils.



- **Demonstration Team Members/Contacts:** A list of the Participants' names, addresses, and phone numbers is given at the end of this statement.

### SIGNATURES

Some of the participants chose to endorse this Verification Statement through separate concurrence letters. These letters are attached immediately following this page.

 <b>Mr. Gerald G. Boyd</b> , Acting Deputy Assistant Secretary for Science and Technology, Office of Environmental Management, USDOE	 <b>E. Timothy Oppelt</b> , Director, National Risk Management Research Laboratory, Office of Research and Development, U. S. Environmental Protection Agency
 <b>Mr. John E. Kilkenny</b> , President, Enviro-Chem Systems, Inc.	 <b>James T. Allen</b> , Ph.D., Chief Office of Pollution Prevention and Technology Development, Department of Toxic Substances Control, California Environmental Protection Agency
 <b>Mr. Kenneth J. Nemeth</b> , Executive Director Southern States Energy Board	 <b>Mr. Rodney Nelson</b> , Assistant Manager for Environmental Management, Oak Ridge Operations
 <b>Mr. Mike Tuck</b> , President, MSE-Technology Applications, Inc.	<u>(See attached letter of concurrence)</u> <b>Mr. Robert Daniell</b> , Director Kentucky Division of Waste Management
<u>(See attached letter of concurrence)</u> <b>Mr. Bill Neimes</b> , Florida Department of Environmental Protection	<u>(See attached letter of concurrence)</u> <b>Mr. Graham Mitchell</b> , Ohio EPA/SWDO
<u>(See attached letter of concurrence)</u> <b>Mr. Brian Moran</b> , Branch Chief, Policy and Regulatory Development, Bureau of Waste Site Cleanup, Massachusetts DEP	<u>(See attached letter of concurrence)</u> <b>Mr. Robert A. (Andy) Binford</b> , State of Tennessee Division of Superfund
<u>(See attached letter of concurrence)</u> <b>Mr. Willie Morgan</b> , Permitting Liaison, EQC Administration, South Carolina Department of Health and Environmental Control	 <b>Dr. B. Mason Hughes</b> , RCI Project Manager, Monsanto Company

JAMES E. BICKFORD  
SECRETARY



PAUL E. PATTON  
GOVERNOR

COMMONWEALTH OF KENTUCKY  
**NATURAL RESOURCES AND ENVIRONMENTAL PROTECTION CABINET**  
DEPARTMENT FOR ENVIRONMENTAL PROTECTION  
FRANKFORT OFFICE PARK  
14 REILLY RD  
FRANKFORT KY 40601

March 24, 1998

Mr. Jimmie Hodges, Site Manager  
U.S. Department of Energy  
Paducah Site Office  
P.O. Box 1410  
Paducah, Kentucky 42001

Dr. B. Mason Hughes  
Senior Analytical Consultant  
Monsanto Company  
800 N. Lindbergh Boulevard  
St. Louis, MO 63167

Re: Concurrence letter for RCI Final Report for an Integrated In-Situ Remediation  
Technology (LASAGNA™) (DOE/OR/22459-1)

Gentlemen:

The Kentucky Division of Waste Management (Division) has reviewed the February 24, 1998, *Rapid Commercialization Initiative (RCI) Final Report for an Integrated in-situ Remediation Technology (Lasagna™)* (DOE/OR/22459-1) and the *Verification Statement* for the same. This letter, as an attachment to the *Verification Statement* provides the Division's concurrence with the RCI Final Report for Lasagna™.

The Division staff has followed the demonstration of this technology through review of reports, data, monthly conference calls, and site demonstration tour. In the demonstration at Paducah, significant reductions in TCE concentrations were obtained during the eleven-month test period. The Division recognizes Lasagna™ as a viable technology for the remediation of the low-permeability contaminated soils in Paducah provided the Remedial Action Plan or other design documents incorporating this technology are approved by the Division.

Please note that this concurrence is based on an evaluation of the technology performance under specific, predetermined criteria, and appropriate quality assurance procedures. The

Division makes no expressed or implied warranties as to the performance of the technology and does not certify that this technology will always, under circumstances other than those tested, operate at the levels demonstrated. This concurrence is not an endorsement of this technology by the Division and should not be used as such. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements.

If you have any questions about Division's involvement in RCI LASAGNA™ technology, please contact Tuss Taylor at (502)-564-4797.

Sincerely,



Robert H. Daniell, Director  
Division of Waste Management

RHD/TT/jkg

cc: Robert Sleeman, DOE  
Carl R. Freode Jr., USEPA  
Jimmy C. Massey, LMES  
Myrna Redfield, DOE  
John Morgan, LMES  
KDWM-Paducah  
DWM Main File #980



## Department of Environmental Protection

Lawton Chiles  
Governor

Twin Towers Office Building  
2600 Blair Stone Road  
Tallahassee, Florida 32399-2400

Virginia B. Wetherell  
Secretary

February 18, 1998

Mr. B. Mason Hughes  
Monsanto Company  
800 N. Lindbergh Boulevard  
St. Louis, Missouri 63161

Re: Verification Statement  
Lasagna™ In-situ Soil Remediation Technology  
Rapid Commercialization Initiative

Dear Mr. Hughes:

As an active participant in the review of the Lasagna™ In-situ Soil Remediation Technology and a representative from the Florida Department of Environmental Protection, I am writing this letter as an attachment letter of concurrence to the verification statement. I was privileged to be one of several state representatives who attended the demonstration tour in Paducah, Kentucky on September 17, 1996. Subsequent to this tour I have been involved through the review of reports, monitoring data, and monthly teleconferences detailing the Lasagna™ in-situ soil remediation test site in Paducah.

Working with the RCI participants, I recognize Lasagna™ as a viable technology for the remediation of low-permeability contaminated soils in Florida provided: (a) considerations in the Rapid Commercialization Initiative (RCI) Final Report for an in-situ Remediation Technology (Lasagna™) are taken into account; and (b) a Remedial Action Plan or other design document incorporating this technology is approved by the Department.

While the Department of Environmental Protection does not provide endorsement of a specific remediation process, it does recognize the need to determine acceptability from an environmental standpoint with respect to applicable rules and regulations, and the interests of public health, safety, and welfare. A vendor must then market the process on its own merits regarding performance, cost, and safety in comparison to competing alternatives in the marketplace.

Preparers of a Remedial Action Plan or other design document may include a copy of this letter and a copy of the verification statement in the appendix of plans they submit, and call attention to it in the text of their document. In this way, technical reviewers throughout the state will be informed that the Department of Environmental Protection has determined the environmental acceptability of this remediation process.

*"Protect, Conserve and Manage Florida's Environment and Natural Resources"*

*Printed on recycled paper.*

Mr. Mason Hughes  
February 18, 1998  
Page Two

The Department acceptance of this remediation process does not imply it has been deemed applicable for all cleanup situations, or that it is preferred over other treatment or cleanup techniques in any particular case.

A site specific evaluation of applicability and cost-effectiveness must be considered for any remediation process, whether conventional or innovative, and adequate site specific design details must be provided in a Remedial Action Plan or other design document prescribing the remediation process.

Please incorporate this letter into the verification statement. If you have any questions on this letter or my involvement in the RCI Lasagna™ In-situ Soil Remediation Technology review demonstration, please call me at (850) 488-3935.

Sincerely,

*Bill Neimes*

Bill Neimes, P.E.  
Bureau of Waste Cleanup  
Florida Department of Environmental Protection



State of Ohio Environmental Protection Agency

## Southwest District Office

401 East Fifth Street  
Dayton, OH 45402-2911

TELE: (937) 285-6357 FAX: (937) 285-6249

George V. Voinovich, Governor  
Nancy P. Hollister, Lt. Governor  
Donald R. Schregardus, Director

March 3, 1998

RE: LASAGNA<sup>(TM)</sup> RCI  
CONCURRENCEMr. B. Mason Hughes  
Senior Analytical Consultant  
Monsanto Company  
800 N. Lindbergh Boulevard  
St. Louis, Missouri 63161

Dear Mr. Hughes:

The purpose of this letter is to provide Ohio EPA's concurrence with the "Rapid Commercialization Initiative Final Report for an Integrated In-Situ Remediation Technology (LASAGNA<sup>(TM)</sup>)". This is a soil treatment technology for in-situ removal of TCE and other chlorinated solvents from low permeability soils. The technology uses electro-osmosis to move contaminated water through specially designed treatment zones.

Ohio EPA staff have followed the demonstration of this technology for the past two years through review of work plans, conference calls, reports, etc. Ohio EPA staff have visited and toured the demonstration site at the DOE Paducah Gaseous Diffusion Plant in Kentucky. In addition, staff have also observed this technology demonstrated at the DOD Rickenbacker Air National Guard Base near Columbus, Ohio.

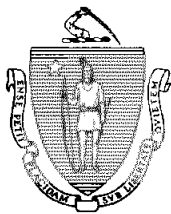
In the demonstration in Paducah, significant reductions in TCE concentrations occurred during the eleven month test period. Ohio EPA observed that this technology has been adequately demonstrated and that this technology may be applicable to many other contaminated sites.

Please note that this concurrence is based on an evaluation of technology performance under specific, predetermined criteria and appropriate quality assurance procedures. Ohio EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that any technology will always, under circumstances other than those tested, operate at the levels in these demonstrations. This concurrence is not an endorsement of this technology by Ohio EPA, and should not be used as such. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements.

If you have any questions about Ohio EPA's involvement in this RCI LASAGNA<sup>(TM)</sup> technology demonstration, please contact me at 937-285-6018.

Sincerely,

Graham E. Mitchell  
Chief, Office of Federal Facilities Oversightcc: Catherine Stroup, Legal  
Tom Velalis, DERR/CO



ARGEO PAUL CELLUCCI  
Governor

COMMONWEALTH OF MASSACHUSETTS  
EXECUTIVE OFFICE OF ENVIRONMENTAL AFFAIRS  
DEPARTMENT OF ENVIRONMENTAL PROTECTION  
ONE WINTER STREET, BOSTON, MA 02108 617-292-5500

TRUDY COXE  
Secretary  
DAVID B. STRUHS  
Commissioner

May 7, 1998

Mr. B. Mason Hughes  
Monsanto Company  
800 N. Lindbergh Boulevard  
St. Louis, Missouri 63161

RE: Verification Statement  
Lasagna™ In-situ Soil Remediation Technology  
Rapid Commercialization Initiative

Dear Mr. Hughes:

The Massachusetts Department of Environmental Protection (DEP) supports all efforts to encourage the efficient, safe and effective cleanup of waste sites through accelerating the acceptance and commercial use of innovative technologies.

Several staff from the Department's Waste Site Cleanup Program have reviewed the ITRC Lasagna™ In-situ Soil Remediation Technology related documents and participated the conference calls concerning data generated from the test site in Paducah, Kentucky. I concur with the other RCI participants that the technology is viable for remediating low-permeability contaminated soils. The flexibility allowed by the Massachusetts Contingency Plan (MCP) in the implementation of a remedial technology, however, prevents the agency from approving this technology for application at any specific site in Massachusetts. The "privatized" site cleanup program uses Licensed Site Professionals (LSPs), who are senior consultants licensed by a state board, to ensure compliance with the MCP (see 310 CMR 40.0000). Although subject to audits by the DEP, LSPs apply their professional judgment to clean up sites without direct state oversight to meet the MCP performance standards.

Please incorporate this letter into the verification statement. If you have any questions concerning this letter or the Department's involvement in the Lasagna™ In-situ Soil Remediation Technology review process please contact Kendall Marra, Innovative Technology Coordinator, Bureau of Waste Site Cleanup at 617.292.5966.

This information is available in alternate format by calling our ADA Coordinator at (617) 574-6872.

DEP on the World Wide Web: <http://www.magnet.state.ma.us/dep>

Printed on Recycled Paper

Sincerely

A handwritten signature in cursive script that reads "Brian P. Moran". The signature is written in black ink and is positioned above the printed name.

Brian P. Moran  
Chief, Policy and Regulation Development  
Bureau of Waste Site Cleanup

March 2, 1998

Appendix A-12





STATE OF TENNESSEE  
DEPARTMENT OF ENVIRONMENT AND CONSERVATION

Division of Superfund  
4th Floor, L&C Annex  
401 Church Street  
Nashville, Tennessee 37243-1538

March 5, 1998

Mr. B. Mason Hughes  
Senior Analytical Consultant  
800 N. Lindbergh Boulevard  
St. Louis, Missouri 63161

Subject: *Lasagna*<sup>TM</sup> demonstration  
DOE, Paducah, Gaseous Diffusion Plant

Dear Mr. Hughes:

The Tennessee Department of Environment and Conservation, Division of Superfund (DSF), has participated in several of the *Lasagna*<sup>TM</sup> RCI activities. These activities included participating in several conference calls; reviewing reports including the Final Quality Assurance Project Plan (QAPP) dated August 20, 1996 and the Rapid Commercialization Initiative (RCI) Final Report for an Integrated in-situ Remediation Technology (*Lasagna*<sup>TM</sup>) dated February 6, 1998; and touring the demonstration site at the DOE Paducah Gaseous Diffusion Plant in Kentucky.

Based on these activities, the DSF concurs that it has been demonstrated that the *Lasagna*<sup>TM</sup> treatment technology utilizing electro-osmosis in conjunction with treatment cells composed of a mixture of peerless iron filings and kaolin has removed significant amounts of TCE from soil at the Paducah Gaseous Diffusion Plant demonstration site. Pre and post soil sampling data given in Tables F-5 through F-7 and Figures F-1 and F-2, and treatment effectiveness for the zone of contamination given in Table G-2 of the final report demonstrate this TCE removal. The "Summary of C<sub>2</sub> Hydrocarbon Degradation Products Measured in Water Samples" included in Table F-11 of the final report shows that the TCE removal was due to the *Lasagna*<sup>TM</sup> demonstration since the acetylene, ethane, and ethylene degradation products are present in the *Lasagna*<sup>TM</sup> treatment demonstration area and are not present in the TCE contaminated control area (MW-160). Figure C-4 shows that MW-106 is approximately 25 feet from the *Lasagna*<sup>TM</sup> demonstration area.

There is some uncertainty in the demonstration final report associated with the site soil composition. Page C-9 (Table C-1) shows that the soil at the demonstration site is a clay loam with a low (estimated  $1 \times 10^{-7}$  cm/sec) hydraulic conductivity. On page F-12 it states that there is a permeable sand layer located at a depth of 20 to 25 feet. On page F-12 it further states that the water samples may not be a good indicator of soil cleanup during the test if the TCE is migrating in from outside the demonstration area. It is therefore unclear how much contaminant migration through the treatment zones

March 2, 1998

Appendix A-13

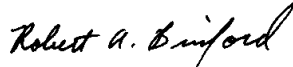
in the sand layer was caused by electro-osmosis and how much was caused by ground water movement through the sand layer.

The DSF believes that with the abundance of low permeability soil in the environment, there are many sites where the *Lasagna*<sup>TM</sup> technology may be applicable. Each of these sites has unique characteristics including, but not limited to: soil composition, sorting, roundness and porosity; the distribution of soil composition and characteristics in the contaminated area; soil hydraulic and electrical conductivity; the type and amount of contamination; the required cleanup levels; the size, depth, and shape of the contamination; the presence of buildings and structures at the site; etc. The applicability, site specific design specifications, and cost effectiveness of the *Lasagna*<sup>TM</sup> technology should be determined by the end user and the manufacturer prior to implementation. The end user is solely responsible for determining the suitability of *Lasagna*<sup>TM</sup> for his specific application and for complying with any and all applicable federal, state, and local requirements.

The DSF makes no expressed or implied warranties as to the performance of the technology and does not certify that any technology will operate at the levels obtained in the demonstration. This concurrence letter is meant to facilitate and encourage the acceptance of this technology for *in-situ* remediation, and to reduce or eliminate the generation of hazardous waste and/or toxic emissions associated with the excavation and above ground treatment of contaminated soils.

If you have any questions, please call me at (615) 532-0911.

Sincerely,



Robert A. (Andy) Binford, Manager  
Technical and Contracting Section



2600 Bull Street  
Columbia, SC 29201-1708

March 11, 1998

**CERTIFIED MAIL**

Mr. B Mason Hughes, Project Manager  
Monsanto Company  
800 N. Lindbergh Boulevard  
St. Louis, Missouri 63167

RE: Rapid Commercialization Initiative (RCI) Verification Statement for the Lasagna™ In-Situ Soil Remediation Technology.

Dear Mr. Hughes:

The purpose of this letter is to provide South Carolina Department of Health and Environmental Control's (SCDHEC) concurrence with the technical approach employed for the development, field testing, and performance of the Lasagna™ In-Situ Soil Remediation Technology under appropriate quality control assurance procedure and specific criteria.

As a representative of SCDHEC, I was privilege to actively participate in the review of various documents (work plan and reports), phone conferences for comment discussion, and the field demonstration held at Paducah, Kentucky in September 1996. Working with other State representatives and from the real time field data, it is acknowledged that Lasagna™ is a viable technology for remediating the low-permeability soils and clays and sand mixed with these materials contaminated with TCE and other chlorinated solvents.

This concurrence by SCDHEC is not an endorsement for the referenced technology and should not be used as such. The vender while marketing the referenced technology for its performance, costs, acceptability, safety and use, is solely responsible for complying with any federal, state, and/or local regulations that may apply. A vender may include the copy of this letter along with the verification statement while proposing the use of the referenced technology to any SCDHEC representative. This will allow the involved state regulator to communicate with appropriate individuals within the agency and will facilitate the vender in presenting its approach.

SOUTH CAROLINA DEPARTMENT OF HEALTH AND ENVIRONMENTAL CONTROL

If you should have any questions about my or SCDHEC involvement in the RCI Lasagna™ technology demonstration, please contact me at 896-4088.

Sincerely,

*m. p. mehta*

Mihir P. Mehta, Environmental Engineering Associate  
Federal Facility Agreement Section  
Division of Site Assessment and Remediation  
Bureau of Land and Waste Management

cc: Keith Collinworth, P.G., Manager (Federal Facility Agreement Section)  
J. Keith Lindler, P.E., Director (Division of Site Assessment & Remediation)  
Ken Taylor, P.G., Director (Division of Hydrogeology)  
Willie Morgan (EQC Administration)

**DISCLAIMER**

**Notice:** This verification is based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. The signatories make no expressed or implied warranties as to the performance of the technology and do not certify that a technology will always, under circumstances other than those tested, operate at the levels verified. The end user is solely responsible for complying with any and all applicable Federal, State and Local requirements.

**For more information, you may contact any of the following *Lasagna*<sup>TM</sup> Soil Remediation Technology Participants:**

<p><b>Mr. Grover (Skip) Chamberlain</b> DOE Project Coordinator USDOE, 19901 Germantown Road, Mail Stop EM 53 CL, Germantown, MD 29874 <u>Voice:</u> (301) 903- 7248 <u>FAX:</u> (301) 903-7457 <u>FAX2:</u> (301) 903-7234 grover.chamberlain@em.doe.gov</p>	<p><b>Mr. Thomas J. Holdsworth</b> Federal EPA Representative USEPA, NRMRL ,Mail Stop 489, 26 W. Martin Luther King Drive, Cincinnati, OH 45268 <u>Voice:</u> (513) 569-7675 <u>FAX:</u> (513) 569-7676; alt. (513) 569-7620 holdsworth.thomas@epamail.epa.gov</p>	<p><b>Mr. Stephen Antonioli</b> MSE Cost Coordinator Manager, Integrated Economic Analysis Project, MSE-TA, 200 Technology Way or P. O. Box 4078 Butte, MN 59701 or 59702 <u>Voice:</u> (406) 494-7343, <u>FAX:</u> (406) 494-7230, e-mail: santonio@in-tch.com</p>
<p><b>Mr. Tuss Taylor</b> Kentucky EPA Kentucky Div of Waste Manag, 14 Reilly Road, Frankfort, KY 40601 <u>Voice:</u> (502)564-4797, <u>FAX:</u> (502)564-5096 tmtayl1@service1.uky.edu and Dan Moore ext. #295, djmoor1@service1.uky.edu <b>Mr. Daniel Moore, U. of Kentucky-FFOU</b> P. O. Box 776, Frankfort, KY 40602 <b>Mr. Jayant Gotpagar, UK-FFOU,</b> jayant@enr.uky.edu</p>	<p><b>Dr. Jerry Hill</b> SSEB Representative 6325 Amherst Court Norcross, GA 30092 Voice: (770)242-7712, FAX: (770)242-0421 e-mail: hill@clever.net and Ted Joy, joy@clever.net, Voice: (770)242-7712, FAX: (770)242-0421</p>	<p><b>Dr. B. Mason Hughes</b> RCI and Phase IIa Project Manager Monsanto Co. 800 N. Lindbergh Boulevard St. Louis, MO. 63167 <u>Voice:</u> (314) 694-1466 <u>FAX1:</u> (314) 694-8080 <u>FAX2:</u> (314) 694-1531 b.mason.hughes@monsanto.com</p>
<p><b>Mr. Brian Moran</b> Branch Chief, Policy and Regulatory Development, Mass. DEP/BWSC 1 Winter St., 7th Floor Boston, MA 02108 <u>Voice:</u> (617)292-5767 <u>FAX:</u> (617)992-5530</p>	<p><b>Mr. Bill Neimes</b> Florida DEP 2600 Blairstone Road Tallahassee, FL 32399-2400 <u>Voice:</u> (904)921-9986 <u>Fax:</u> (904)922-6657</p>	<p><b>Mr. David Carden</b> DOE Verification Entity USDOE, 55 Jefferson Avenue Oak Ridge, Tenn. 37831 <u>Voice:</u> (423) 576-9262, <u>FAX:</u> (423) 576-6074, e-mail: cardendm@oro.doe.gov</p>
<p><b>Mr. Carl Froede</b> Region IV EPA 100 Alabama St. N. W., Atlanta, GA 30303, <u>Voice:</u> (404) 562-8550, <u>FAX:</u> (404) 562-8518, e-mail:carl@epamail.epa.gov</p>	<p><b>Mr. Willie Morgan</b> EQC Administration SCDHEC, 2600 Bull Street Columbia, SC 29201 <u>Voice:</u> (803)734-5179, <u>Fax:</u> (803)734-9196</p>	<p><b>Mr. Graham Mitchell, OEPA/SWDO</b> 401 East 5th Street Dayton, Ohio 45402 <u>Voice:</u> (937)285-6018, <u>FAX:</u> (937)285-6249</p>
<p><b>Mr. Jim Haynes, PE</b> Tenn. Environmental Policy Office, Dept. of Environment and Conservation 20th Floor, L&amp;C Tower, 401 Church Street Nashville, Tenn 37243-0454 <u>Voice:</u> (615)532-0227, <u>FAX:</u> (615)532-0740, jhaynes@mail.state.tn.us</p>	<p><b>Mr. Sam Hayes</b> QAPP Reviewer for Federal EPA USEPA, NRMRL , 26 W. Martin Luther King Drive, Cincinnati, OH 45268, <u>Voice:</u> (513) 569-7514, <u>FAX:</u> (513) 569-7585, e-mail: hayes.sam@epamail.epa.gov</p>	<p><b>Mr. Robert A. (Andy) Binford</b> State of Tennessee Division of Superfund 4<sup>th</sup> Floor LNC Annex, 401 Church St. Nashville, Tenn. 37243-1538, <u>Voice:</u> (615)532-0911, <u>FAX:</u> (615)532-0938</p>

Mr. Mihia P. Mehta  
 SCDHEC  
 2600 Bull Street  
 Columbia, SC. 29201  
 Voice: (803)896-4088  
 FAX: (803)896-4001

Mr. Ronald E. Lewis, PE  
 Cal/EPA Representative  
 DTSC (HQ-27), P. O. Box 806  
 Sacramento, CA 95812-0806, or  
 301 Capitol Mall, Sacramento, CA  
 95814  
 Voice: (916) 322-6872, FAX: (916)  
 324-3107

### DESCRIPTION OF RCI

Rapid Commercialization Initiative (RCI) is a component of the federal Administration's efforts to build cooperative interactions between the private sector, states, and federal agencies to advance a national environmental strategy and bring environmental technologies to market more rapidly and efficiently. As a result of RCI, a Memorandum of Understanding (MOU) was written to accelerate private sector commercialization of innovative environmental technologies and to facilitate regulatory acceptance across state and federal jurisdictions. The desired product of the MOU is multi-state acceptance of innovative environmental technologies following verification of the performance of those technologies.

The MOU was made and entered into by and between the following parties:

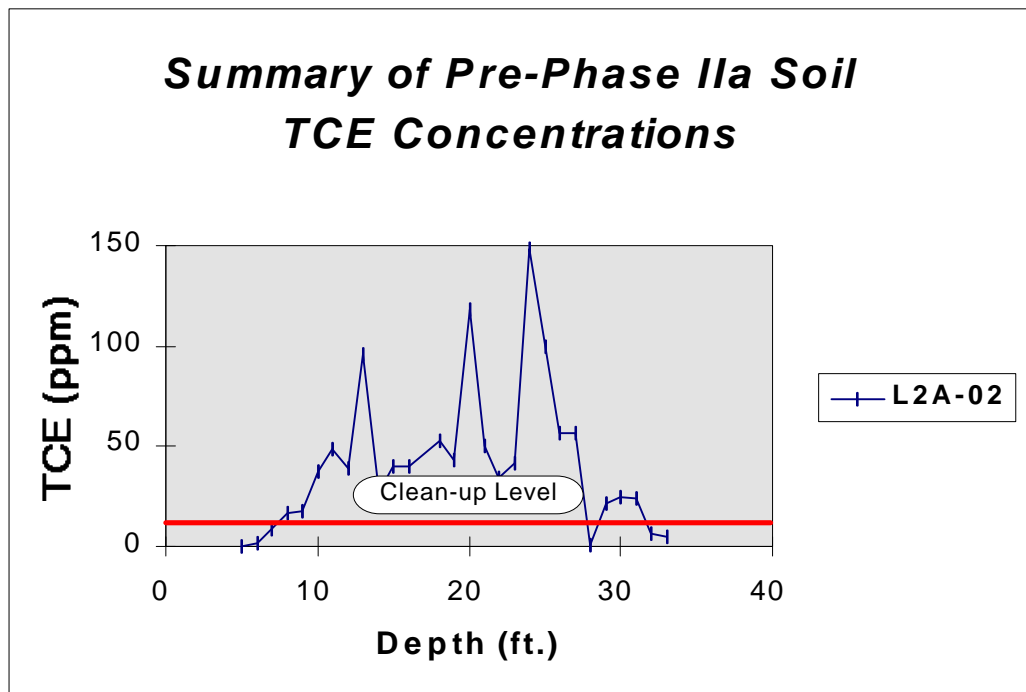
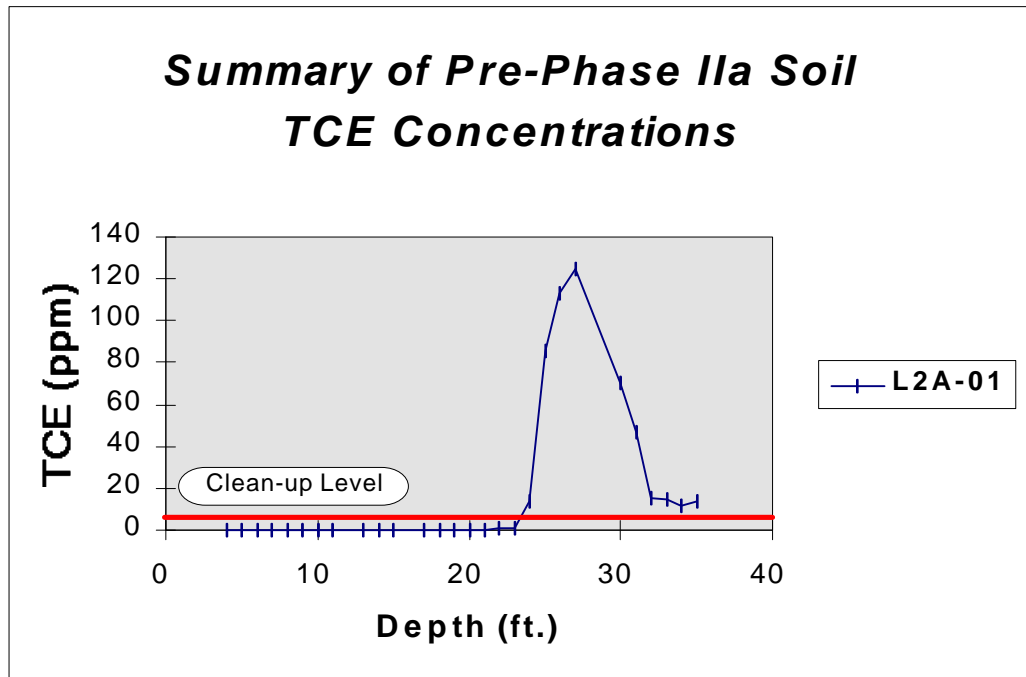
U.S. Department of Commerce, U.S. Department of Defense, U.S. Department of Energy, U.S. Environmental Protection Agency, Southern States Energy Board, Western Governors' Association, and the State of California Environmental Protection Agency.

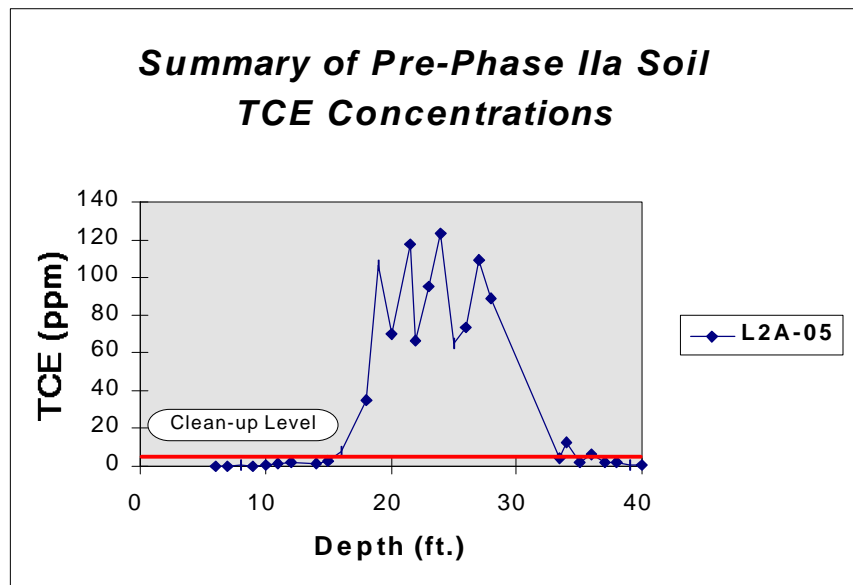
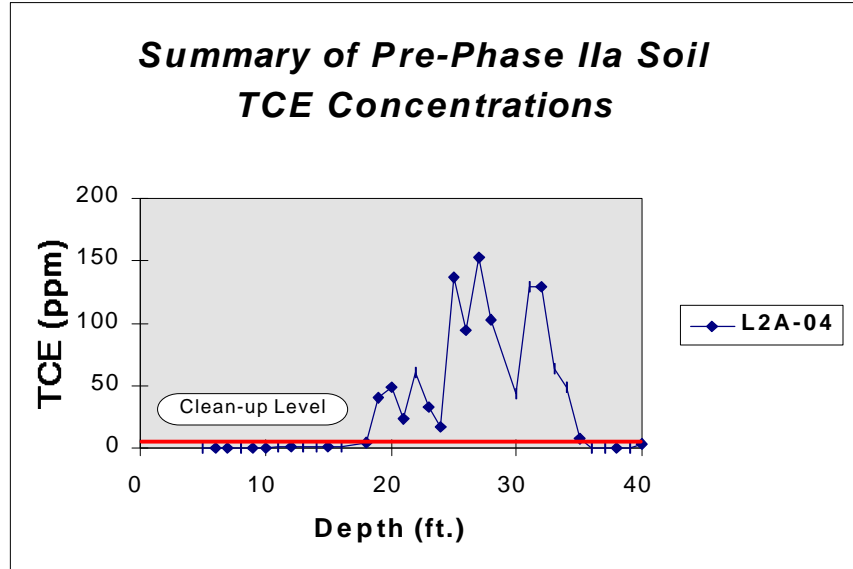
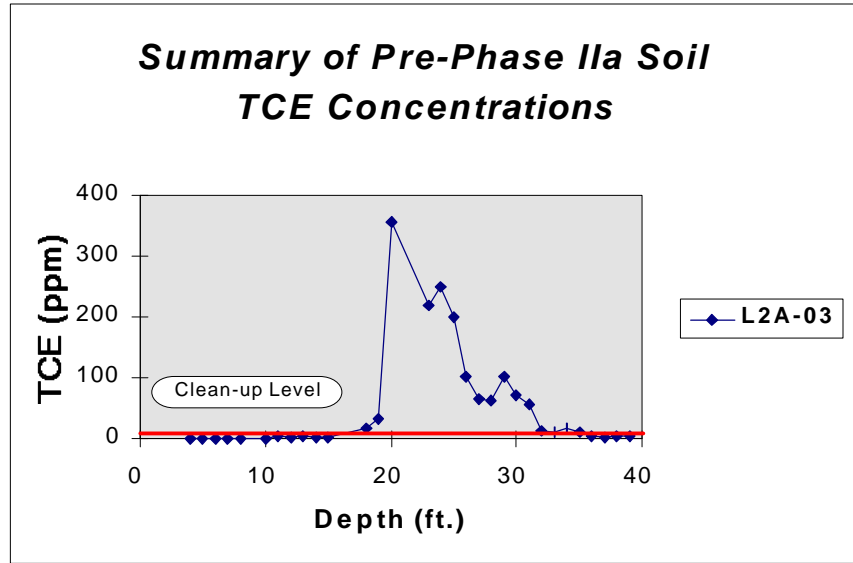
Concurrence and sign off of the MOU was completed August 14, 1995.

The MOU resulted in a federal/state/private cooperative effort (the RCI Program or the Program) to expedite the application of new environmental technologies. The RCI Program identifies barriers to the acceptance and use of new technologies and makes use of cooperative demonstration projects to remove these barriers, if possible. The Program includes 10 individual demonstration projects, each of which will involve a different environmental technology.

Implementation of an MOU between Monsanto, the technology holder, and the U.S. Department of Energy is authorized by Cooperative Demonstration Agreement number 96-RCI-02. For *Lasagna*<sup>™</sup>, participating federal agencies include the Department of Commerce, Department of Defense, Department of Energy and the Environmental Protection Agency. Participating states and state organizations include the State of California Environmental Protection Agency, Southern States Energy Board, and the Western Governors Association. Additional participants and also signatory states for this verification of the *Lasagna*<sup>™</sup> *in-situ* Treatment Technology are Florida, Massachusetts, Kentucky, Ohio, and South Carolina.

**Appendix B. Pre-Test Concentrations Obtained in March 1996**





March 2, 1998



**Table Appendix B-1. Pre-Lasagna™ Soil TCE Concentrations**

Depth(ft)	L2A-01	Depth(ft)	L2A-02	Depth(ft)	L2A-03	Depth(ft)	L2A-04	Depth(ft)	L2A-05
4	43								
5	56	5	106			5	8		
6	71	6	1,533	4	0	6	41	6	188
7	42	7	8,577	5	507	7	50	7	327
8	38	8	16,992	6	390	8	120	8	360
9	40	9	17,530	7	782	9	109	9	331
10	24	10	37,330	8	728	10	91	10	516
11	16	11	49,007	10	937	11	996	11	1,080
13	0	12	38,794	11	5,162	12	772	12	1,979
14	7	13	96,064	12	2,341	13	913	14	1,607
15	13	14	27,183	13	4,009	14	900	15	3,106
16		15	39,888	14	2,182	15	824	16	7,539
17	0	16	39,934	15	2,638	16	891	17	35,113
18	0	17		18	17,684	18	4,114	18	106,311
19	0	18	52,930	19	31,909	19	40,232	19	117,887
20	14	19	43,303	20	357,067	20	48,589	20	66,432
21	63	20	118,250	23	218,566	21	23,166	21	95,270
22	800	21	50,131	24	250,465	22	59,951	22	123,366
23	1,020	22	34,220	25	199,155	23	32,322	23	65,100
24	13,693	23	41,380	26	101,415	24	16,595	24	73,267
25	85,830	24	148,159	27	64,480	25	136,417	25	108,905
26	113,043	25	99,561	28	63,874	26	94,367	26	88,592
27	124,915	26	57,014	29	101,501	27	152,025	27	4,350
30	70,189	27	56,319	30	71,074	28	102,216	28	12,271
31	46,645	28	697	31	57,048	30	43,639	29	1,870
32	15,219	29	21,669	32	13,571	31	129,399	30	6,586
33	14,885	30	25,094	33	11,269	32	129,332	31	2,297
34	11,134	31	23,903	34	17,246	33	63,678	32	1,816
35	13,518	32	6,767	35	11,470	34	48,924	33	647
		33	4,806	36	4,834	35	7,139	34	410
				37	2,739	36	0	35	
32	21,085			38	4,004	37	0	36	
32D	9,352	31	30,741	39	3,747	38	197	37	
33	11,922	31D	17,065			39	444	38	
33D	17,847	32	4,067			40	3,086	39	
		32D	9,468					40	
				20	308,686				
				20D	405,448	24	16,124		
				39	4,143	24D	17,066		
				39D	3,351	38	0	40	427
						38D	393	40D	394
<b>Average:</b>	<b>18,261</b>	<b>41,327</b>		<b>52,348</b>		<b>33,575</b>		<b>34,398</b>	
<b>Concentrations (ug/kg)</b>									

**Average Concentration for Total Phase Ila Site: 35,982**

Note: The above data shown in double-line boxes are plotted in the 5 figures shown on the preceding pages. Below each boring, are duplicate data for selected depths. For example, the L2A-01 boring has duplicate values for the 32-ft. and 33-ft. depths. The average values for these depths were calculated and placed in the table and plotted in the figures. This was done for QA purposes and to determine how uniform the soil TCE concentrations were within a core sample.

## Appendix C. Sections E - G in QAPP

### E. Data Quality Objectives

*The evaluation of a new integrated in-situ remediation technology, which combines electro-osmosis with zero valent iron degradation techniques, will be made by using well-defined QA objectives.*

The present QA Objectives have been developed so that the data obtained from the Phase II study may be used to produce validated data that can be used by DOE, DOD, and state environmental agencies, to evaluate the process' success and thus to allow the use of *Lasagna*<sup>TM</sup> for remediation of low permeability soils with TCE contamination. For the present site, a wide range of TCE concentrations have been measured in soil in the Phase I study and in the pre-test samples which ORNL/GJ obtained in March 1996 for the Phase IIa study. Therefore soil and carbon analytical data will be obtained by ORNL/GJ at 1-ft depth intervals after the field experiments are complete to more accurately determine the average TCE concentration after conducting the field experiment. The average TCE concentrations will be calculated from the concentrations measured at 1-ft. Intervals for the 5 soil borings after performing the *Lasagna*<sup>TM</sup> process. In order to visualize the pre-test concentration-depth profiles of this pre-test data, figures summarizing the concentrations measured in March 1996 are shown in Appendix A. The soil boring locations shown in the appendix (i. e. L2A-01, L2A-02, etc.) are the same sampling locations shown in Figure C-3 as 2A-01, 2A-02, etc. Discrete soil TCE concentrations are also shown in the table in Appendix A. These pre-test soil boring locations have been used to create the sampling wells from which bi-weekly water samples will be obtained. The data shown in Appendix A shows that several soil TCE concentrations were measured to be greater than 100 ppmw ( $\mu\text{g/g}$ ) at depths between 15 and 30 feet below ground surface (bgs), with an extreme value exceeding 400 ppmw ( $\mu\text{g/g}$ ).

#### Phase II Field Experiment

In order for the *Lasagna*<sup>TM</sup> process to be a technical success for this site in the State of Kentucky, the mean TCE soil concentrations will be determined after 1 - 2 years of operation. These soil samples will be taken adjacent to the pre-test samples and the means must be below approximately 5.6 ppmw ( $\mu\text{g/g}$ ). (This action level was determined by LMES, DOE, and the State of Kentucky, based on groundwater modeling calculations.) The figures in Appendix A show graphically this approximate action level. The overall success of the *Lasagna*<sup>TM</sup> process for the Paducah site will be decided by the success in achieving TCE treatment of contaminated soils to a mean concentration of less than 5.6 ppm wet weight. This will

be determined by statistical analysis of the soil concentration data after 1 - 2 years of operation of the Phase II field experiment and applying the following decision rule:

- **If the upper 95% confidence level of the mean of the soil sample population is less than 5.6 ppm then the *Lasagna*<sup>TM</sup> process can be judged to be successful as a remediation technology for the specific application at Paducah.**

If this criterion is not met after 1 - 2 years of *Lasagna*<sup>TM</sup> operation, it will be possible to extend the treatment time, without re-installing the electrodes and treatment zones. In order to also evaluate the data for use by other states, a destruction and removal efficiency (DRE) will also be calculated in several ways. The overall DRE will be calculated, and DRE's will be calculated at specific depths from the paired pre- and post-*Lasagna*<sup>TM</sup> soil concentrations. Efforts will be made to show whether TCE was destroyed or re-located at the site.

#### Phase IIa Field Experiment

The present QAPP is designed to evaluate data only from the Phase IIa field experiment. The anticipated length of operation of this phase is only between 3 and 6 months. This operation time will occur if the TCE concentrations measured in water and carbon samples are consistent with cleanup of the Phase IIa cube of soil. If Phase IIa is judged successful from the TCE soil concentration data, then the full Phase II installation will be completed and the whole site will be remediated for 1 - 2 years. In order for the Phase IIa to be considered successful for the Paducah site, the above decision rule will be applied to the post-experiment core samples 2A-04 and 2A-05, since during 3 - 6 months of operation, contaminated water will pass through two treatment zones before arriving at these two sampling points. The mean pre-test TCE concentration of these two points (shown in Appendix A) is approximately 34 ppm. (This mean was calculated by averaging soil concentration data between 5' or 6' and 40' bgs.) After 3 - 6 months, this mean should be reduced to below 5.6 ppm if the Phase IIa field experiment is a success. The Phase IIa field experiment will be considered a success by applying the following decision rule:

- **If the upper 95% confidence level of the mean of the soil sample population is less than 5.6 ppm for 2A-**

**04 and 2A-05 then the *Lasagna*<sup>TM</sup> process can be judged to be successful as a remediation technology for the specific application at Paducah.**

However, the post-experiment core samples 2A-01, 2A-02, and 2A-03 are located such that, TCE-contaminated water will pass either only one treatment zone or none at all. In addition, the average TCE concentration for these three points are 18.5, 19.3, and 52.3 ppm, respectively. (These means were calculated by averaging soil concentration data between 4' and 35' bgs for 2A-01, between 5' and 35' bgs for 2A-02, and between 4' and 39' bgs for 2A-03.) Therefore the above decision rule cannot be used for these latter 3 points. The increased distance between the sampling points and treatment zones is such that less than one pore volume water flowing past these three points in 3 - 6 months. Therefore a successful process may not result in the same mean concentrations for these 3 core samples as is anticipated for the other 2 cores. It will be likely that the average soil concentrations at the core sampling points 2A-01 and 2A-02 will not be reduced over the pre-test concentrations in the 3 - 6 month period. It is also anticipated that sampling point 2A-03 will be partially cleaned up. It is expected that the mean concentration of the post-*Lasagna*<sup>TM</sup> core samples at point 2A-03 will be reduced below 25 ppm. However, there is no way at this time to use this criterion for success of the Phase IIa experiment, especially if the means for points 2A-04 and 2A-05 are below the 5.6 ppm target. Therefore only the results from 2A-04 and 2A-05 will be used to indicate whether the full Phase II should be installed after the first 3 - 6 months of Phase IIa operation.

## Minimum Data Quality

The measurement data used in making the above decision must be of adequate quality to ensure that false positive and false negative decision errors are minimized. A false negative decision occurs when the sample data indicate that treatment below the action level has been achieved when actually it has not. A false positive decision is made when the sample data indicate that the process has been ineffective in treating TCE when it actually has been effective.

With the large number of soil samples being collected, the false positive/negative decision error rates due to sample size alone should be minimized and the focus of the quality control program should be on controlling the error of the analytical process. The following discussion establishes measurement objectives for analytical data.

### A. Precision

Precision will be assessed from the % Relative Standard Deviation (%RSD) from the analysis of replicate samples. The precision of the ORNL/GJ, CDMFPC, and

LMES-PGDP instrumental analyses will be determined from replicate analysis of standard solutions; the precision of the ORNL/GJ soil and carbon analyses and the LMES-PGDP water analyses will be determined from replicate soil, carbon, and water analyses. Similar precision will be determined by the Weston Laboratory which will be used by the Verification Entity, and by the laboratory used by the Kentucky Division of Waste Management. Typical %RSDs on the order of 10% are expected for instrumental analyses and <30% for replicate soil, carbon, and water analyses. In the event that the analytical laboratories cannot meet these expected %RSDs, corrective action should be taken before continuing these analyses, or the respective analytical results may not be used in assessing the success of the field experiment.

### B. Accuracy

Accuracy of data from ORNL/GJ, CDMFPC, and LMES-PGDP will be assessed from the comparison of measured values to "true" values of concentrations measured in reference standards purchased from Supelco, and from comparison of concentrations measured in soil, carbon, and water samples by Weston Laboratories, the Verification Entity's contract laboratory. Typical accuracies from the Phase I study were generally measured to be between 80 and 120% of the correct values for TCE concentrations in soil samples which were greater than 5 µg TCE/g soil.

### C. False Positives and Negatives

False Positives and Negatives will be assessed from the evaluation of data from the analysis of method blanks and samples known to contain the target analytes. (False Positives are the incorrect reporting of the presence of target compounds and False Negatives are the incorrect reporting of the absence of target compounds.) It is anticipated that there will be <10% False Positives as assessed from the analysis of "clean" soil, carbon, and water. It is also anticipated that there will be <10% False Negatives as assessed from the GC/EC analysis of hexane extracts of soil and carbon which are shown by the Weston Laboratory to contain greater than 5 ppm and from P&T analysis of water samples which are shown to contain greater than 20 ppbw (µg/L) target compounds.

## Data Quality Assessment

Section G entitled "Treatment Effectiveness Verification" discusses how the sampling data and analytical quality control indicators will be assessed to determine the acceptability of the actual error rate for decision concerning treatment effectiveness.

## F. Sampling and Analysis

*The results of analysis of a large of number of water, soil, and carbon samples will be used to evaluate the effectiveness of the Lasagna™ process. Standard statistical evaluation procedures will be used to determine the accuracy of the obtained data.*

Figure F-1 describes the estimated sample times and Table F-1 describes the samples which will be taken and analyzed on-site by ORNL/GJ, LMES-PGDP, or CDMFPC during the Phase IIa field study, and Table F-2 describes the samples which will be taken after the complete cleanup of the site (after Phase IIb), assuming that Phase IIa is successful.

The Kentucky Division of Waste Management may also split samples with the Phase IIa study.

### A. LMES Sampling and Analysis Plan

A copy of the LMES Sampling and Analysis Plan and associated QA Project Plan are included in Appendix B. Appendix C gives a short summary of this document. This plan has been reviewed by the RCI Verification Organization and determined to be adequate for meeting project Data Quality Objectives.

The role of the RCI is to verify the adequacy of the sampling and analysis being conducted by the LMES project team. Verification will be accomplished by field sampling audits of LMES and split analysis of selected LMES field samples. These activities are described in the following subsections:

#### Field Sampling Audits

Onsite field audits will be conducted for each major type of field sampling activity. At least one audit will be conducted for biweekly groundwater sampling, carbon cassette sampling, and soil sampling. The scope of these audits will include:

- (a) adherence to standard procedures and plans
- (b) representativeness of sampling
- (c) control of cross contamination
- (d) use of field quality control samples
- (e) field logkeeping
- (f) use of proper equipment, containers, and preservatives
- (g) equipment decontamination
- (h) precision and accuracy of field analytical methods
- (i) personnel training

Findings noted during audits will be immediately transmitted to project staff for corrective action.

Findings will also be documented in audit reports and analyzed for possible data quality impacts during the Data Quality Assessment phase of the RCI effort (see section H).

### B. Laboratory Analysis Verification

#### 1. Frequency of Split Sampling

At an approximate 10% frequency, LMES will split field samples for confirmation analysis at Weston Laboratories, an independent laboratory selected by the RCI Verification Organization.

Figures F-1 and F-2 describe the samples that will be collected by the LMES project team.. Table F-3 describes the split sampling activity that will be conducted. The Kentucky Division of Waste Management may also split samples with the Phase IIa study.

#### 2. Sampling and Analytical Procedures

Split samples will be collected after the field processing steps required to prepare the sample for laboratory analysis. These processing steps are described in the SOPs listed in Table Appendix C-3. This will minimize the variability introduced by field sampling and processing techniques and will allow the RCI team to focus on laboratory analytical acceptability.

The GC/EC analytical procedures used by Weston Laboratories, the independent laboratory, will be as identical as possible to those used by the LMES project team. Weston's purge-and-trap procedure which will use a Hall Detector (HD) as described in SW846 8010. A description of the analytical methods for each type of sample are included below:

#### Performance Evaluation Samples

Weston Laboratory, the verification analytical laboratory, will be required to demonstrate it's qualification BEFORE starting actual field sample analyses by successfully analyzing a liquid standard containing known quantities of TCE, DCE, and vinyl chloride. Although the concentrations in this standard will be known to the RCI, it will be blind to the Weston and other laboratories.

Analysis will include 5 replicate analyses by purge-and-trap and 5 replicate analysis by hexane dilution and direct injection GC/EC. The latest update of SW-846 will be used. Detection limits required will be 20 ppbw

( $\mu\text{g/L}$ ) in water for purge-and-trap and 0.01 ppmw ( $\mu\text{g/g}$ ) in the soil hexane extract, for GC/EC.

Monitoring Well Samples

Sixteen split samples will be collected for analysis by purge-and-trap using the latest final update of SW-846 methods. Four samples will be collected prior to project initiation and two per week will be collected over 6 bi-weekly periods. Sample analysis will include TCE, DCE, and vinyl chloride by purge-and-trap using the latest version of SW-846 methodology. Sample detection limits will be 20 ppbw ( $\mu\text{g/L}$ ) in water for the purge-and-trap analysis.

Soil Cores

Eighteen soil samples which will be taken at the depths shown in Appendix A to contain the highest concentration of TCE, will be split and analyzed by ORNL/GJ, LMES-PGDP, and Weston Laboratories. These laboratories will use hexane extraction GC/EC analysis for TCE, and water extraction followed by purge-and-trap analysis for VC, DCE, and TCE. The latest version of SW-846 methodology will be used. Required sample detection limits will be 20 ppbw ( $\mu\text{g/L}$ )

in water for all analytes in the water extracts and 0.01 ppm for TCE in the hexane extracts for GC/EC analyses.

Carbon Cassettes

Ten carbon cassette samples collected during two sampling events will be analyzed for TCE using hexane dilution and direct injection GC/EC analysis. Required detection limits will be 0.01 ppm for TCE in the hexane extract.

Field Blanks

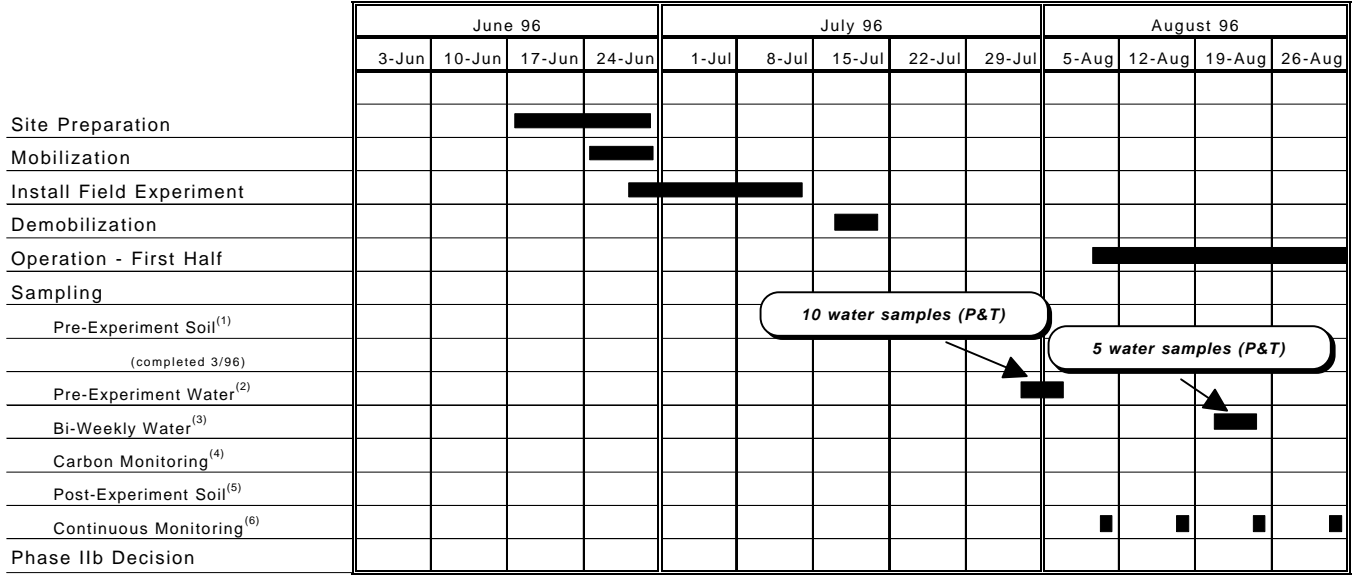
Five hexane field blanks will be collected and analyzed by GC/EC for TCE. Five deionized water blanks will also be collected and analyzed for TCE, DCE, and Vinyl Chloride by purge-and-trap. These blanks will function as trip blanks for confirming that target compounds are not introduced during sample shipment and laboratory storage prior to analysis.

**3. Data Validation**

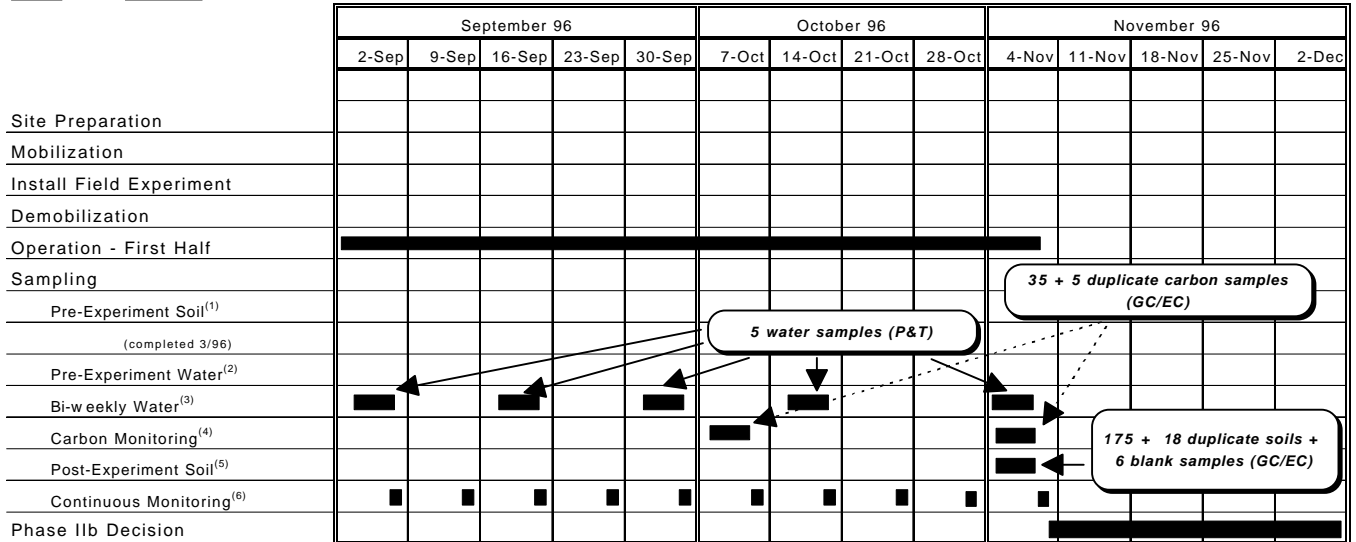
Laboratory Data Consultants will be retained to perform normal validation of all analyses performed by the verification laboratory. This organization has no known conflict of interest in the success of the Lasagna™ process.

**Figure F-1. Sampling and Analysis Schedule for the Phase IIa Study.**

**Date:** 7/31/96



**Date:** 7/31/96



- Notes:
- (1) Over 150 soil samples were taken in March by ORNL-GJ at 1-ft. intervals.
  - (2) 10 Water samples will be taken for analysis by LMES-PGDP or CDMFPC laboratories.
  - (3) 5 Water samples will be taken twice each month for analysis by LMES-PGDP or CDMFPC laboratories.
  - (4) Over 80 carbon samples (2 and 3 months after beginning study) will be analyzed for TCE by ORNL-GJ.
  - (5) Over 150 soil samples will be analyzed for TCE by ORNL-GJ.
  - (6) *In-Situ* monitor developed and supported by Monsanto will be used weekly. (5 probes)

Table F-1. Phase IIa Sampling and Analysis Activities

Timeframe	Type of Activity	Number	Type of Analyses/Analytes	Comments
March 1996 (complete)	Soil Borings	5 borings, 175 samples + 18 duplicates + 6 blanks	TCE using hexane extraction - <u>GC/EC</u> TCE, DCE, VCM using water extraction - <u>purge-and-trap</u>	Total of ~181 samples taken at 1' intervals between 5' - 40' Total of ~18 samples taken at 1' intervals between 5' - 40'
June 1996	Water Samples	5 wells, 10 samples	TCE, DCE, VCM - <u>purge-and-trap</u>	Total of 5 samples taken at the ~20'-depth
"	Install TCE membrane monitoring device	6 locations, 30-60 samples	TCE, DCE, VCM - <u>GC/FID</u>	Total of 6 membrane devices installed to a depth of 20'.
3-, 5-month monitoring	Carbon Cassette Analysis	4 locations, 80 samples + 10 duplicates	TCE using hexane extraction - <u>GC/EC</u>	Total of 160 samples taken at 1' intervals (80 samples for each month)
"	Blank Carbon Analyses	1 set, 10 samples	"	10 samples
irregular monitoring	Water Samples	5 wells, 35 samples	TCE, DCE, VCM - <u>purge-and-trap</u>	Total of 15 samples (5 samples for each month)
6-month monitoring	Soil Borings	5 borings, 175 samples + 18 duplicates + 6 blanks	TCE using hexane extraction - <u>GC/EC</u> TCE, DCE, VCM using water extraction - <u>purge-and-trap</u>	Total of ~181 samples taken at 1' intervals between 5' - 40' Total of ~18 samples taken at 1' intervals between 5' - 40'
			<b>Total of 372 soil samples and 170 carbon samples using hexane extraction, and 40 samples using purge-and-trap</b>	

Table F-2. Phase IIb Sampling and Analysis Activities

Timeframe	Type of Activity	Number	Type of Analyses/Analytes	Comments
February 2000	Soil Borings	9 borings	TCE using hexane extraction - <u>GC/EC</u> TCE, DCE, VCM using water extraction - <u>purge-and-trap</u>	Total of <u>63</u> samples taken at 5' intervals between 5' - 40' Total of <u>~10</u> samples taken at 5' intervals between 5' - 40'
			<b>Total of 63 samples using hexane extraction + 10 purge-and-trap</b>	



**Table F-3. Summary of Split Samples to be Sent to Verification Entity's Contract Laboratory and Kentucky Division of Waste Management.**

Week of .....	# Samples	# Replicates/Sample	Sample Type ( <u>Method</u> )
8 August	4	1	Pre-Experiment Water Samples ( <u>P&amp;T</u> )
	1	5	Certified Standard ( <u>GC/EC</u> )
	1	5	Certified Standard ( <u>P&amp;T</u> )
27 August	2	1	1 <sup>st</sup> Bi-weekly water ( <u>P&amp;T</u> )
10 September	2	1	2 <sup>nd</sup> Bi-weekly water ( <u>P&amp;T</u> )
24 September	2	1	3 <sup>rd</sup> Bi-weekly water ( <u>P&amp;T</u> )
30 October	2	1	4 <sup>th</sup> water ( <u>P&amp;T</u> )
14 November	2	1	5 <sup>th</sup> water ( <u>P&amp;T</u> )
14 December	2	1	6 <sup>th</sup> water ( <u>P&amp;T</u> )
14 January	2	1	7 <sup>th</sup> Bi-weekly water ( <u>P&amp;T</u> )
14 November	5	1	Carbon Cassette Samples ( <u>GC/EC</u> )
	5	1	QA/QC ( <u>GC/EC</u> )
14 January	5	1	Carbon Cassette Samples ( <u>GC/EC</u> )
14 February	18	1	Soil Cores ( <u>GC/EC</u> )
	18	1	Soil Cores ( <u>P&amp;T</u> )
	1	5	Blank Hexane ( <u>GC/EC</u> )
<b>Totals:</b>	<b>72 Samples</b>		
	<b>84 Determinations</b>		
<b>Total Cost:</b>	<b>\$ 10,858</b>		
<b>Data Validation Cost:</b>	<b>\$ 3,200</b>		
<b>Total Validation/Analytical Cost:</b>	<b>\$ 14,058</b>		

## G. Treatment Effectiveness Verification

*The results of analysis of a large of number of water, soil, and carbon samples will be used to evaluate the effectiveness of the Lasagna™ process. Standard statistical evaluation procedures will be used to determine the accuracy of the obtained data.*

### Field Sampling Audits

The results of field sampling activities will be assessed to determine if any data quality impacts occurred. A discussion of these impacts, if any, will be presented in the Treatment Effectiveness Verification Report.

### Procedures to Assess Precision, Accuracy, and Completeness

#### A. Precision

The precision of the data reported by the ORNL/GJ and LMES-PGDP laboratories will be estimated from the results of analysis of replicate samples which contain analytes above the level of detection. %RSDs for each replicate will be calculated. The %RSD values thus obtained will be used in the subsequent interpretation of analytical precision. Target %RSDs for the analysis of target compounds above 5 ppm in soil and carbon and above 100 ppb in water will be 30%. If the %RSDs are larger than this amount, this data may not be included in the overall assessment of treatment effectiveness.

#### B. Accuracy

The accuracy of the data reported by the analytical laboratories will be estimated from the analysis of certified standards containing the target compounds in methanol. In addition, samples will be split with R. F. Weston Laboratory which was designated by the Verification Entity. Assuming the contract laboratory reports valid concentrations for the concentrations of the target compounds in the certified standard, the contract laboratory's results will be considered as the correct values. Weston's values will be compared to the results obtained by ORNL/GJ and LMES-PGDP or CDMFPC laboratories to arrive at an estimate of accuracy.

#### C. Detailed Description of How Results will be Calculated

All samples will be submitted to the analytical laboratories in a blind manner.

##### 1. Certified Standard Solutions

Five replicates of the certified standard and a solvent method blank will be analyzed. The analytical laboratories will be asked to report

concentrations in units of  $\mu\text{g}$  target compounds/mL methanol. They will also be asked to use the same procedures for the dilution of the certified standard, that would be used for dilution of the hexane extract for GC/EC analyses, or for the water extract for the purge-and-trap analysis.

##### 2. Determination of Accuracy

The mean of five determinations of target compound concentrations in the certified standards will be used to calculate the % Accuracy. The following equations will be used for TCE accuracy. Similar equations will also be used for *cis*-1,2-DCE and VC:

$$\% \text{Accuracy} = 100 \times (\text{Measured Concen}) / (\text{Correct Concen})$$

**where:** Measured Concen = (TCE concentration reported)

Correct Concen = Concentration in certified standard

##### 3. False Positives and Negatives

The analysis of reagent grade hexane and "clean" soil and water as method blanks will show whether there is a significant source of target compounds in the analytical laboratories' test procedures which would result in the incorrect reporting of the presence of target compounds (*False Positives*). The analysis of spiked samples will be used to determine whether there are significant extraction problems associated with the sample matrix which would result in the incorrect reporting of the absence of PCBs (*False Negatives*). Each laboratory will use standard procedures to provide matrix spike recoveries of TCE, *cis*-1,2-DCE, and VC.

##### 4. Correction of Data for Extraction Efficiencies

No soil or water data will be corrected for extraction efficiency. It will be assumed that 100% of target compounds which may be present in soil and water will be quantitatively analyzed using these techniques. **An extraction efficiency of 33.3%** for extraction of TCE from carbon using hexane will be used. See Reference 13 for further information.

### 5. Completeness (Sampling and Analysis)

The ratio of samples taken to those described in the work plan shown in Appendix B, will be used as a measure of sampling completeness. (It is anticipated that no samples will be obtained at locations if drilling obstructions prevent reaching the planned 40' {12.2 meter} depths.) The percent of samples submitted for analysis for which valid concentrations of target compounds are reported, will be used as a measure of analytical completeness. (If concentrations of target compounds are reported as None Detected with Levels of Detection (LOD) greater than 5.0 ppm, these results will be considered as not reported.)

### 6. Comparison of Split Samples

Split sample data will be compared to determine if the results are significantly different at a 80 % confidence level. Individual soil sample results from split samples will also be compared to determine if they yield identical conclusions regarding exceeding the 5 ppm limit.

### **D. Data Quality Assessment**

All variables that could potentially impact the reliability of the data in adequately representing the *Lasagna*<sup>TM</sup> site including field sampling error, laboratory error, and statistical sampling plan design will be integrally assessed to determine if the aggregate data set is suitable for treatment effectiveness verification.

### **E. Treatment Effectiveness Verification Report**

Mr. David Carden, the RCI Verification Entity will issue a report that describes the ability of the data collected to meet project Data Quality Objectives. This will be accomplished by discussing the implications of field sampling audit results, comparison of project quality control data with defined limits, and comparison of split sample data with project data. All split sample data will be obtained from a fixed, pre-defined set of samples. Table G-1 describes each set of split samples which will be analyzed by each analyzing organization. Note that Weston Laboratories will analyze representative water, hexane, and soil samples which will also be analyzed by the CDMFPC, ORNL/GJ, and LMES-PGDP laboratories.

#### GC/EC Analyses

All GC/EC analyses will be performed on aliquots of hexane extracts which will be shipped to Weston, and therefore these analyses will be for TCE in exactly the same extracts that will be analyzed in the field by ORNL/GJ.

#### P&T Analyses

All Weston P&T analyses of groundwater will be performed on aliquots of the same water samples which will also be analyzed by CDMFPC. Weston will use P&T GC/HD and CDMFPC will use P&T GC/ELCD. All Weston P&T analyses of soil will be performed on water extracts of soil sample aliquots that will be analyzed by LMES-PGDP using P&T GC/MS methods. Weston will use a P&T GC/HD method.

#### Verification Report

Once the limitations of the data set are known, the verification report will describe the capability of the data to make a decision on treatment effectiveness.

Table G-1. Summary of Split Samples and Extracts to be Analyzed by Various Laboratories.

Week of .....	# Samples (# Replicates/Sample)	Sample type (method)	Analyzing Laboratory
5 August	4 (1) 1 (5) 1 (5)	Pre-experiment water (P&T) Certified Std. in methanol (P&T) Certified Std. in methanol (GC/EC)	Weston and CDMFPC “ “
19 August	2 (1)	1 <sup>st</sup> Bi-weekly water (P&T)	“
2 September	2 (1)	2 <sup>nd</sup> Bi-weekly water (P&T)	“
16 September	2 (1)	3 <sup>rd</sup> Bi-weekly water (P&T)	“
30 September	2 (1)	4 <sup>th</sup> Bi-weekly water (P&T)	“
14 October	2 (1)	5 <sup>th</sup> Bi-weekly water (P&T)	“
4 November	2 (1)	6 <sup>th</sup> Bi-weekly water (P&T)	“
7 October	5 (1)  5 (1)	Hexane from Carbon (GC/EC)  Certified Std. in Methanol (GC/EC)	Weston and ORNL/GJ  “
4 November	5 (1) 18 (1) 18 (1) 1 (5)	Hexane from Carbon (GC/EC) Hexane from Soil Cores (GC/EC) Water from Soil Cores (P&T)  Blank Hexane (GC/EC)	“ “ Weston and LMES-PGDP  Weston and ORNL/GJ
<b>Totals:</b>	<b>70 Samples</b>  <b>82 Determinations</b>		
<b>Total Cost:</b>	<b>\$ 10,858</b>		
<b>Data Validation Cost (Estimated):</b>	<b><u>\$ 3,200</u></b>		
<b>Total Validation/Analytical Cost:</b>	<b>\$ 14,058</b>		

## Appendix D. Tabular Data Plotted in the Figures of Sections F and G

The present appendix gives the tabular data which is shown graphically in Figures F-1 and F-2, , G-1a through G-1c, G-5a and G-5b and G-6 through G-8. All other figures in Section F shows the tabular data as a part of the display.

Table Appendix D-1. Table for Data Shown in Figure G-6

Sample ID	Location	Sample Type	Soil Wt (g)	Water (ml)	Extract (ug/l)	ORNL/GJ GC/EC	LMUS GC/MS	RPD(%)
04P-19	19	soil	6.8	35	1,700	8,750	10,658	20%
04P-24	24		5.5	35	1,600	10,182	12,236	18%
04P-35	35		7.7	35	940	4,273	6,008	34%
05P-19	19		6.7	35	22,000	114,925	81,757	34%
05P-23	23		7	35	20,000	100,000	89,898	11%
05P-28	28		1.8	35	1,300	25,278	14,886	52%
05P-34	34		8	35	510	2,231	1,994	11%
03P-15	15		7.6	35	5	23	21	7%
03P-27	27		6.9	35	92	467	344	30%
02P-11	11	(also detected cis at 32 ug/L)	5	35	32	224	245	9%
02AP-11	11	(also detected cis at 7 ug/L)	8.2	35	0	0	0	
02AP-31	31		9.2	35	340	1,293	2,462	62%
02AP-39	39		7	35	160	800	996	22%

Table Appendix D-2. Table for Data Shown in Figure G-7

Sample ID	TCE Soil Conc. (ppm)		% RPD
	ORNL/GJ	Weston	
2015-PT	1.07	1.60	39.33%
2020-PT	0.41	0.34	19.00%
2030-PT	0.00	<.1	-
2037-PT	0.00	<.1	-
2050-PT	0.00	<.1	-
2063-PT	7.86	6.55	18.22%
2079-PT	0.00	<.1	-
2098-PT	39.96	48.11	18.52%
2107-PT	0.34	0.49	37.98%
2120-PT	2.56	2.41	6.18%
2141-PT	36.10	21.85	49.19%
2158-PT	0.23	0.16	37.09%
2181-PT	7.28	1.05	149.55%
2212-PT	0.49	0.62	23.05%
2228-PT	25.59	27.90	8.64%
2244-PT	7.66	5.41	34.39%
2258-PT	41.54	42.30	1.82%
2272-PT	0.00	0.22	200.00%
2294-PT	74.64	62.48	17.74%
2299-PT	118.63	120.27	1.38%

*Table Appendix D-3. Table for Data Shown in Figure G-1a*

Sample ID	Depth (ft.)	Concentration (ppm)		RPD (%)
		Analysis #1	Analysis #2	
LA01-32	32	21.30	11.20	62.1%
LA01-33	33	16.50	25.02	41.0%
LA02-31	31	43.28	18.84	78.7%
LA02-32	32	5.07	12.02	81.4%
LA03-20	20	493.90	617.09	22.2%
LA03-39	39	6.01	5.30	12.5%
LA04-24	24	22.06	25.70	15.3%
LA04-38	38	0.00	0.61	-
LA05-40	40	0.61	0.58	4.8%

*Table Appendix D-4. Table for Data Shown in Figure G-1b*

Soil ID	Depth (ft.)	Concentration (ppm)		RPD(%)
		Analysis #1	Analysis #2	
02P-8	8	0.42	0.35	18.4%
02AP-8	8	0.01	0.03	105.9%
02AP-26	26	128.99	142.09	9.7%
02AP-35	35	4.94	4.06	19.5%
03P-11	11	0.09	0.06	40.6%
03P-24	24	25.04	21.07	17.2%
04P-11	11	0.19	0.17	14.4%
04P-24	24	7.93	2.96	91.2%
04P-35	35	6.95	4.80	36.4%
05P-11	11	0.59	0.78	26.7%
05P-21	21	40.58	38.24	5.9%
05P-25	27	14.22	16.54	15.1%
05P-31	31.5	17.96	8.04	76.4%

*Table Appendix D-5. Table for Data Shown in Figure G-1c*

Soil ID	Depth (ft.)	Concentration (ppm)		RPD(%)
		Analysis #1	Analysis #2	
01FS-15	15	0.00	0.00	-
01FS-31	31	1.70	0.43	119.9%
01FS-35	35	0.05	0.06	7.6%
02F3-12	12	0.43	0.47	8.0%
02F3-24	24	77.62	69.88	10.5%
02F3-35	35	10.91	12.33	12.2%
03FS-15	15	0.00	0.00	-
03FS-19	19	1.24	0.90	31.8%
03FS-31	31	0.00	0.00	-
03FS-35	35	0.01	0.01	17.5%
04FS-11	11	0.02	0.02	11.3%
04FS-22	22	5.30	7.81	38.2%
04AFS-11	11	0.02	0.01	68.5%
04AFS-27	27	38.08	41.75	9.2%
04AFS-35	35	14.30	12.43	14.0%
05FS-11	11	0.19	0.27	37.6%
05FS-19	19	12.39	15.31	21.1%
05FS-36	36	4.58	3.36	30.7%

Sample ID	Sampling Date	Concentration (ppm)		RPD (%)
		Analysis #1	Analysis #2	
C-05-11	11-Mar-97	58	60	3.51%
C-05-22	"	3,963	4,156	4.75%
C-04-11	"	2,126	2,096	1.42%
C-04-24	"	4,842	4,841	0.01%
C-01-12	"	11	11	0.00%
C-01-24	"	5,462	5,239	4.16%
C-05-12	24-Feb-97	417	406	2.50%
C-05-23	"	1,922	1,906	0.86%
C-04-22	"	6,410	6,452	0.65%
C-01-09	"	4,079	3,659	10.85%
C-01-21	"	9,861	10,323	4.58%
C-05-18	9-Jan-97	3,619	6,541	57.51%
C-04-19	"	12,651	11,956	5.65%
C-04-20	"	12,141	12,636	4.00%
C-01-20	"	63,564	63,970	0.64%
C-01-21	"	63,424	53,663	16.67%
C-05-01	13-Dec-96	10	24	83.17%
C-02-19	16-Dec-96	75,298	68,003	10.18%
C-02-20	"	68,003	56,019	19.33%
C-05-10	13-Nov-96	92	111	18.65%
C-05-18	"	21,950	21,143	3.74%
C-04-10	"	30	39	24.94%
C-04-17	"	4,439	6,518	37.94%
C-04-18	"	35,601	35,635	0.10%
C-02-18	"	22,683	21,110	7.18%
C-02-19	"	22,098	24,165	8.94%
C-01-16	"	32,323	31,276	3.29%
C-01-17	"	27,611	30,055	8.48%

Sample ID	Depth (ft.)	Conc. (ppm)		RPD(%)
		ORNL/GJ	Weston	
04P-11	11	0.17	0.16	4.3%
04P-24	24	2.96	2.30	25.2%
04P-35	35	4.80	3.80	23.3%
05P-11	11	0.78	0.65	17.7%
05P-21	21	38.24	29.00	27.5%
05P-27	27	16.54	9.00	59.0%
05P-31	31	8.04	7.00	13.8%
03P-11	11	0.06	0.07	15.5%
03P-24	24	21.07	22.00	4.3%
02P-8	8	0.35	0.39	12.2%
02PA-8	8	0.03	0.04	32.3%
02PA-26	26	142.09	130.00	8.9%
02PA-35	35	4.06	4.70	14.7%

*Table Appendix D-7. Table for Data Shown in Figure G-5b*

Sample ID	Depth (ft.)	Conc. (ppm)		RPD(%)
		ORNL/GJ	Weston	
01FS-15	15	0.00	0.00	-
01FS-31	31	0.43	1.28	100.40%
01FS-35	35	0.06	0.06	1.94%
02FS-12	12	0.47	0.43	7.86%
02FS-24	24	69.88	60.25	14.80%
02FS-35	35	12.33	10.25	18.40%
03FS-15	15	0.00	0.00	-
03FS-19	19	0.90	0.86	4.44%
03FS-31	31	0.00	0.00	-
03FS-35	35	0.01	0.01	1.98%
04FS-11	11	0.02	0.03	38.30%
04FS-22	22	7.81	7.41	5.25%
04AFS-11	11	0.01	0.02	70.97%
04AFS-27	27	NA	NA	NA
04AFS-35	35	12.43	12.54	0.8%
05FS-11	11	0.27	0.19	36.3%
05FS-19	19	15.31	12.94	16.8%
05FS-36	36	3.36	3.09	8.3%
10FS-11	11	1.61	1.56	3.0%
10FS-23	23	64.87	55.33	15.9%
11FS-12	12	35.24	31.03	12.7%
11FS-24	24	118.63	103.93	13.2%



*Table Appendix D-8. Table for Data Shown in Figure G-8*

<b>Sample No.</b>	<b>ORNL/GJ</b>	<b>Weston</b>	<b>RPD (%)</b>
LCA-017	116,965	113,415	3
LCA-018	115,045	130,106	12
LCA-037	81,175	75,610	7
LCA-038	84,992	109,843	26
LCA-056	16,395	17,900	9
LCD-008	814	54	175
LCD-018	430	790	59
LCD-028	837	54	176
LCD-038	6,010	4,900	20
LCE-016	231	260	12
LCE-032	35	3	167
LCE-037	402	29	173
LCE-058	388	650	50
LCF-004	85	96	12
LCF-099	77	96	22
LCF-026	2	2	11
LCF-031	4	7	47
LCF-048	387	336	14
LCF-056	51	41	21
LCF-062	27,043	22,314	19
LCG-025	6	9	44
LCG-030	2	3	9
LCG-048	684	645	6
LCG-057	4,022	3,259	21
LCG-062	56,652	38,571	38

Table Appendix D-9. Table for Data Shown in Figures F-3 and F-4 taken 8/8/96 - 1/10/97

Well ID	Date	Vial ID	Measured Concentrations (ppb)		
			TCE-CDM	TCE-Weston	TCE-LMUS
PZ-2A-01	8/8/96	PZ-2A-01(8/8)	168,085	150,000	240,000
PZ-2A-02	8/8/96	PZ-2A-02(8/8)	40,472	NA	NA
PZ-2A-03	8/8/96	PZ-2A-03-A1(8/8)	57,240	NA	NA
PZ-2A-04	8/8/96	PZ-2A-04-A1(8/8)	260,000	340,000	NA
PZ-2A-05	8/8/96	PZ-2A-05-A1(8/8)	460,000	NA	700,000
PZ-2A-01	8/12/96	PZ-2A-01(8/12)	90,113	300,000	NA
PZ-2A-02	8/12/96	PZ-2A-02(8/12)	335,175	NA	NA
PZ-2A-03	8/12/96	PZ-2A-03-A1(8/12)	68,456	NA	NA
PZ-2A-04	8/12/96	PZ-2A-04-A1(8/12)	185,224	430,000	NA
PZ-2A-05	8/12/96	PZ-2A-05-A1(8/12)	102,108	NA	NA
PZ-2A-01	8/27/96	PZ-2A-01(8/27)	28,000	420,000	NA
PZ-2A-02	8/27/96	PZ-2A-02(8/27)	790,000	NA	NA
PZ-2A-03	8/27/96	PZ-2A-03-A1(8/27)	300,000	NA	NA
PZ-2A-04	8/27/96	PZ-2A-04-A1(8/27)	480,000	410,000	NA
PZ-2A-05	8/27/96	PZ-2A-05-A1(8/27)	770,000	NA	NA
MW-160(8/27/96)	8/27/96	MW-160(8/27/96)	60,000	36,000	NA
PZ-2A-01	9/10/96	PZ-2A-01-A1(9/10)	48,000	NA	NA
PZ-2A-01	9/10/96	PZ-2A-01-A2(9/10)	49,000	NA	NA
PZ-2A-02	9/10/96	PZ-2A-02-A1(9/10)	130,000	NA	NA
PZ-2A-02	9/10/96	PZ-2A-02-A2(9/10)	NA	NA	NA
PZ-2A-02	9/10/96	PZ-2A-02-B1(9/10)	NA	NA	NA
PZ-2A-03	9/10/96	PZ-2A-03-A1(9/10)	190,000	NA	260,000
PZ-2A-03	9/10/96	PZ-2A-03-A2(9/10)	180,000	NA	230,000
PZ-2A-03	9/10/96	PZ-2A-03-B1(9/10)	NA	NA	260,000
PZ-2A-03	9/10/96	PZ-2A-03-B2(9/10)	NA	NA	220,000
PZ-2A-04	9/10/96	PZ-2A-04-A1(9/10)	ND	400,000	570,000
PZ-2A-04	9/10/96	PZ-2A-04-A2(9/10)	510,000	NA	570,000
PZ-2A-04	9/10/96	PZ-2A-04-B1(9/10)	NA	NA	580,000
PZ-2A-04	9/10/96	PZ-2A-04-B2(9/10)	710,000	NA	570,000
PZ-2A-05	9/10/96	PZ-2A-05-A1(9/10)	410,000	260,000	410,000
PZ-2A-05	9/10/96	PZ-2A-05-A2(9/10)	380,000	NA	370,000
PZ-2A-05	9/10/96	PZ-2A-05-B1(9/10)	460,000	NA	370,000
PZ-2A-05	9/10/96	PZ-2A-05-B2(9/10)	390,000	NA	370,000
PZ-2A-01	9/24/96	PZ-2A-01-A1(9/24)	91,000	NA	NA
PZ-2A-02	9/24/96	PZ-2A-02(9/24)	110,000	NA	NA
PZ-2A-03	9/24/96	PZ-2A-03(9/24)	110,000	110,000	NA
PZ-2A-04	9/24/96	PZ-2A-04-A1(9/24)	470,000	550,000	530,000
PZ-2A-05	9/24/96	PZ-2A-05(9/24)	180,000	NA	NA
PZ-2A-02	11/13/96	PZ-2A-02(11/13)	50,000	NA	NA
PZ-2A-03	11/13/96	PZ-2A-03-A1(11/13)	130,000	NA	NA
PZ-2A-03	11/13/96	PZ-2A-03-A2(11/13)	83,000	NA	NA
PZ-2A-04	11/13/96	PZ-2A-04(11/13)	200,000	260,000	310,000
PZ-2A-05	11/13/96	PZ-2A-05(11/13)	530,000	640,000	NA
PZ-2A-02	12/12/96	PZ-2A-02(12/12)	410,000	NA	NA
PZ-2A-03	12/12/96	PZ-2A-03(12/12)	59,000	NA	78,000
PZ-2A-04	12/12/96	PZ-2A-04(12/12)	220,000	310,000	390,000
PZ-2A-05	12/12/96	PZ-2A-05(12/12)	490,000	470,000	850,000
PZ-2A-03	1/10/97	PZ-2A-03(1/10)	91,000	NA	NA
PZ-2A-04	1/10/97	PZ-2A-04(1/10)	160,000	170,000	200,000
PZ-2A-05	1/10/97	PZ-2A-05(1/10)	710,000	730,000	NA

Table Appendix D-10. Table for Data Shown in Figures F-3 and F-4 taken 2/12/97 - 8/6/97

Well ID	Date	Vial ID	Measured Concentrations (ppb)		
			TCE-CDM	TCE-Weston	TCE-LMUS
PZ-2A-03	2/12/97	PZ-2A-03(2/12)	33,000	NA	13,000
PZ-2A-05	2/12/97	PZ-2A-05(2/12)	950,000	550,000	750,000
PZ-2A-02	2/12/97	PZ-2A-02(2/12)	710,000	NA	730,000
PZ-2A-04	2/12/97	PZ-2A-04(2/12)	140,000	5,800	140,000
PZ-2A-02	3/7/97	PZ-2A-02(3/07)	160,000	NA	NA
PZ-2A-03	3/7/97	PZ-2A-03(3/07)	35,000	NA	NA
PZ-2A-04	3/7/97	PZ-2A-04(3/07)	120,000	NA	NA
PZ-2A-04	3/7/97	PZ-2A-04(3/07)	150,000	NA	NA
PZ-2A-05	3/7/97	PZ-2A-05(3/07)	590,000	NA	NA
PZ-2A-03	4/30/97	PZ-2A-03(4/30)	22,000	39,000	NA
PZ-2A-04	4/30/97	PZ-2A-04(4/30)	42,000	80,000	NA
PZ-2A-05	4/30/97	PZ-2A-05(4/30)	101,000	NA	NA
Cathode SE	6/6/97	Cathode SE(6/6)	10,000	2,500	1,400
MW-160	6/6/97	MW-160(6/6)	83,000	61,000	50,000
PZ-2A-03	6/6/97	PZ-2A-03(6/6)	94,000	45,000	39,000
PZ-2A-04	6/6/97	PZ-2A-04(6/6)	160,000	81,000	65,000
PZ-2A-05	6/6/97	PZ-2A-05(6/6)	700,000	490,000	360,000
Cathode SE	7/9/97	Cathode SE(7/9)	16,000	1,700	2,300
MW-160	7/9/97	MW-160(7/9)	90,000	51,000	61,000
PZ-2A-03	7/9/97	PZ-2A-03(7/9)	94,000	33,000	47,000
PZ-2A-04	7/9/97	PZ-2A-04(7/9)	109,000	63,000	76,000
PZ-2A-05	7/9/97	PZ-2A-05(7/9)	15,000	230,000	280,000
Cathode SE	8/6/97	Cathode SE(8/6)	10,000	230	850
MW-160	8/6/97	MW-160(8/6)	31,000	42,000	53,000
PZ-2A-03	8/6/97	PZ-2A-03-A1(8/6)	30,000	26,000	36,000
PZ-2A-03	8/6/97	PZ-2A-03-A2(8/6)	30,000	NA	NA
PZ-2A-04	8/6/97	PZ-2A-04(8/6)	44,000	32,000	55,000
PZ-2A-05	8/6/97	PZ-2A-05(8/6)	210,000	170,000	160,000

**Table Appendix D-11. Calibration Checks for March 1997 Soil Samples**

<b>Sample Type</b>	<b>Analysis Date</b>	<b>TCE Conc. Initial Cal (ug/l)</b>	<b>TCE Conc. Cal Check (ug/l)</b>	<b>% Difference</b>
100 ppb TCE	6-Mar-97	106.5	110.6	3.85
100 ppb TCE	6-Mar-97	106.5	113.2	6.29
100 ppb TCE	6-Mar-97	106.5	112.1	5.26
100 ppb TCE	6-Mar-97	106.5	113.1	6.20
100 ppb TCE	6-Mar-97	106.5	113.8	6.85
100 ppb TCE	6-Mar-97	106.5	113.6	6.67
100 ppb TCE	6-Mar-97	106.5	114.7	7.70
100 ppb TCE	6-Mar-97	106.5	114.6	7.61
250 ppb TCE	7-Mar-97	267.6	277.6	3.74
100 ppb TCE	7-Mar-97	106.5	113.7	6.76
100 ppb TCE	7-Mar-97	106.5	114.1	7.14
100 ppb TCE	7-Mar-97	106.5	115.4	8.36
100 ppb TCE	7-Mar-97	106.5	117	9.86
250 ppb TCE	7-Mar-97	267.6	278.1	3.92
100 ppb TCE	7-Mar-97	106.5	116.7	9.58
100 ppb TCE	7-Mar-97	106.5	117.3	10.14
100 ppb TCE	7-Mar-97	106.5	116.9	9.77
100 ppb TCE	7-Mar-97	106.5	116.9	9.77
100 ppb TCE	7-Mar-97	106.5	116.3	9.20
250 ppb TCE	7-Mar-97	267.6	289.4	8.15
100 ppb TCE	7-Mar-97	106.5	117.2	10.05
750 ppb TCE	8-Mar-97	754.4	817.6	8.38
100 ppb TCE	8-Mar-97	106.5	118	10.80
100 ppb TCE	8-Mar-97	106.5	117.5	10.33
100 ppb TCE	8-Mar-97	106.5	118	10.80
100 ppb TCE	8-Mar-97	106.5	117.3	10.14
100 ppb TCE	8-Mar-97	106.5	119.4	12.11
100 ppb TCE	8-Mar-97	106.5	118.8	11.55
100 ppb TCE	8-Mar-97	106.5	119.1	11.83
250 ppb TCE	8-Mar-97	267.6	292	9.12
250 ppb TCE	8-Mar-97	267.6	291	8.74
250 ppb TCE	8-Mar-97	267.6	295	10.24
250 ppb TCE	8-Mar-97	267.6	294.7	10.13
250 ppb TCE	9-Mar-97	267.6	291.4	8.89
100 ppb TCE	9-Mar-97	106.5	115.5	8.45
250 ppb TCE	9-Mar-97	267.6	278.6	4.11
250 ppb TCE	9-Mar-97	267.6	279	4.26
250 ppb TCE	9-Mar-97	267.6	278.1	3.92
100 ppb TCE	9-Mar-97	106.5	114.2	7.23
100 ppb TCE	9-Mar-97	106.5	110	3.29

**Table Appendix D-12. Laboratory Duplicates for March 1997 Soil Samples Shown in Figure G-11**

Sample ID	Location	Sample Type	Analysis Date	Sample Extract TCE, ug/l	Duplicate Extract TCE, ug/l	% Difference
508-LD	L2A-04P-13	Lab Duplicate	6-Mar-97	41	41	-0.25
517-LD	24	Lab Duplicate	6-Mar-97	7929	7847	-1.03
525-LD	32	Lab Duplicate	6-Mar-97	13912	14075	1.17
533-LD	40	Lab Duplicate	6-Mar-97	537	561	4.47
543-LD	L2A-05P-13	Lab Duplicate	7-Mar-97	248	247	-0.40
552-LD	21	Lab Duplicate	7-Mar-97	40578	39422	-2.85
559-LD	28	Lab Duplicate	7-Mar-97	13695	13466	-1.67
566-LD	35	Lab Duplicate	7-Mar-97	2677	2634	-1.61
573-LD	41	Lab Duplicate	7-Mar-97	164	164	0.00
583-LD	L2A-03P-13	Lab Duplicate	7-Mar-97	21	21	0.47
593-LD	20	Lab Duplicate	7-Mar-97	3944	3963	0.48
602-LD	28	Lab Duplicate	7-Mar-97	90	90	0.00
609-LD	35	Lab Duplicate	7-Mar-97	88	88	-0.23
617-LD	L2A-02P-12	Lab Duplicate	8-Mar-97	403	443	9.93
627-LD	21	Lab Duplicate	8-Mar-97	5244	5533	5.51
636-LD	L2A-02AP-09	Lab Duplicate	8-Mar-97	11	11	3.64
645-LD	17.4	Lab Duplicate	8-Mar-97	58514	58889	0.64
655-LD	24	Lab Duplicate	8-Mar-97	79612	79601	-0.01
670-LD	39	Lab Duplicate	8-Mar-97	1183	1277	7.95

**Table Appendix D-13. Field Duplicates for March 1997 Soil Samples**

Sample ID	Location	Sample Type	Analysis Date	Soil Sample TCE, ug/kg	Duplicate Sample TCE, ug/kg	% Difference
506-FD	L2A-04P-11	Field Duplicate	6-Mar-97	508	447	-12.01
517-FD	24	Field Duplicate	6-Mar-97	12236	4971	-59.37
528-FD	35	Field Duplicate	6-Mar-97	6008	5314	-11.55
541-FD	L2A-05P-11	Field Duplicate	7-Mar-97	819	876	6.96
552-FD	21	Field Duplicate	7-Mar-97	43916	32077	-26.96
558-FD	27	Field Duplicate	7-Mar-97	18755	17670	-5.79
562-FD	31.5	Field Duplicate	7-Mar-97	15353	9365	-39.00
581-FD	L2A-03P-11	Field Duplicate	7-Mar-97	75	57	-24.00
598-FD	24	Field Duplicate	7-Mar-97	18576	22318	20.14
613-FD	L2A-02P-08	Field Duplicate	8-Mar-97	361	324	-10.25
635-FD	L2A-02AP-08	Field Duplicate	8-Mar-97	7	23	228.57
657-FD	26	Field Duplicate	8-Mar-97	91224	97055	6.39
666-FD	35	Field Duplicate	8-Mar-97	3673	4124	12.28

*Table Appendix D-14. Equipment Rinsates for March 1997 Soil Samples*

Sample ID	Location	Sample Type	Analysis Date	TCE Conc. Extract (ug/l)
514-ER	L2A-04P-19	Equip. Rinsate	6-Mar-97	5 U
520-ER	27	Equip. Rinsate	6-Mar-97	5 U
534-ER	40	Equip. Rinsate	6-Mar-97	5 U
545-ER	L2A-05P-15	Equip. Rinsate	7-Mar-97	5 U
554-ER	23	Equip. Rinsate	7-Mar-97	5 U
570-ER	36	Equip. Rinsate	7-Mar-97	5 U
609-ER	L2A-03P-35	Equip. Rinsate	8-Mar-97	5 U
616-ER	L2A-02P-11	Equip. Rinsate	8-Mar-97	5 U
638-ER	L2A-02AP-11	Equip. Rinsate	8-Mar-97	5 U
649-ER	L2A-02AP-18.5	Equip. Rinsate	8-Mar-97	5 U
671-ER	39	Equip. Rinsate	8-Mar-97	5 U

*Table Appendix D-15. Comparison of Passive vs. Sonic Extraction for March 1997 Soil Samples*

Sample ID	Location	Sample Type	Analysis Date	Passive Extraction	Sonication	% Difference
				TCE in Extract, ug/l	TCE in Extract, ug/l	
500	L2A-04P-05	Soil Sample	6-Mar-97	2568	2747	6.97
501	6	Soil Sample	6-Mar-97	1913	1973	3.14
502	7	Soil Sample	6-Mar-97	2253	2300	2.09
503	8	Soil Sample	6-Mar-97	850	918	8.00
504	9	Soil Sample	6-Mar-97	1551	1584	2.13
509	14	Soil Sample	6-Mar-97	11.2	11.6	3.57
510	15	Soil Sample	6-Mar-97	19.2	19.1	-0.52
511	16	Soil Sample	6-Mar-97	86	88	2.33
				248	261	5.24

## Appendix E. QA Audits

Table Appendix E-1. Rapid Commercialization Initiative Lasagna™ Project at Paducah Gaseous Diffusion Plant Independent Verification Assessments

<u>Dates</u>	<u>Focus</u>	<u>Key Issue(s)</u>	<u>Corrective Actions</u>
August 8-9, 1996	Field sampling of monitoring wells and field screening laboratory	Sampling technique resulted in unnecessary sample agitation.	Sampling technique modified to use a bailer to take the samples.
		Field samples were not immediately placed in an iced cooler after collection.	Iced cooler was utilized in future sampling events.
		Standards used for quality control checks were not documented.	Documentation was enhanced to trace the standards to the preparation logbook and the certifications.
		Deficiencies in the field screening laboratory HVAC system could compromise the stability of gas chromatography.	Issue not yet resolved, however, increased attention given to instrument stability.
		Communications of requirements between LMES and CDMFPC field laboratory were inadequate.	Analytical requirements documented by field variances. CDMFPC laboratory personnel were aware of LMES method requirements and made the necessary changes to reflect laboratory practices.
August 13-15, 1996	Audit of the Roy F. Weston Laboratory (now RECRA), reviewed scope of the project	No issues were identified.	Not applicable.

September 23-24, 1996	Field sampling of monitoring wells, field screening laboratory, and LMUS GC/MS techniques	Groundwater was sampled without purging within two weeks of the event.	Purging of wells changed to two days before sampling event.
		Plastic sheeting used for sampling is not maintained clean therefore the potential of cross-contamination.	New plastic sheets were instituted for next sampling events.
		Field screening laboratory had difficulty in sample dilution and aliquoting.	Syringe delivery accuracy was corrected by procedure modification that included opening the septa to release the vacuum and taking larger initial aliquots to make the primary dilutions.
November 12-14, 1996	Field sampling of monitoring wells, air monitoring, carbon cassette sampling, field laboratory GC/MS, LMUS volatiles laboratory	Flow rates for air sampling were not checked at the time intervals as stated in the procedure.	Field observations at subsequent monitoring verified that the procedure was being followed as written.
		Air monitoring did not start at the time specified in the procedure.	A procedure deviation was added to the QA Plan to allow adequate time for the ground vapor flux to reach equilibrium.
		Carbon cassette "socks" were placed in the same bucket and sheets of plastic without decon between or clean buckets and sheeting, possible cross contamination.	Subsequent field observations of carbon cassette techniques indicated that "socks" were placed on individual clean plastic sheets.



		The iron core of the cassettes was pulled through the entire length of the sausage which could result in cross contamination.	Teflon rope was substituted for the iron core rope. Eliminates the iron/TCE cross contamination.
		Field Laboratory QA Plan Rev 2 was a draft and has not been issued.	Plan was issued and formally controlled as Rev. 2.
January 9-10, 1997	Field sampling of monitoring wells, field record keeping, carbon cassette sampling, field laboratory GC/MS, LMUS volatiles laboratory	Carbon cassettes were not of uniform (one foot) lengths as specified in the plan. No documentation of length of the socks when put together.	Corrective measures were taken to ensure that the socks were one foot in length and tied with plastic tie wraps.
		Method documentation of the ethane/ethene procedure for the LMUS GC was incomplete.	"Analysis of Water for Ethane, Ethene, Acetylene" Procedure CP4-TS-OA7212 issued 03/26/97
		ORNL/GJ laboratory deviated from the EPA Method in quantifying organic concentrations (i.e. samples were being quantified based on continuing calibration response factor).	Inspection of the data packages found no significant variances of the data.
February 5, 1997	Air flux monitoring, screening laboratory ion trap mass spectrometry, documentation, and sample packaging	Custody seals were not placed on samples going to the commercial laboratory.	After the field crews were informed to use custody seals, all subsequent shipments had the proper seals placed on the sample containers.
		Calibration of air sampling pumps was not representative of the actual flow configuration used during the field sampling.	Subsequent tests by sampling personnel concluded that there was no significant difference in the airflow (<0.5%) with or without the charcoal tube installed.

March 4-5, 1997	Soil boring techniques, soil sample packaging, field documentation, and field GC techniques.	Field analysis by GJ staff lacked periodic verification of analyte recovery.	Subsequent samples were analyzed as replicates; half sonicated, half shaken. Results were within 3% of each other. No bias associated.
		Field procedures and the work plan were not present at the sampling location.	Subsequent review found procedures (dated 6/20/97) and other documentation was at the work site.
August 12-13, 1997	Soil boring techniques, soil sample packaging, field documentation, and carbon cassette sampling	No issues were identified.	Not applicable.
September 10-12, 1997	Audit of Roy F. Weston (now called Recra Labnet). Observed ongoing sample analysis of soil extracts, interviewed chemists, and reviewed QC for earlier analysis including direct injection and purge and trap GC analysis.	No issues were identified.	Not applicable.

Table Appendix E-2. Field Blank Results Lasagna™ Phase IIa Field Sampling

**GROUNDWATER**

<u>Date</u>	<u>Sample No.</u>	<u>Sample Description</u>	<u>Laboratory that Analyzed Sample</u>	<u>TCE Results (ppbw (µg/L))</u>
07-Aug-96	500	Equipment Rinsate	CDMFPC	ND
07-Aug-96	501	Trip Blank	CDMFPC	ND
08-Aug-96	503	Trip Blank	CDMFPC	ND
08-Aug-96	504	Trip Blank	LMUS	26
12-Aug-96	514	Trip Blank	CDMFPC	ND
12-Aug-96	515	Trip Blank	LMUS	ND
27-Aug-96	524	Trip Blank	CDMFPC	ND
27-Aug-96	525	Trip Blank	LMUS	ND
27-Aug-96	526	Rinsate	CDMFPC	ND
10-Sep-96	538	Trip Blank	CDMFPC	ND
10-Sep-96	539	Trip Blank	Weston	ND
10-Sep-96	540	Field Blank	CDMFPC	ND
10-Sep-96	551	Trip Blank	LMUS	ND
24-Sep-96	552	Trip Blank	CDMFPC	ND
24-Sep-96	553	Trip Blank	LMUS	ND
24-Sep-96	562	Equipment Rinsate	CDMFPC	ND
13-Nov-96	564	Trip Blank	CDMFPC	ND
13-Nov-96	565	Trip Blank	Weston	ND
13-Nov-96	566	Trip Blank	LMUS	ND
13-Nov-96	567	Equipment Rinsate	CDMFPC	1.9
13-Nov-96	568	Field Blank	CDMFPC	1.4
12-Dec-96	578	Trip Blank	CDMFPC	ND
12-Dec-96	579	Trip Blank	LMUS	ND
12-Dec-96	580	Trip Blank	Weston	Canceled
12-Dec-96	591	Trip Blank	CDMFPC	ND
12-Dec-96	592	Trip Blank	LMUS	ND
13-Feb-97	601	Trip Blank	CDMFPC	ND
13-Feb-97	602	Trip Blank	LMUS	ND
13-Feb-97	609	Rinsate	CDMFPC	ND
13-Feb-97	610	Field Blank	CDMFPC	ND
13-Feb-97	611	DI-Water	CDMFPC	1
13-Feb-97	612	Trip Blank	LMUS	1.7
07-Mar-97	622	Trip Blank	LMUS	ND
07-Mar-97	623	Trip Blank	LMUS	ND
30-Apr-97	635	Equipment Rinse	CDMFPC	ND
06-Jun-97	649	Field Blank	CDMFPC	ND
07-Aug-97	695	Field Blank	CDMFPC	ND

Table Appendix E-3a. Lasagna™ RCI Program Reference Laboratory Data Validation Results

<u>SDG#</u>	<u>Matrix</u>	<u>Sample ID</u>	<u>LDC Report #</u>	<u>Comments/Problems</u>
9608L576	Water	111070302-510 111070302-511 111070302-511MS 111070302-511MSD	1934A37	Samples Received at 9.4C
9608L598	Water	LOT:LA59417-1 LOT:LA59417-2 LOT:LA59417-3 LOT:LA59417-4 LOT:LA59417-5	1934B37	PE Samples
9608L614	Water	111070302-518 111070302-520	1934C37	None
9608L598	Water	LOT:LA59417-6 LOT:LA59417-7 LOT:LA59417-8 LOT:LA59417-9 LOT:LA59417-10	1934D37	PE Samples
9608L933	Water	111070302-529 111070302-531 111070302-537 111070302-537MS 111070302-537MSD	1968A37	Vinyl Chloride - 17.8%D outside QC limits
9609L119	Water	111070302-543 111070302-546 111070302-539 111070302-546MS 111070302-546MSD	1980A37	Samples received at 7.2C

**Table Appendix E-3b. Lasagna™ RCI Program Reference Laboratory Data Validation Results**

9609L353	Water	111070302-556 111070302-560 111070302-560MS 111070302-560MSD	2004A37	None
9611L231	Water	111070302-565 111070302-571 111070302-574	2049A37	No MS, MSD associated with these samples.
9611L293	Air Tubes	FC-1 FRONT FC-2 FRONT FC-3 FRONT FC-4 FRONT FC-5 FRONT FC-6 FRONT FC-7 FRONT FC-8 FRONT FC-9 FRONT FC-10 FRONT FC-11 FRONT FC-12 FRONT FC-13A FC-13B FC-13C FRONT FC-13D	2058A37	Initial calibration
9611L292	Extracts	C-06-1 C-06-2 C-06-3 C-06-4 C-06-5 C-01-16-D C-01-17-D C-02-18-D C-02-19-D C-04-17-D	2072A37	None
9612L664	Water	111070302-583 111070302-586	2105A37	No MS, MSD associated with these samples. Missed Holding Times (-586 only) by 5 days
9701L925	Water	111070302-595 111070302-598	2129A37	No MS, MSD associated with these samples.

**Table Appendix E-3c. Lasagna™ RCI Program Reference Laboratory Data Validation Results**

9706L901	Water	111070302-652 111070302-655 111070302-658 111070302-661 111070302-661MS 111070302-661MSD 111070302-664	2300A37	No Problems Found
9702L275	Water	111070302-605 111070302-608  <b>111070302-613</b> <b>111070302-618</b>	2167A37	No MS, MSD associated with these samples. %R for vinyl chloride was outside QC limits. <b>No Inorganic LDC validation packages has been received.</b>
9702L229	Air Tubes			
9703L613	Extract	LCD-008 LCD-008RE LCD-018 LCD-028 LCD-028RE LCD-038 LCD-016 LCE-032 LCE-032RE LCE-037 LCE-037RE LCE-058	2207B37	
9703L546	Extract	506-FD 517-FD 528-FD 541-FD 552-FD 558-FD 562-FD 581-FD 598-FD 613-FD 635-FD 657-FD 666-FD	2207A37	Samples received at 15.2C
9705L315	Water	111070302-639 111070302-644	2253A37	No MS, MSD associated with these samples.

**Table Appendix E-4. Laboratory Hexane Blanks - ORNL/GJ Field Laboratory**

<b><u>EXTRACTION DATE</u></b>	<b><u>TCE (ppbw (µg/L))</u></b>
6-Mar-97	5 U
6-Mar-97	5 U
6-Mar-97	5 U
6-Mar-97	5 U
7-Mar-97	5 U
7-Mar-97	5 U
7-Mar-97	5 U
7-Mar-97	5 U
7-Mar-97	5 U
7-Mar-97	5 U
7-Mar-97	5 U
7-Mar-97	5 U
7-Mar-97	5 U
8-Mar-97	5 U
8-Mar-97	5 U
8-Mar-97	5 U
8-Mar-97	5 U
8-Mar-97	5 U
8-Mar-97	5 U
9-Mar-97	5 U
9-Mar-97	5 U
9-Mar-97	5 U
9-Mar-97	5 U

**Table Appendix E-5. ORNL/GJ Field Laboratory Soil Analysis by Gas Chromatography  
Instrument Calibration Verification**

<b>Sample Type</b>	<b>Analysis Date</b>	<b>TCE Conc. Initial Cal (ppbw (µg/L))</b>	<b>TCE Conc. Cal Check (ppbw (µg/L))</b>	<b>% Difference</b>
100 ppb TCE	6-Mar-97	106.5	110.6	3.85
100 ppb TCE	6-Mar-97	106.5	113.2	6.29
100 ppb TCE	6-Mar-97	106.5	112.1	5.26
100 ppb TCE	6-Mar-97	106.5	113.1	6.20
100 ppb TCE	6-Mar-97	106.5	113.8	6.85
100 ppb TCE	6-Mar-97	106.5	113.6	6.67
100 ppb TCE	6-Mar-97	106.5	114.7	7.70
100 ppb TCE	6-Mar-97	106.5	114.6	7.61
250 ppb TCE	7-Mar-97	267.6	277.6	3.74
100 ppb TCE	7-Mar-97	106.5	113.7	6.76
100 ppb TCE	7-Mar-97	106.5	114.1	7.14
100 ppb TCE	7-Mar-97	106.5	115.4	8.36
100 ppb TCE	7-Mar-97	106.5	117	9.86
250 ppb TCE	7-Mar-97	267.6	278.1	3.92
100 ppb TCE	7-Mar-97	106.5	116.7	9.58
100 ppb TCE	7-Mar-97	106.5	117.3	10.14
100 ppb TCE	7-Mar-97	106.5	116.9	9.77
100 ppb TCE	7-Mar-97	106.5	116.9	9.77
100 ppb TCE	7-Mar-97	106.5	116.3	9.20
250 ppb TCE	7-Mar-97	267.6	289.4	8.15
100 ppb TCE	7-Mar-97	106.5	117.2	10.05
750 ppb TCE	8-Mar-97	754.4	817.6	8.38
100 ppb TCE	8-Mar-97	106.5	118	10.80
100 ppb TCE	8-Mar-97	106.5	117.5	10.33
100 ppb TCE	8-Mar-97	106.5	118	10.80
100 ppb TCE	8-Mar-97	106.5	117.3	10.14
100 ppb TCE	8-Mar-97	106.5	119.4	12.11
100 ppb TCE	8-Mar-97	106.5	118.8	11.55
100 ppb TCE	8-Mar-97	106.5	119.1	11.83
250 ppb TCE	8-Mar-97	267.6	292	9.12
250 ppb TCE	8-Mar-97	267.6	291	8.74
250 ppb TCE	8-Mar-97	267.6	295	10.24
250 ppb TCE	8-Mar-97	267.6	294.7	10.13
250 ppb TCE	9-Mar-97	267.6	291.4	8.89
100 ppb TCE	9-Mar-97	106.5	115.5	8.45
250 ppb TCE	9-Mar-97	267.6	278.6	4.11
250 ppb TCE	9-Mar-97	267.6	279	4.26
250 ppb TCE	9-Mar-97	267.6	278.1	3.92
100 ppb TCE	9-Mar-97	106.5	114.2	7.23
100 ppb TCE	9-Mar-97	106.5	110	3.29



## ***Appendix F. Measurement of C<sub>2</sub> Hydrocarbons in Well Water Samples***

---

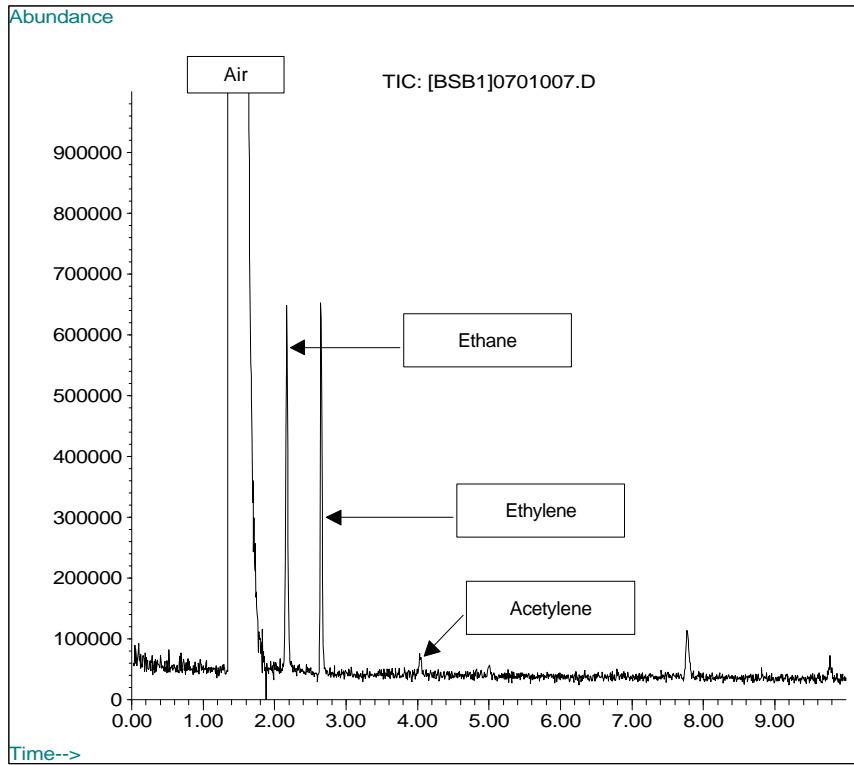
The present appendix outlines the consortium's plans to measure the concentrations of C<sub>2</sub> hydrocarbons (acetylene, ethylene, and ethane) in well water from the Phase IIa field experiment. There does not appear to be any standard EPA method for the analysis of these very volatile compounds in water. However LMUS laboratory has two different SOPs which they combine to provide this information. Appendix L of the QAPP shows these two SOPs and the present appendix describes how those SOPs will be used to measure C<sub>2</sub> hydrocarbons in water.

### **Background**

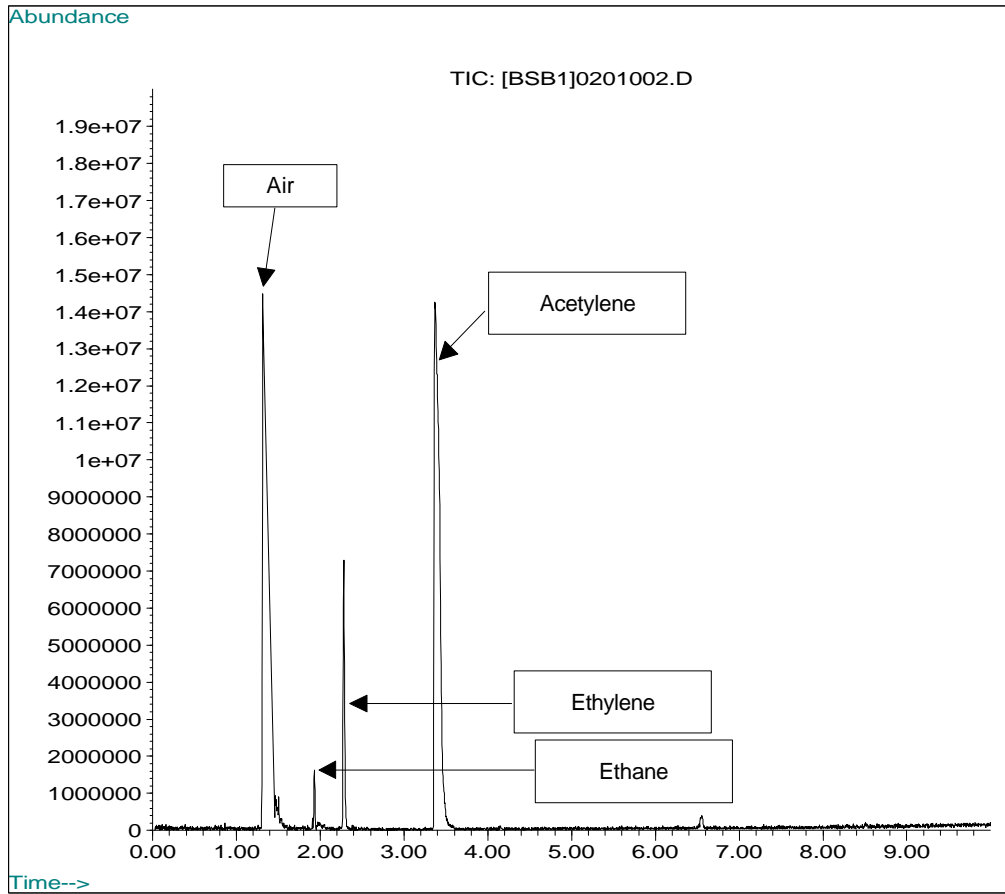
Documenting the fact that TCE is being degraded in Phase IIa is critical in order to show that the *Lasagna*<sup>TM</sup> process could be used to clean up clay contaminated with TCE. The current field study at Paducah has been complicated due to the presence of free organic liquid called DNAPL (dense non-aqueous phase liquid). In laboratory studies, degradation of TCE has been shown to occur when TCE is at concentrations below the saturation limit as well as above the saturation limit. Figures Appendix F-1 and Appendix F-2 show the chromatograms of the C<sub>2</sub> degradation products from these two different TCE concentration regions. Figure Appendix F-3 summarizes the major degradation products from DNAPL or dissolved TCE.

Table F-11 and Figure F-5 show the analysis of the C<sub>2</sub> hydrocarbons in water samples taken during the field experiment. These data were used to demonstrate that TCE degradation is occurring, which can, in turn, showed that DNAPL degradation is occurring. The degradation of DNAPL is an extension to the original *Lasagna*<sup>TM</sup> concept where it was thought that only TCE which was completely solubilized in water could be treated.

**Figure Appendix F-1. GC/MS Chromatogram of C<sub>2</sub> Degradation Products When TCE is Present Below the Solubility Limit (280 ppmw (µg/g) TCE in Water).**



**Figure Appendix F-2. GC/MS Chromatogram of C<sub>2</sub> Degradation Products When TCE is Present as DNAPL (Prepared With 5,800 ppmw (µg/g) TCE in Water).**



**Figure Appendix F-3. Degradation Products When TCE is DNAPL or Dissolved.**

