Enhanced Oxidative Bioremediation of cis-dichloroethene (cis-DCE) and Vinyl Chloride (VC) using Electron Shuttles

ESTCP Project ER-0316

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CDM

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<th>Acronym</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>AFB</td>
<td>Air Force base</td>
</tr>
<tr>
<td>AFCEE</td>
<td>Air Force Center for Engineering and the Environment</td>
</tr>
<tr>
<td>AQDS</td>
<td>Anthraquinone disulfonic acid</td>
</tr>
<tr>
<td>AQS</td>
<td>2-Anthraquinone sulfonic acid</td>
</tr>
<tr>
<td>BAFeIII</td>
<td>Bioavailable ferric iron</td>
</tr>
<tr>
<td>CDM</td>
<td>Camp Dresser &amp; McKee Inc.</td>
</tr>
<tr>
<td>cis-DCE</td>
<td>cis-1,2-Dichloroethene</td>
</tr>
<tr>
<td>COCs</td>
<td>Contaminants of concern</td>
</tr>
<tr>
<td>DO</td>
<td>Dissolved oxygen</td>
</tr>
<tr>
<td>EPA</td>
<td>Environmental Protection Agency</td>
</tr>
<tr>
<td>ESTCP</td>
<td>Environmental Security Technology Certification Program</td>
</tr>
<tr>
<td>FA</td>
<td>Fulvic acid</td>
</tr>
<tr>
<td>Fe$^{+2}$</td>
<td>Ferrous iron</td>
</tr>
<tr>
<td>Fe$^{+3}$</td>
<td>Ferric iron</td>
</tr>
<tr>
<td>FeRB</td>
<td>Iron-reducing bacteria</td>
</tr>
<tr>
<td>ft bgs</td>
<td>Feet below ground surface</td>
</tr>
<tr>
<td>g/L</td>
<td>Grams per liter</td>
</tr>
<tr>
<td>GC/RD</td>
<td>Gas chromatography with radiometric detection</td>
</tr>
<tr>
<td>HA</td>
<td>Humic acid</td>
</tr>
<tr>
<td>IHSS</td>
<td>International Humic Substances Society</td>
</tr>
<tr>
<td>IR</td>
<td>Installation Restoration</td>
</tr>
<tr>
<td>KOC</td>
<td>Organic carbon partition coefficient</td>
</tr>
<tr>
<td>L/kg</td>
<td>Liters per kilogram</td>
</tr>
<tr>
<td>LF-3</td>
<td>Landfill 3</td>
</tr>
<tr>
<td>mg</td>
<td>Milligrams</td>
</tr>
<tr>
<td>mg/L</td>
<td>Milligrams per liter</td>
</tr>
<tr>
<td>mg-N/L</td>
<td>Milligrams nitrogen per liter</td>
</tr>
<tr>
<td>mL/min</td>
<td>Milliliters per minute</td>
</tr>
<tr>
<td>mM</td>
<td>Millimoles per liter</td>
</tr>
<tr>
<td>MSL</td>
<td>Mean sea level</td>
</tr>
<tr>
<td>MTBE</td>
<td>Methyl tertiary butyl ether</td>
</tr>
<tr>
<td>mV</td>
<td>Millivolts</td>
</tr>
<tr>
<td>NA</td>
<td>Not available or not applicable</td>
</tr>
<tr>
<td>NAVFAC</td>
<td>Naval Facilities Engineering Command</td>
</tr>
<tr>
<td>NFESC</td>
<td>Naval Facilities Engineering Service Center</td>
</tr>
<tr>
<td>ORP</td>
<td>Oxidation-reduction potential</td>
</tr>
<tr>
<td>PCE</td>
<td>Tetrachloroethene</td>
</tr>
<tr>
<td>SSC-OTC</td>
<td>Space and Naval Warfare Systems Center Old Town Campus</td>
</tr>
<tr>
<td>Acronym</td>
<td>Definition</td>
</tr>
<tr>
<td>---------</td>
<td>----------------------------------</td>
</tr>
<tr>
<td>TCE</td>
<td>Trichloroethene</td>
</tr>
<tr>
<td>TOC</td>
<td>Total organic carbon</td>
</tr>
<tr>
<td>µg/L</td>
<td>Micrograms per liter</td>
</tr>
<tr>
<td>µmole</td>
<td>Micromole</td>
</tr>
<tr>
<td>USCS</td>
<td>Unified Soil Classification System</td>
</tr>
<tr>
<td>USGS</td>
<td>United States Geological Survey</td>
</tr>
<tr>
<td>VC</td>
<td>Vinyl chloride</td>
</tr>
<tr>
<td>VOCs</td>
<td>Volatile organic compounds</td>
</tr>
<tr>
<td>WHO</td>
<td>World Health Organization</td>
</tr>
</tbody>
</table>
Section 1
Introduction

1.1 Project Scope

The original scope of this project involved demonstration and validation of the use of electron shuttles compounds for the oxidative biodegradation of cis-1,2-dichloroethene (cis-DCE) and vinyl chloride (VC). As results from the treatability study using radiolabeled vinyl chloride were not encouraging, the project was cancelled. Since this project was not completed as planned, this final report does not follow the standard Environmental Security Technology Certification Program (ESTCP) report format. This report presents the findings from field sampling and analysis and treatability testing that was conducted at both Dr. Derek Lovley’s and Dr. Frank Chapelle’s laboratories. Specifically, this report includes the following:

- A brief description of electron shuttling processes and example electron shuttle properties.
- The revised objectives of this study and a diagram indicating the interrelationships amongst the different aspects.
- Materials and methods used in the study along with brief descriptions of the sites where samples were collected.
- Results of the study including: 1) electron shuttle characterization (work performed at Dr. Derek Lovley’s laboratory), 2) site sampling and analysis; and 3) microcosm testing (work performed at Dr. Frank Chapelle’s laboratory).
- Conclusions of the study.

Upon cancellation of ER0316, obligated ESTCP funds were redirected towards two related and parallel efforts. The first effort was a Workshop on In Situ Biogeochemical Transformation. A separate report on the findings of this workshop was previously delivered and has been published (AFCEE, 2008). The second effort involved sampling and analysis of Air Force biowall sites where biogeochemical transformation appears to have been occurring. This work is in progress and a report is in preparation.

1.2 Electron Shuttle Background

Electron shuttles are compounds that stimulate biodegradation of contaminants by facilitating electron transfer to and from bacteria (Benz et al. 1998, Bradley et al. 1998, Finneran and Lovley 2001, Hernandez and Newman 2001, Lovley et al. 1996a, Lovley et al. 1998, Newman and Kolter 2000, Scott et al. 1998). These compounds can accept electrons from bacteria that are oxidizing a contaminant such as VC (Bradley et al. 1998) and subsequently donate those electrons to a terminal electron acceptor such as ferric iron. Electron shuttles can facilitate oxidative and potentially reductive modes of contaminant biodegradation.
In the oxidative role, an electron shuttle serves as the initial electron acceptor. Bacteria donate electrons to the electron shuttle following oxidation of the contaminant. If another terminal electron acceptor such as ferric iron (Fe$^{3+}$) is present, the reduced electron shuttle donates electrons to ferric iron and in doing so is regenerated (i.e., oxidized) and ferrous iron (Fe$^{2+}$) is produced. The regenerated electron shuttle is then capable of facilitating additional contaminant oxidation. **Figure 1** is a schematic representation of the electron shuttling process using VC as an example contaminant. This shuttling of electrons between bacteria and ferric iron highlights the utility of electron shuttles. Ferric iron is present as a solid phase in aquifers and is not always in intimate contact with bacteria. The electron shuttle functions as an electronic bridge between the bacteria and the ferric iron and in doing so maximizes the bioavailability of this ubiquitous terminal electron acceptor.

**Figure 1 Example Electron Shuttling Schematic Diagram Showing Cycling of Reduced (ES-Red) and Oxidized (ES-Ox) Electron Shuttles.**

One reason that the availability of electron shuttles has a significant impact on contaminant oxidation is that iron-reducing bacteria (FeRB) are capable of degrading organic contaminants including VC, *cis*-DCE, benzene, toluene, and methyl tertiary butyl ether (MTBE) (Anderson et al. 1998, Bradley and Chapelle 1996, Bradley and Chapelle 1998, Finneran and Lovley 2001, Lovley et al. 1996b, Lovley 1997). The ability of FeRB to degrade these contaminants *in situ* is often limited by bioavailability of ferric iron. Electron shuttles such as humic acids and anthraquinone disulfonic acid (AQDS) have been shown to enhance biodegradation of organic chemicals including VC and *cis*-DCE (Bradley et al. 1998), benzene (Lovley et al. 1996a,b), toluene (Evans 2000), and MTBE (Finneran and Lovley 2001). Column studies have demonstrated that electron shuttles stimulated toluene biodegradation under iron-reducing conditions (Evans 2000).

Enhancement of bioremediation using electron shuttles is a potentially feasible and cost-effective approach to site remediation. This technology could be applied at chlorinated solvent sites where the accumulation of *cis*-DCE and VC has been observed. For example, electron shuttles have been observed to increase the biodegradation rates of *cis*-DCE and VC in microcosms (Bradley et al. 1998). *cis*-DCE and VC are of particular interest since they are ubiquitously found at sites where complete reductive dechlorination of tetrachloroethene (PCE) or trichloroethene (TCE) was incomplete. This is often the situation at natural attenuation sites as well as many sites where enhanced anaerobic bioremediation has been attempted.
In a typical remediation scenario, the selected electron shuttle formulation would be added to groundwater through a series of injection wells or in a trench. Ideally, the electron shuttle formulation would be transported downgradient along the natural groundwater flow path. Groundwater can be recirculated if regulatory requirements require capture of the injected electron shuttles or if increased residence times are desired. If downgradient groundwater needs to be extracted it can be reintroduced through the injection trench or wells. Additional electron shuttles could be added to the extracted groundwater as necessary. As the electron shuttles are transported downgradient, they will accept electrons from bacteria that have oxidized VC and cis-DCE. The reduced electron shuttles will then donate the electrons to bioavailable ferric iron or other terminal electron acceptors. The regenerated electron shuttles will continue to shuttle electrons between the bacteria and the terminal electron acceptors until they are transported beyond the plume boundary or capture zone.

Examples of electron shuttles include humic and fulvic acids, quinones such as the humic acid analogue AQDS, phenazines, and potentially thiol-containing molecules such as cysteine (Hernandez and Newman 2001; Lovley et al. 1998). The chemical structures of these compounds typically include aromatic resonance structures as shown in Figure 2. Humic and fulvic acids are naturally abundant in nature. AQDS is a synthetic compound that is unlikely to be used in the field because of regulatory restrictions on its injection. Alternative compounds such as the food colorant indigo disulfonic acid are potential alternatives that were evaluated in this study. In general, factors that affect practical applications include electron shuttling efficiency, fate and transport in the environment, toxicity, solubility, and cost. Table 1 lists various electron shuttle candidates and gives an overview of their chemical and toxicological properties. Additional information on the toxicology of humic acids, AQDS, and indigo disulfonate is presented in Appendix A.

<table>
<thead>
<tr>
<th>Example Electron Shuttles and Structures</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Humic acids</strong></td>
</tr>
<tr>
<td>AQDS</td>
</tr>
<tr>
<td>AQS</td>
</tr>
<tr>
<td>Alizarin</td>
</tr>
<tr>
<td>Emodin*</td>
</tr>
<tr>
<td>Lawson!</td>
</tr>
<tr>
<td>Henna*</td>
</tr>
<tr>
<td>Juglone*</td>
</tr>
<tr>
<td>Popurin*</td>
</tr>
<tr>
<td>Vitamin K*</td>
</tr>
<tr>
<td>Menadione*</td>
</tr>
<tr>
<td><strong>Melanin</strong></td>
</tr>
<tr>
<td><strong>Methyl viologen</strong></td>
</tr>
<tr>
<td><strong>ACNQ</strong></td>
</tr>
<tr>
<td><strong>Menaquinone</strong></td>
</tr>
<tr>
<td><strong>2,6-bi</strong></td>
</tr>
<tr>
<td><strong>Indigo carmine</strong></td>
</tr>
<tr>
<td><strong>Ubiquinone</strong></td>
</tr>
<tr>
<td><strong>Cysteine</strong></td>
</tr>
<tr>
<td><strong>Co-enzyme Q</strong></td>
</tr>
<tr>
<td><strong>Mulch</strong></td>
</tr>
</tbody>
</table>

*Derived from natural sources

Figure 2 Electron Shuttle Chemical Structures
<table>
<thead>
<tr>
<th>Common Name(s)</th>
<th>Chemical Name</th>
<th>CAS No.</th>
<th>Formula</th>
<th>Molecular Weight</th>
<th>Health Hazards Acute and Chronic</th>
<th>LD50 for Mouse/Rat (mg/kg)</th>
<th>Solubility in Water</th>
<th>Standard Potential, E° (V)</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACNQ 2-Amino-5-carboxy-1,4-naphthoquinone</td>
<td>NA</td>
<td>NA</td>
<td>C_{11}H_{10}NO_{4}</td>
<td>217</td>
<td>NA</td>
<td>NA</td>
<td>--</td>
<td>-0.071</td>
<td>1, 2, 14</td>
</tr>
<tr>
<td>Alizarin, Pigment Red 1,2-dihydroxyanthraquinone</td>
<td>NA</td>
<td>72-48-0</td>
<td>C_{14}H_{8}O_{4}</td>
<td>240.21</td>
<td>Acute: causes irritation to the respiratory tract, symptoms may include coughing, shortness of breath, causes irritation to skin, symptoms include redness, itching, and pain, may cause dermatitis, causes irritation, redness, and pain of the eyes. Chronic: No information found. Based on animal studies, substance is presumed to be moderately toxic.</td>
<td>NA</td>
<td>2.5 µM</td>
<td>--</td>
<td>13</td>
</tr>
<tr>
<td>Anthraquinone-2-sulfonic Acid Sodium Salt; Sodium Anthraquinone-beta-sulfonate; AQS Anthraquinone-2-sulfonic acid</td>
<td>131-08-8</td>
<td>C_{14}H_{8}O_{4}Na</td>
<td>310.26</td>
<td>Acute: may cause eye irritation, skin irritation, irritation of the digestive tract, respiratory tract irritation. Chronic: No information found. The toxicological properties of this substance have not been fully investigated.</td>
<td>663 (630 to 730)</td>
<td>Freely Soluble</td>
<td>-0.23</td>
<td>9</td>
<td></td>
</tr>
<tr>
<td>AQDS, 2AQDS; Anthraquinone-2,6-Disulfonate Anthraquinone-2,6-disulfonic acid</td>
<td>853-60-9</td>
<td>C_{14}H_{6}O_{8}S_{2}Na</td>
<td>412.29</td>
<td>Acute: may cause eye irritation, skin irritation, irritation of the digestive tract, respiratory tract irritation. Chronic: No information found. The toxicological properties of this substance have not been fully investigated.</td>
<td>2900</td>
<td>Soluble</td>
<td>-0.18</td>
<td>2, 3, 15, 16</td>
<td></td>
</tr>
<tr>
<td>Coenzyme Q 2,3-Dimethoxy-5-methyl-1,4-benzoquinone</td>
<td>605-94-7</td>
<td>C_{9}H_{10}O_{4}</td>
<td>182.17</td>
<td>NA</td>
<td>NA</td>
<td>11.1g/L</td>
<td>0.174</td>
<td>15</td>
<td></td>
</tr>
<tr>
<td>Cysteine</td>
<td>Cysteine</td>
<td>3374-22-9</td>
<td>C_{3}H_{7}NOS</td>
<td>121.16</td>
<td>Acute: No adverse health effects expected from inhalation, ingestion (large oral doses may cause general depressed activity), skin exposure. Chronic Exposure: No adverse health effects expected.</td>
<td>NA</td>
<td>Freely soluble</td>
<td>-0.39</td>
<td>18</td>
</tr>
<tr>
<td>Emolin 6-Methyl-1,3,8-trihydroxyanthraquinone</td>
<td>518-62-1</td>
<td>C_{14}H_{8}O_{4}</td>
<td>270.23</td>
<td>Acute: causes eye irritation, skin irritation, respiratory tract irritation, may cause irritation of the digestive tract. Chronic: No information found.</td>
<td>NA</td>
<td>Insoluble</td>
<td>--</td>
<td>20</td>
<td></td>
</tr>
<tr>
<td>Fulvic acid, Sodium Fulvate; Fulvic acid, sodium salt Fulvic acid</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>Acute: may be harmful if swallowed. Chronic: none reported. RTC CLASS OF COMPOUND- Drug; Natural Product</td>
<td>1093 (1009 to 1176)</td>
<td>Freely soluble</td>
<td>--</td>
<td>11</td>
</tr>
<tr>
<td>Henna Powdered Natural Henna</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>Been used by humans for centuries for hair and body treatment.</td>
<td>NA</td>
<td>Made - some components may be soluble</td>
<td>--</td>
<td>13</td>
</tr>
<tr>
<td>HGA-Melanin (polymerized homogenetic acid), Homogenetic Acid 2,5-Dihydroxyphenylacetic acid (polymerized)</td>
<td>451-13-8</td>
<td>C_{8}H_{9}O_{4}</td>
<td>168.15</td>
<td>Acute: may cause eye irritation, skin irritation, irritation of the digestive tract, respiratory tract irritation. Chronic: No information found. The toxicological properties of this substance have not been fully investigated.</td>
<td>NA</td>
<td>Freely soluble</td>
<td>--</td>
<td>21</td>
<td></td>
</tr>
<tr>
<td>Humic acid, Huminsaure natrum; Sodium Humate; Humic acid</td>
<td>68131-04-4</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>Acute: causes eye irritation, causes skin irritation, may cause irritation of the digestive tract, causes respiratory tract irritation. Chronic: No information found. The toxicological properties of this substance have not been fully investigated.</td>
<td>596 (111 to 1176)</td>
<td>Freely soluble at pH&gt;2</td>
<td>--</td>
<td>10</td>
</tr>
<tr>
<td>Indigosulfonate Dipotassium Salt; Indigo carmine; Acid blue Indigosulfonate</td>
<td>860-22-0</td>
<td>C_{14}H_{8}N_{2}Na_{2}O_{3}S</td>
<td>466.35</td>
<td>Acute: no adverse health effects expected from inhalation, ingestion, skin exposure; similar dyes have caused permanent injury to the cornea and conjunctiva in documented exposure cases with human or rabbit eyes. Chronic Exposure: No adverse health effects expected. RTC CLASS OF COMPOUND- Tumorigen; Drug; Mutagen</td>
<td>1250 (93 to 2500)</td>
<td>10 g/L or 0.021 M</td>
<td>-0.13</td>
<td>7, 8, 12</td>
<td></td>
</tr>
<tr>
<td>Juglone 5-Hydroxy-1,4-naphthoquinone</td>
<td>481-39-0</td>
<td>C_{10}H_{6}O_{2}</td>
<td>174.15</td>
<td>Acute: Not expected in humans, some irritation in animal studies. Chronic: Isomeric juglones stimulated cardiac effects on the isolated frog heart.</td>
<td>NA</td>
<td>Slightly soluble in hot water</td>
<td>0.05</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>Lawsonite; Natural brown 2-Hydroxy-1,4-naphthoquinone</td>
<td>83-72-7</td>
<td>C_{10}H_{6}O_{2}</td>
<td>174.15</td>
<td>Acute: slight to moderate skin and mucous membrane irritation, respiratory problems. Chronic: mutagenic in mice/hamster studied, one report deemed it to be not suitable for cosmetic use. RTC CLASS OF COMPOUND - Drug; Mutagen</td>
<td>330 (100 to 570)</td>
<td>up to 2% or 0.11% M</td>
<td>-0.14</td>
<td>9, 16</td>
<td></td>
</tr>
</tbody>
</table>
### Table 1, Continued

**Electron Shuttle Chemistry and Toxicity**

<table>
<thead>
<tr>
<th>Common Name(s)</th>
<th>Chemical Name</th>
<th>CAS No.</th>
<th>Formula</th>
<th>Molecular Weight</th>
<th>Health Hazards Acute and Chronic</th>
<th>LD50 for Mouse/Rat (mg/kg)</th>
<th>Solubility in Water</th>
<th>Standard Potential, Eo (V)</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methyl red</td>
<td>Benzoic acid, 2-[N-(dimethylamino)phenyl][azo]-</td>
<td>405-52-7</td>
<td>C₁₅H₁₅N₃O₂</td>
<td>269.3</td>
<td>Acute: May cause mild irritation to the mucous membranes, large oral doses may cause gastrointestinal disturbances, mild skin irritation. Chronic Exposure: POSSIBLE RISK OF IRREVERSIBLE EFFECTS. LABORATORY EXPERIMENTS HAVE SHOWN MUTAGENIC EFFECTS.</td>
<td>NA</td>
<td>Almost insoluble in water</td>
<td>--</td>
<td>19</td>
</tr>
<tr>
<td>Methyl viologen</td>
<td>Methyl viologen</td>
<td>1910-42-5</td>
<td>C₁₂H₁₄Cl₂N₂</td>
<td>287.18</td>
<td>Acute: Toxic if swallowed or absorbed through skin, may be harmful if inhaled, causes severe irritation, symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, vomiting, high concentrations are extremely destructive to tissues of mucous membranes and upper respiratory tract, eyes and skin. Chronic: May alter genetic material, contains components reported to be carcinogenic.</td>
<td>NA</td>
<td>up to at least 150 uM</td>
<td>-0.446</td>
<td>7, 8</td>
</tr>
<tr>
<td>Methylene blue</td>
<td>3,7- Bis(dimethylamino)phenothiazin-5-ium chloride trihydrate</td>
<td>61-73-4</td>
<td>C₁₂H₁₄ClN₃S</td>
<td>319.85</td>
<td>This material is relatively non-hazardous in routine industrial situations. Acute: No adverse health effects expected from inhalation, a burning sensation of the mouth may be noted following ingestion of methylene blue, may cause nausea, vomiting, diarrhea, and gas, large doses may cause abdominal and chest pain, headache, profuse sweating, mental confusion, painful micturition, and methemoglobinemia, not expected to be a health hazard from skin exposure, may color the skin a bluish color, may cause photosensitization, no adverse effects expected, may cause mechanical irritation. Chronic: Exposure: No information found.</td>
<td>NA</td>
<td>40 g/L or 0.125 M</td>
<td>0.011</td>
<td>7, 8</td>
</tr>
<tr>
<td>Naphthazarone</td>
<td>5,8-Hydroxy-1,4-naphthoquinone</td>
<td>475-38-7</td>
<td>C₁₀H₁₀O₅</td>
<td>190.15</td>
<td>Acute: May cause eye irritation, skin irritation, irritation of the digestive tract, respiratory tract irritation. Chronic: No information found. The toxicological properties of this substance have not been fully investigated.</td>
<td>NA</td>
<td>5.52 g/L</td>
<td>--</td>
<td>17</td>
</tr>
<tr>
<td>P-2,6bT</td>
<td>Pyridine-2,6-bis(thiocarboxylate)</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>2, 4</td>
</tr>
<tr>
<td>Purpurin</td>
<td>1,2-4-Trihydroxyanthraquinone</td>
<td>81-54-9</td>
<td>C₁₉H₁₂O₅</td>
<td>256.21</td>
<td>Acute: Causes eye irritation, may cause chemical conjunctivitis, causes skin irritation, may cause gastrointestinal irritation with nausea, vomiting and diarrhea, causes respiratory tract irritation, can produce delayed pulmonary edema. Chronic: Effects may be delayed.</td>
<td>NA</td>
<td>slightly more soluble than Alizarin</td>
<td>13</td>
<td></td>
</tr>
<tr>
<td>Ubiquinone, Coenzyme Q10</td>
<td>2,3 dimethyl-5 methyl-6-decaprenyl benzoquinone</td>
<td>303-98-0</td>
<td>C₁₉H₁₈O₄</td>
<td>880.34</td>
<td>NA</td>
<td>NA</td>
<td>5.23±0.013 mg/L</td>
<td>0.113</td>
<td>2, 5</td>
</tr>
<tr>
<td>Vitamin K3, Menadione, Menadione sodium bisulfate, Menaquinone</td>
<td>2-Methyl-1,4-naphthoquinone</td>
<td>58-27-5</td>
<td>C₁₀H₁₀O₅</td>
<td>172.18</td>
<td>Acute: Causes eye irritation, causes skin irritation, harmful if swallowed, causes respiratory tract irritation. Chronic: Not available. The toxicological properties of this substance have not been fully investigated.</td>
<td>NA</td>
<td>Bisulfate form is freely soluble</td>
<td>-0.20</td>
<td>2, 5</td>
</tr>
</tbody>
</table>

**Reference List:**

Reference List, Continued
1.3 Project Tasks

The original objective of this study was to validate the role of electron shuttles in stimulating contaminant degradation with a focus on the cis-DCE and VC. Two conceptual approaches were planned:

- An engineered approach where an aqueous solution of electron shuttle compounds is injected into the contaminated groundwater and the groundwater is potentially recirculated.

- A natural approach where mulch is used as a source of naturally occurring humic acids that have electron shuttling properties. The mulch is incorporated into biowalls such as those employed by the Air Force for contaminant degradation.

Figure 3 shows the project tasks associated with the evaluation of these approaches.

Figure 3 Project Tasks and Relationships

A brief description of each of these tasks is presented below. Section 2 presents the additional details regarding methodology used to execute the field and laboratory tasks.

Task 1
Task 1 involved a compilation and review of toxicological information and chemical properties for compounds or substances that have been identified in the scientific literature as having electron shuttling properties. This compilation was presented as Table 1. Based on this information, specific compounds were selected for further
evaluation. Compounds that had limited commercial availability, were toxic, had low water solubility, or had limited information were screened out. Compounds that were retained for further evaluation in Task 2 included:

- Humic acids
- Sulfonated humic acids
- Fulvic acids
- Henna
- Menadione sodium bisulfite
- 2,6-Anthraquinone disulfonic acid (AQDS)
- 2-Anthraquinone sulfonic acid (AQS)
- Indigo disulfonate
- Lawsone

**Task 2**
Task 2 involved measurement of electron shuttling capacity of the compounds selected in Task 1. The electron shuttling capacity is a quantitative indication of the potential of specific compounds to transfer electrons to ferric iron and cause its reduction to ferrous iron. These measurements were conducted at the University of Massachusetts using a standardized procedure described in Section 2 (Lovley, et al. 1996a; Nevin and Lovley 2002). Humic acids are a complex mixture of compounds with no single chemical structure; therefore, several commercial sources of humic acids were evaluated in this study. Section 2 presents the commercial sources of these products.

**Task 3**
Task 3 involved collection of soil and groundwater samples from the Space and Naval Warfare Systems Center Old Town Campus site in San Diego, California (SSC-OTC). SSC-OTC had been selected for a pilot scale demonstration based on site selection criteria, which included accumulation of VC in groundwater and potential for stimulation of biological iron reduction by electron shuttles. Site information is presented in Section 1.4. Additional details on this task are presented in Section 2.

**Task 4**
Task 4 involved further evaluation of the electron shuttling compounds evaluated in Task 2. These evaluations were conducted using SSC-OTC soil and groundwater and included evaluation of compound precipitation in site groundwater and soil-water partitioning (i.e., KOC). Additional details on these measurements are presented in Section 2.
Task 5
Task 5 involved selection of electron shuttling compounds to be tested in a microcosm study (Task 6). Results from Tasks 1, 2, and 4 were used to identify compounds that: a) had the lowest cost per unit of electron shuttling capacity, b) were soluble in site groundwater, and c) had different KOC values. The selected compounds included:

- 2,6-Anthraquinone disulfonic acid (AQDS)
- Indigo disulfonate
- Monterey Ag Products HA-12 humic acid
- LignoTech Borregro HA-1 sulfonated humic acid.

Task 6
Task 6 involved a microcosm study conducted by the United States Geological Survey (USGS) to evaluate the fate of radiolabeled VC in the presence of the electron shuttling compounds selected in Task 5. Additional details on this study are presented in Section 2.

Task 7
Task 7 was conducted by Parsons and involved sampling of biowall solids (i.e., mulch and sand) and groundwater from the biowall located at Altus Air Force Base (AFB) Landfill 3 (LF-3). Groundwater samples were collected upgradient, within, and downgradient of the biowall. Site information is presented in Section 1.4. Additional details on sampling are presented in Section 2 and Appendix B.

Task 8
Task 8 involved analysis of the biowall and groundwater samples for multiple analytes. Additional description is provided in Section 2. In general, analyses were conducted to characterize the geochemistry and electron shuttling potential within the biowall. The analyses included several standard analyses plus specialized analyses including electron shuttling capacity and bioavailable ferric iron. Additional details on sampling are presented in Section 2 and Appendix B.

1.4 Site Descriptions
This section presents an overview of the SSC-OTC and Altus AFB sites. The SSC-OTC site was used for evaluation of the engineered electron shuttle approach, i.e., injection of electron shuttling compounds. Sampling and analysis was conducted specifically to support Tasks 3, 4, and 6 as shown in Figure 3. The Altus AFB biowall site was used for evaluation of the natural electron shuttle approach, i.e., presence of humics in the mulch potentially acting as electron shuttles. Sampling and analysis was conducted specifically to support Tasks 7 and 8 as shown in Figure 3.
1.4.1 SSC-OTC

The SSC-OTC Site consists of approximately 60 acres located at 4297 Pacific Highway in San Diego, California. In the past, fill derived from dredged bay sediments was utilized to construct usable area at the Site. Currently, about 95 percent of the Site is developed, covered with buildings and pavement. Site topography is generally flat with elevations across the site ranging from approximately nine to ten feet above mean sea level (MSL).

Figure 4 shows an overview of the SSC-OTC Site with groundwater contours. The study was conducted in the vicinity of 11MW07 near Installation Restoration (IR) Site 11. IR Site 11 is located in Building 3 where a former sanitary sewer line break occurred beneath the northeastern area of the building. Processes within the building may have generated waste oil, paint sludge, spent acids, plating materials, degreasing solvents, and Oakite cleaners.

The SSC-OTC Site is located within the Pueblo San Diego Hydrologic Unit, San Diego Mesa Area, Lindbergh Sub area. Historically (Bechtel 2000) local groundwater elevations were measured between 7 and 18 feet below ground surface (ft bgs) or at approximately zero ft MSL. Groundwater flow direction is oriented to the northwest at a gradient of 0.003 ft/ft (Bechtel 2000). Groundwater flow, as indicated by the gradient, is generally to the north to northeast in the demonstration area in IR Site 11.

In general, historical data indicate the presence of high cis-DCE and VC concentrations in demonstration area groundwater. TCE was detected in groundwater up-gradient of the demonstration area in well 11GP-22. Gasoline range petroleum hydrocarbons were also detected in groundwater in the vicinity of the demonstration area. Evidence of anaerobic iron reduction is demonstrated by the elevated dissolved iron and manganese concentrations and low oxidation reduction potential. The SSC-OTC site was concluded to be suitable for the demonstration based on presence of cis-DCE and VC and evidence of iron reduction.
1.4.2 Altus AFB

The Altus AFB mulch biowall was constructed in June 2002 to stimulate reductive dechlorination of chlorinated VOCs in groundwater at Landfill 3 of the site (Henry 2004). This biowall was selected for evaluation in this study based on its demonstrated activity to remove TCE and cis-DCE without accumulation of VC. Appendix B includes a simplified plan view of the biowall location. This biowall was constructed to contain and attenuate a shallow groundwater plume contaminated with TCE and cis-DCE, in order to prevent surface water discharge or off-base migration. The source of impacts to shallow groundwater at the site is historical waste management activities.

The biowall is composed of shredded bark mulch, cotton gin compost, and sand (to maintain permeability). The mulch and compost substrates are intended to be used as solid-phase, long-term carbon sources to stimulate reductive dechlorination of TCE and its biodegradation products over periods of 5 years or more.

The mulch trench is intended to intercept over 80 percent of the groundwater plume contaminant flux (subject to depth limitations of the trencher). A monitoring well network was installed, including wells upgradient, downgradient, and within the biowall.

1.4.2.1 Site Geology

Surface soils in the mulch biowall vicinity consist of approximately 5 feet of clayey silt, and weathered and fractured stiff silty clay that extends to depth of approximately 25 to 30 ft bgs. These sediments are underlain by well-cemented silt and dense shale of the Hennessey Group of Permian age. Shallow groundwater occurs under unconfined conditions and generally flows towards the east-southeast to Stinking Creek. Shallow groundwater at the site occurs at a seasonally variable depth of approximately 6 to 12 ft bgs. The groundwater surface slopes toward the southeast with an average horizontal hydraulic gradient of approximately 0.003 ft/ft.

Through March 2003, the mulch biowall had caused measurable changes in groundwater geochemistry supportive of reductive dechlorination (Henry, 2004). Groundwater dissolved oxygen (DO) has been depleted and sulfate has been reduced. Sulfate has been reduced to 17 to 300 milligrams per liter (mg/L) with a pre-installation background level of 1,400 to 2,200 mg/L. Oxidation-reduction potential (ORP) in the biowall and immediately downgradient has been lowered to −212 millivolts (mV) to −325 mV. Methane levels in the biowall are elevated at concentrations of 7.0 to 8.8 mg/L.

The primary contaminants of concern (COCs) at the site are TCE and its daughter products. Since installation of the mulch biowall, TCE levels in the biowall have decreased by up to 98 percent to less than 2 micrograms per liter (µg/L). TCE levels downgradient of the biowall have decreased from a range of 190 to 2,500 µg/L to a range of 2.6 to 2,000 µg/L. For all monitoring locations downgradient of the biowall, the average decrease in TCE has been 60 percent. While concentrations of cis-DCE
increased in many locations over the same period, the concentration of *cis*-DCE subsequently declined without an accumulation of VC. Concentrations of *cis*-DCE range from 42 to 1,500 µg/L within and downgradient of the biowall, with the lower concentrations closer to wall. Other chlorinated solvents present are typically below drinking water standards.
Section 2
Materials and Methods

This section describes materials and methods used in support of the study tasks outlined in Figure 3.

2.1 Task 1 – Electron Shuttle Screening

Table 1 included the various electron shuttling compounds that were considered for further evaluation. Various factors including commercial availability, toxicity, water solubility, and availability of information were used to screen the compounds. Various commercial sources for potential electron shuttling compounds were identified. Table 2 lists the compounds that were evaluated in subsequent tasks and Appendix C includes additional information regarding the sources of these materials. These compounds included various humic and fulvic acid products plus pure chemical compounds. Prices for these materials were also obtained and were used to calculate unit costs that were normalized with respect to electron shuttling capacity as described in Section 3.

2.2 Task 2 – Electron Shuttling Assays

The 21 electron shuttling compounds listed in Table 2 were evaluated by an electron shuttle bioassay (Lovley, et al. 1996a; Nevin and Lovley 2002). The bioassay was developed by Dr. Derek Lovley and involves microbial reduction of the electron shuttling compounds followed by incubation of the reduced electron shuttle with ferric citrate. Ferric iron reduction to ferrous iron is measured and is used as a quantitative measurement of electron accepting capacity of the electron shuttle. Electron shuttling capacity is measured in units of millimoles per liter (mM) of iron reduced and then compared to the capacity for a well-characterized soil humic acid standard obtained from the International Humic Substances Society (IHSS). The IHSS standard is known to shuttle electrons (Lovley, et al. 1996a; Nevin and Lovley 2002). Electron shuttling capacity is then reported as a percentage of that for the IHSS material.

The humic/fulvic acids, henna products, and AQS (refer to Table 2) were evaluated at a concentration of 1 g/L. Liquid products were tested at a concentration of 1 g/L based on the humic/fulvic acid solids content in the liquid. The 15% Concentrated Liquid Humus provided by Humus Products of America was added at a concentration of 6.7% by volume to obtain an effective humic acid in the test of 1 g/L. The remaining electron shuttles (i.e., AQDS, lawson, indigo sulfonate, and menadione sodium bisulfate) were added at a concentration of 0.1 g/L. Different concentrations were selected for this test to attain an approximately equivalent economic basis. The pure chemical products are significantly more expensive than the humic/fulvic acid and henna products.
Table 2
Evaluated Electron Shuttles

<table>
<thead>
<tr>
<th>No.</th>
<th>Supplier</th>
<th>Product</th>
<th>Form</th>
<th>Humic Acid</th>
<th>Fulvic Acid</th>
<th>Source</th>
<th>Unit cost</th>
<th>Unit cost basis</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>TeraVita SP-85</td>
<td>85% soluble solid</td>
<td>80</td>
<td>5</td>
<td>Leonardite</td>
<td>$8.02</td>
<td>lb</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Luscar Ltd Dry Soluble 80</td>
<td>100% soluble solid</td>
<td>38</td>
<td>17</td>
<td>Subbituminous coal</td>
<td>$2.50</td>
<td>lb</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Luscar Ltd Liquid 12000</td>
<td>12% Liquid</td>
<td>8.3</td>
<td>3.7</td>
<td>Subbituminous coal</td>
<td>$2.65</td>
<td>gal</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Triad Huma K</td>
<td>100% soluble solid</td>
<td>56</td>
<td>30</td>
<td>Leonardite</td>
<td>$3.00</td>
<td>lb</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>Live Earth Products 12% Liquid Humic Acid</td>
<td>12% Liquid</td>
<td>10</td>
<td>2</td>
<td>Humic shale</td>
<td>$6.50</td>
<td>gal</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Monterey AgResources HA-12</td>
<td>12% Liquid</td>
<td>9</td>
<td>3</td>
<td>Leonardite</td>
<td>$7.50</td>
<td>gal</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Humate International Humate AS</td>
<td>100% soluble solid</td>
<td>75</td>
<td>25</td>
<td>Leonardite</td>
<td>$8.64</td>
<td>lb</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Humus Products of America 15% Concentrated Liquid Humus</td>
<td>15% Liquid</td>
<td>NA</td>
<td>NA</td>
<td>Leonardite</td>
<td>$11.00</td>
<td>gal</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>LignoTech BorreGro HA-2</td>
<td>100% soluble solid</td>
<td>50</td>
<td>20</td>
<td>Leonardite</td>
<td>$3.26</td>
<td>lb</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>LignoTech BorreGro HA-1</td>
<td>100% soluble solid</td>
<td>50</td>
<td>20</td>
<td>Modified leonardite</td>
<td>$2.88</td>
<td>lb</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>Horizon Ag Products Quantum H</td>
<td>7% Liquid</td>
<td>6.9</td>
<td>0.1</td>
<td>Leonardite</td>
<td>$3.75</td>
<td>gal</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>UAS America Super Hume</td>
<td>17% Liquid</td>
<td>3</td>
<td>14</td>
<td>Leonardite</td>
<td>$3.90</td>
<td>gal</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>Northwest Agricultural Products Ful-Vac 3</td>
<td>3% Liquid</td>
<td>0</td>
<td>3</td>
<td>Leonardite</td>
<td>$3.00</td>
<td>gal</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>Natural Resources Group F Power 10%</td>
<td>10% Liquid</td>
<td>0</td>
<td>10</td>
<td>Leonardite</td>
<td>$7.75</td>
<td>gal</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>Humatech Aqua F Liquid</td>
<td>0</td>
<td>3</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>Henna Global Wholesale Henna Paste</td>
<td>NA</td>
<td>NA</td>
<td>Plant origin</td>
<td>$17.27</td>
<td>lb</td>
<td></td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>Spectrum Menadione sodium bisulfite Powder</td>
<td>NA</td>
<td>NA</td>
<td>Pure chemical</td>
<td>$181.36</td>
<td>lb</td>
<td></td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>Acros Fisher 26AQDS Powder</td>
<td>NA</td>
<td>NA</td>
<td>Pure chemical</td>
<td>$127.12</td>
<td>lb</td>
<td></td>
<td></td>
</tr>
<tr>
<td>19</td>
<td>Spectrum 2AQS Powder</td>
<td>NA</td>
<td>NA</td>
<td>Pure chemical</td>
<td>$10.53</td>
<td>lb</td>
<td></td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>Spectrum Indigo disulfonate Powder</td>
<td>NA</td>
<td>NA</td>
<td>Pure chemical</td>
<td>$217.92</td>
<td>lb</td>
<td></td>
<td></td>
</tr>
<tr>
<td>21</td>
<td>Acros Fisher Lawsone Powder</td>
<td>NA</td>
<td>NA</td>
<td>Pure chemical</td>
<td>$175.26</td>
<td>lb</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Note:
NA - not applicable or available.
All tests were conducted in duplicate. Electron shuttling capacity for each compound was compared to that of a well characterized humic acid standard (i.e., International Humic Substance Society (IHSS) soil humic acids).

### 2.3 Task 3 – SSC-OTC Sample Collection and Analysis

Figure 5 shows the sample locations for soil and groundwater and Table 3 shows the analyses that were conducted on each of the samples. Soil samples were collected continuously from 10 to 25 ft bgs using direct push technology. The Unified Soil Classification System (USCS) was used to describe soil types. Lithologic observations were conducted as quickly as practicable to minimize exposure of the samples to air which would compromise the Treatability Study objectives. Composite soil samples for laboratory analysis and treatability studies were collected within the saturated zone of each borehole. Once the sample interval was retrieved, soil samples were extracted from the acetate sleeves and homogenized in a stainless steel bowl. Homogenized soil samples were then containerized in glass jars and labeled for delivery to the appropriate analytical laboratory. Groundwater from the soil boring was poured over the soil prior to sealing the jars to minimize oxygen exposure.

One depth-discrete groundwater sample was collected at each of the direct push locations correlating to the soil borings using low-flow sampling. The samples collected from the existing monitoring well 11MW07 were from an approximate depth of 15 ft bgs correlating to the approximate middle of the screened interval. Groundwater was sampled from the boreholes and well using a peristaltic pump at a flow rate of about 300 milliliters per minute (mL/min) or at a flow rate that resulted in less than 5 percent drawdown of the static water level. Field meters were used to monitor dissolved oxygen, pH, temperature, conductivity, turbidity, and oxidation-reduction potential in the groundwater and recorded on a groundwater purge and sample form. Dissolved oxygen, sulfide, total and ferrous iron measurements were conducted using field test kits.

Following purging of the boring location or monitoring well, groundwater samples were collected. Groundwater samples collected for dissolved metals analyses were filtered through a 0.45-micron filter. Unfiltered groundwater samples were collected for the remaining analyses. Groundwater samples were collected in the appropriate sampling containers with preservatives for the analyses as described in the work plan (CDM 2005). This work plan was a modification of a previous work plan that was submitted to ESTCP (CDM 2004).

Samples were analyzed for conventional parameters by Applied P & CH Laboratories in Chino, California. Bioavailable ferric iron analysis was conducted by CDM using the bioavailable ferric iron assay validated in ESTCP project number ER0009 (NAVFAC 2005).
### Table 3
Sampling and Analysis Schedule for SSC-OTC Site

<table>
<thead>
<tr>
<th>Location</th>
<th>Location Type</th>
<th>Depth</th>
<th>Sample Matrix</th>
<th>pH, Temperature, Conductivity, Turbidity, ORP, Dissolved Oxygen</th>
<th>Dissolved Oxygen, Ferrous Iron, Sulfide</th>
<th>Volatile Organic Compounds</th>
<th>Total Petroleum Hydrocarbons</th>
<th>Dissolved Fe, Mn, As, V</th>
<th>Total Fe, Mn, As, V</th>
<th>Br, Cl, NO₃, NO₂, PO₄, SO₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>11MW07</td>
<td>Existing Well New geoprobe</td>
<td>10 - 20 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP24</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>saturated soil</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP24</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP25</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>saturated soil</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP25</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP26</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>saturated soil</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP26</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP27</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>saturated soil</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP27</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>
Table 3, Continued
Sampling and Analysis Schedule for SSC-OTC Site

<table>
<thead>
<tr>
<th>Location</th>
<th>Location Type</th>
<th>Depth</th>
<th>Sample Matrix</th>
<th>Hardness</th>
<th>EPA 200.7</th>
<th>EPA 160.1</th>
<th>AM18, AM20GAX, or RSK175</th>
<th>Total Organic Carbon</th>
<th>Bioavailable Ferric Iron</th>
<th>Adsorption Treatability Study</th>
<th>Microcosm Treatability Study</th>
</tr>
</thead>
<tbody>
<tr>
<td>11MW07</td>
<td>Existing Well</td>
<td>10 - 20 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP24</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>saturated soil</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP24</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP25</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>saturated soil</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP25</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP26</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>saturated soil</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP26</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP27</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>saturated soil</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>11GP27</td>
<td>New geoprobe</td>
<td>0 - 25 feet</td>
<td>groundwater</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>
2.4 Task 4 – Precipitation and KOC Assessment

Treatability studies were conducted using the electron shuttling products listed in Table 2 in combination with soil and ground water collected from the SSC-OTC site. The studies included evaluation of the potential for product precipitation in site groundwater and development of adsorption isotherms.

Precipitation was evaluated by adding known concentrations of product to groundwater and mixing to dissolve. The solution or mixture was allowed to stand overnight and visual observations of precipitation were made.

The soil-water distribution coefficients of the electron shuttling compounds was conducted by setting up electron shuttle solutions in groundwater at concentrations of 0.00, 0.01, 0.1, 1.0, and 10 g/L. Soil was added to each of these solutions at a nominal concentration of 1 g/L. The bottles were shaken at room temperature and water samples were withdrawn and filtered after 24 and 48 hours. The electron shuttle concentration was measured spectrophotometrically. An adsorption isotherm was prepared for each electron shuttle compound based on the results.

2.5 Task 5 – Electron Shuttle Selection

Results from Tasks 1, 2, and 4 were used to identify compounds that had the lowest cost per unit electron shuttling capacity, were soluble in site groundwater, and had different KOC values. This task did not involve field sampling, analysis, or experimental work and is not described further.

2.6 Task 6 – Microcosms

Vinyl chloride transformation was measured in SSC-OTC soil and groundwater and in the presence of selected electron shuttling compounds by Drs. Paul Bradley and Frank Chapelle of the United States Geological Survey (USGS).

The products of anoxic of vinyl chloride biodegradation were investigated using [1,2-14C] VC (1.6 µCi/µmole; Perkin Elmer Life Sciences, Boston, Massachusetts). Authentic H14CO3- (Sigma Biochemicals, St. Louis, Missouri), 14CH4 (Perkin Elmer Life Sciences, Boston, Massachusetts), and [1,2-14C] ethene (Moravek Biochemicals, Inc., Brea, California) were used as radiolabeled standards for calibration and methods development. The radiochemical purity of the 14C-stocks was evaluated by direct injection radiometric detection gas chromatography (GC/RD) and found to be greater than 98% pure.

Microcosm studies were conducted as summarized below (Bradley and Chapelle 1999a, 1999b, 2000). In general, sediment microcosms were 10-mL serum vials with 5±0.5 g of saturated sediment and an atmosphere of nitrogen. Microcosms were assembled under an atmosphere of nitrogen and subsequently flushed three separate times with 100 times the headspace volume of pure nitrogen. Soil from boring 11GP27 was used for the microcosms because it was collected in the vicinity of the highest recorded cis-DCE and VC concentrations in groundwater. Groundwater was
collected from monitoring well 11MW07 because it was the only developed monitoring well in the area.

Five organic electron shuttle treatments were assessed: groundwater control, AQDS, indigo carmine, Monterey Humic Acids, and Ligno Tech Humic Acids HA-1. Each organic electron shuttle compound was assessed at three shuttle concentration treatments over a concentration range of three orders of magnitude. Table 4 presents the electron shuttle microcosm conditions evaluated in this study.

Triplicate experimental microcosms, duplicate autoclaved control microcosms, and a single sediment-free container control microcosm were prepared for each sediment treatment. All controls were autoclaved three times for 1 h at 15 psig and 121 °C. All microcosms were pre-incubated in the dark for five days prior to the addition of 14C-substrates. Microcosms were amended with 14C-VC to yield initial dissolved substrate concentrations of approximately 120 µg/L. All treatments were incubated in the dark at room temperature (23 °C) for 190 days. Anoxic conditions (headspace [O2] < 10 µM) were confirmed throughout the studies by headspace sampling and gas chromatography with thermal conductivity detection.

Headspace concentrations of CH4, 14CH4, CO2, 14CO2, ethene, 14C-ethene, ethane, 14C-ethane, VC, and 14C-VC were monitored by analyzing 0.5 ml of headspace using packed column gas chromatography with radiometric and thermal conductivity detection (Bradley and Chapelle 1999a, 1999b, 2000, 2002). The headspace sample volumes were replaced with pure nitrogen. Dissolved phase concentrations of 14C-analytes were estimated based on experimentally determined Henry’s partition coefficients. The radiometric detector was calibrated by liquid scintillation counting using H14CO3-.

2.7 Task 7 – Altus AFB Sampling

Soil and groundwater sampling at the Altus AFB was conducted by Parsons. Parsons collected samples in accordance with existing protocols for the Altus AFB. Sampling consisted of collecting soil/mulch samples from two exploratory borings within the mulch biowall and groundwater samples from monitoring wells located within, upgradient, and downgradient of the mulch biowall: OU-1-05, PES-MP-1, PES-MP-2, WL019, PES-MP-6, and PES-MP-7. Soil borings SB-1 and SB-2 were completed in the northern and southern portions of the much wall, respectively. Additional details are presented in Appendix B.
Table 4
Microcosm Set-Up

<table>
<thead>
<tr>
<th>Electron Shuttle</th>
<th>Product Concentration</th>
<th>Electron Accepting Capacity (% of IHSS Soil HA)</th>
<th>Koc (L/kg)</th>
<th>100% Baseline Conc. (g/L)</th>
<th>Shuttle (mg dry basis)</th>
<th>Added Shuttle (mg product)</th>
<th>Equilibrium Concentration (mg/L)</th>
<th>100% of Baseline Conc.</th>
<th>Equilibrium Concentration (mg/L)</th>
<th>1000% of Baseline Conc.</th>
<th>Equilibrium Concentration (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,6-Anthraquinone disulfonic acid, sodium salt (AQDS)</td>
<td>100% Solid</td>
<td>360</td>
<td>NA</td>
<td>0.21</td>
<td>0.063</td>
<td>0.063</td>
<td>NA</td>
<td>0.63</td>
<td>0.63</td>
<td>NA</td>
<td>6.3</td>
</tr>
<tr>
<td>Indigo disulfonate (Indigo Carmine aka FD&amp;C Blue #2)</td>
<td>100% Solid</td>
<td>400</td>
<td>3.5</td>
<td>0.19</td>
<td>0.057</td>
<td>0.057</td>
<td>2.8</td>
<td>0.57</td>
<td>0.57</td>
<td>28</td>
<td>5.7</td>
</tr>
<tr>
<td>Monterey Ag Products HA-12 12% humic acid</td>
<td>12% wt solution</td>
<td>49</td>
<td>50</td>
<td>1.6</td>
<td>0.46</td>
<td>3.9</td>
<td>1.8</td>
<td>4.6</td>
<td>39</td>
<td>18</td>
<td>46</td>
</tr>
<tr>
<td>LignoTech Borregro HA-1 (Sulfonated Humic acid)</td>
<td>100% Solid</td>
<td>14</td>
<td>5.8</td>
<td>5.4</td>
<td>1.6</td>
<td>1.6</td>
<td>51</td>
<td>16.2</td>
<td>16.2</td>
<td>506</td>
<td>162</td>
</tr>
</tbody>
</table>

Notes:

a) In the case of humic acid solutions, activity is based on humic acid mass, not on solution mass. IHSS soil humic acids had 100% activity by definition.
b) Calculated amount of electron shuttle product (solid or liquid) to be added per microcosm. Based on equal electron accepting capacity per bottle.
NA - not applicable or available.
HA - humic acid.
IHSS - International Humic Substance Society.
Koc - organic carbon partition coefficient.
g/L - grams per liter.
L/kg - liters per kilogram.
mg - milligrams.
2.8 Task 8 – Altus AFB Sample Analysis

Analyses of biowall materials included:

- Total organic carbon (TOC) (Method SW9060)
- Volatile organic compounds (VOCs) (EPA Method 8260B)
- Total sulfide, iron (II) sulfide, and iron monosulfide (Microseeps)
- Bioavailable ferric iron (New Horizon Diagnostics Corp. test kit)
- Electron shuttle bioassay (University of Massachusetts)

Analyses of groundwater samples included:

- Total organic carbon (TOC) (Method SW9060)
- Volatile organic compounds (VOCs) (EPA Method 8260B)
- Methane, ethane, and ethene (AM-20GAX)
- Nitrate plus nitrite (EPA 353.1)
- Sulfate and chloride (EPA 300.1)
- Volatile fatty acids (Microseeps)
- Total dissolved solids (EPA 160.3)
- Hardness (EPA 130.2)
- Field measurements including pH, temperature, conductivity, turbidity, oxidation-reduction potential, dissolved oxygen, and ferrous iron, hydrogen sulfide, and alkalinity

In addition, the biowall samples were analyzed for electron shuttling capacity at the University of Massachusetts using procedures described in Section 2.2. The samples were tested at a concentration of 1 g/L.

Additional details are presented in Appendix B.
Section 3
Results and Discussion

3.1 SSC-OTC Sampling and Analysis

Table 5 summarizes analytical results for soil and groundwater samples collected at the SSC-OTC Site in accordance with Task 3. Appendices D and E include soil boring logs and analytical data reports, respectively.

Table 5
SSC-OTC Results

<table>
<thead>
<tr>
<th>Analyte in Groundwater</th>
<th>11GP24</th>
<th>11GP25</th>
<th>11MW07</th>
<th>11GP26</th>
<th>11GP27</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tetrachloroethene (µg/L)</td>
<td>&lt;25</td>
<td>&lt;5</td>
<td>&lt;0.5</td>
<td>&lt;25</td>
<td>&lt;13</td>
</tr>
<tr>
<td>Trichloroethene (µg/L)</td>
<td>&lt;25</td>
<td>&lt;5</td>
<td>&lt;0.5</td>
<td>&lt;25</td>
<td>&lt;13</td>
</tr>
<tr>
<td>cis-1,2-Dichloroethene (µg/L)</td>
<td>8,670</td>
<td>272</td>
<td>1,000</td>
<td>3,140</td>
<td>4,510</td>
</tr>
<tr>
<td>Vinyl Chloride (µg/L)</td>
<td>1,520</td>
<td>174</td>
<td>613</td>
<td>935</td>
<td>2,700</td>
</tr>
<tr>
<td>Ethene (µg/L)</td>
<td>31</td>
<td>28</td>
<td>130</td>
<td>28</td>
<td>89</td>
</tr>
<tr>
<td>Dissolved Oxygen (mg/L)</td>
<td>0.6</td>
<td>1</td>
<td>0.6</td>
<td>0.4</td>
<td>0.6</td>
</tr>
<tr>
<td>Nitrate (mg-N/L)</td>
<td>0.14</td>
<td>0.068</td>
<td>J</td>
<td>0.21</td>
<td>0.59</td>
</tr>
<tr>
<td>Manganese (mg/L)</td>
<td>0.408</td>
<td>0.504</td>
<td>0.561</td>
<td>0.461</td>
<td>0.461</td>
</tr>
<tr>
<td>Iron (mg/L)</td>
<td>2.11</td>
<td>2.3</td>
<td>3.97</td>
<td>2.8</td>
<td>0.0346 J</td>
</tr>
<tr>
<td>Sulfate (mg/L)</td>
<td>557</td>
<td>690</td>
<td>590</td>
<td>560</td>
<td>388</td>
</tr>
<tr>
<td>Sulfide (mg/L)</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;1</td>
<td>&lt;0.11</td>
</tr>
<tr>
<td>Methane (µg/L)</td>
<td>160</td>
<td>99</td>
<td>140</td>
<td>160</td>
<td>200</td>
</tr>
<tr>
<td>Oxidation-Reduction Potential (mV)</td>
<td>-236</td>
<td>-221</td>
<td>-172</td>
<td>-212</td>
<td>-80</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Analyte in Soil</th>
<th>11GP24 (10-20 ft)</th>
<th>11GP25 (10-22 ft)</th>
<th>11GP26 (14-22 ft)</th>
<th>11GP27</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Organic Carbon avg. (mg/kg)</td>
<td>400</td>
<td>2970</td>
<td>660</td>
<td>636</td>
</tr>
<tr>
<td>Iron (mg/kg)</td>
<td>9,830</td>
<td>11,000</td>
<td>6,230</td>
<td>4,300</td>
</tr>
<tr>
<td>Bioavailable Ferric Iron (mg/kg)</td>
<td>1,400 (910)</td>
<td>1,700 (910)</td>
<td>1,300 (1,500)</td>
<td>630 (630)</td>
</tr>
</tbody>
</table>

Notes:
Numbers in parentheses are duplicate results.
µg/L - micrograms per liter.
mg/L - milligrams per liter.
mg-N/L - milligrams nitrogen per liter.
mV - millivolts.
mg/kg - milligrams per kilogram.
J - estimated value.
< - analyte not detected at or greater than the listed concentration.
ft - feet below ground surface

Groundwater concentrations of cis-DCE and VC increased along the groundwater flow path from 11GP25 to 11GP27. PCE and TCE concentrations were below detection limits and cis-DCE and VC concentrations were elevated (e.g., up to 8,670 µg/L cis-DCE and up to 2,700 µg/L VC) indicating that complete reductive dechlorination was not occurring. Additionally, dissolved ethene concentrations were low further indicating that complete reductive dechlorination may not be occurring.
Dissolved oxygen and nitrate concentrations were generally low. Dissolved iron concentrations ranged from 2 to 4 mg/L except at 11GP27 that had an estimated concentration of 0.0346 mg/L. Sulfate concentrations were high (e.g., up to 690 mg/L), methane concentrations were relatively low (e.g., up to 200 µg/L), and the oxidation-reduction potential was low (e.g., ~236 mV) suggesting that sulfate reduction may be occurring. However, sulfide was not detected. Thus the aquifer appeared to be predominately iron-reducing but could have been sulfate reducing. Significant bioavailable ferric iron was also present in site soil to support further iron reduction.

3.2 Altus AFB Sampling and Analysis

Parsons conducted the sampling and analysis of the Altus AFB biowall as part of Tasks 7 and 8; however, ER0316 was cancelled prior to completion of any data development and interpretation. The following data summary is presented for informational purposes:

- In the northern flow path, TCE, cis-DCE, VC, and ethene concentrations were 1,500, 550, <100, 0.071 µg/L upgradient of the biowall, respectively. The concentrations of these constituents in the biowall were and <12, 69, 590, and 3.3 µg/L, respectively. This change reflects a 41 percent reduction in total molar concentration (non-detected concentrations were assumed to be half the practical quantitation limit).

- In the southern flow path, TCE, cis-DCE, VC, and ethene concentrations were 74, 73, <2.5, 0.036 µg/L upgradient of the biowall, respectively. The concentrations of these constituents in the biowall were 5.8, 85, 3.0, and 0.27 µg/L, respectively. This change reflects a 26 percent reduction in total molar concentration (non-detected concentrations were assumed to be half the practical quantitation limit).

- Relative electron shuttling capacity ranged from 0 to 79 percent of the 0.5 g/L humic acids standard. The relative electron shuttling capacity in the northern flow path biowall samples were greater than that of the southern flow path samples. The relative electron shuttling capacity of the northern flow path samples ranged from 47 to 79 percent. The relative electron shuttling capacity of the southern flow path biowall samples ranged from 0 to 33 percent.

- Bioavailable ferric iron (BAFeIII) ranged from <6.4 to 650 mg/kg. BAFeIII in the northern flow path biowall samples were greater than that of the southern flow path samples. BAFeIII of the northern flow path samples ranged from 440 to 650 mg/kg. BAFeIII of the southern flow path biowall samples ranged from <6.4 to 290 mg/kg.

A complete data report is presented in Appendix B.
3.3 Electron Shuttle Screening and Characterization

Table 6 presents characterization results for the various electron shuttle products. The results presented in Table 6 are based on results from Tasks 1, 2, and 4. Economic comparisons are also included in this table. Products 1 through 16 are different humic acid or fulvic acid products. Products 17 through 21 are pure chemicals. The commercial prices and the vendor-reported humic acid/fulvic acid contents or chemical purities were used to calculate the cost per pound of reported active material. The humic acid/fulvic acid products ranged from $3 to $17/lb of reported active material. The pure products ranged from $127 to $313/lb of reported active material with the exception of AQS which was $11/lb.

Aldrich humic acids were not evaluated because Sigma-Aldrich was not willing to sell the product for injection into groundwater due to liability concerns. Specifically, they stated (email from John Green, July 22, 2002):

*We cannot sell this material for your application due to concerns by our company of possible issues that may arise with its use in the environment. This is not to say that the material could not be used in a safe manner that would cause no effects on the environment, but Sigma-Aldrich does not feel that it is in our best interest to sell this material to you for your intended use at this time.*

While Aldrich humic acids have been used extensively in laboratory studies, their commercial unavailability is an important factor to consider when translating academic findings into field applications.

The reported humic acid content is not necessarily a good indicator of electron shuttling capacity. Electron shuttling capacity is defined as the specific ability of a compound to be reduced by iron-reducing bacteria and, in turn, oxidize ferric ion to ferrous ion. A specialized analysis for “electron shuttling capacity” has been developed by the University of Massachusetts as was conducted under Task 2. The electron shuttling capacity for each compound was determined and compared to a humic acid standard. The relative activity (i.e., relative to the humic acid standard) for products 1 through 16 ranged from 6 to 40%. Menadione sodium bisulfate (Product 17) showed no activity. This product is the soluble version of vitamin K3 which is known to have electron shuttling ability. Thus, bisulfate modification resulted in loss of activity. AQS and lawsone (Products 19 and 21) were similar to the humic acid/fulvic acid products with relative activities of 20% and 41%, respectively. AQDS had a very high relative activity of 362% as expected. Unexpectedly, indigo disulfonate had the highest relative activity at 399%. This product is a registered food colorant (FD&C Blue Number 2 also known as indigo carmine) and thus would appear to have good potential for use as an electron shuttle because of high electron shuttling capacity and safety.
### Table 6: Electron Shuttle Results

<table>
<thead>
<tr>
<th>No.</th>
<th>Supplier</th>
<th>Product</th>
<th>Form</th>
<th>Humic Acid (%)</th>
<th>Fulvic Acid (%)</th>
<th>Unit cost</th>
<th>Specific Cost ($/lb reported active material)</th>
<th>Specific cost basis</th>
<th>Shuttling Capacity % of 0.5 g/L IHSS Soil Humic Acid standard</th>
<th>Relative activity (to IHSS humic acids)</th>
<th>Normalized cost ($/lb electron shuttling compounds)</th>
<th>Koc (L/kg)</th>
<th>Tested Conc. in Groundwater (g/L)</th>
<th>Soluble in Groundwater at Tested Conc.?</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>TeraVita</td>
<td>SP-85</td>
<td>85% soluble solid</td>
<td>80</td>
<td>5</td>
<td>$8.02</td>
<td>$10</td>
<td>lb</td>
<td>HA 79</td>
<td>40%</td>
<td>$20</td>
<td>48</td>
<td>1.1</td>
<td>N</td>
</tr>
<tr>
<td>2</td>
<td>Luscar Ltd</td>
<td>Dry Soluble 80</td>
<td>100% soluble solid</td>
<td>38</td>
<td>17</td>
<td>$2.50</td>
<td>$7</td>
<td>lb</td>
<td>HA 76</td>
<td>38%</td>
<td>$7</td>
<td>75</td>
<td>0.5</td>
<td>N</td>
</tr>
<tr>
<td>3</td>
<td>Luscar Ltd</td>
<td>Liquid 12000</td>
<td>12% Liquid</td>
<td>8.3</td>
<td>3.7</td>
<td>$2.65</td>
<td>$4</td>
<td>lb</td>
<td>HA 59</td>
<td>30%</td>
<td>$9</td>
<td>120</td>
<td>1.0</td>
<td>N</td>
</tr>
<tr>
<td>4</td>
<td>Triad</td>
<td>Humic K</td>
<td>100% soluble solid</td>
<td>56</td>
<td>30</td>
<td>$3.00</td>
<td>$5</td>
<td>lb</td>
<td>HA 63</td>
<td>32%</td>
<td>$9</td>
<td>61</td>
<td>0.7</td>
<td>N</td>
</tr>
<tr>
<td>5</td>
<td>Live Earth Products</td>
<td>Liquid Humic Acid</td>
<td>12% Liquid</td>
<td>10</td>
<td>2</td>
<td>$6.50</td>
<td>$7</td>
<td>gal</td>
<td>HA 76</td>
<td>38%</td>
<td>$17</td>
<td>87</td>
<td>1.2</td>
<td>N</td>
</tr>
<tr>
<td>6</td>
<td>Monterey Ag Resources</td>
<td>HA-12</td>
<td>12% Liquid</td>
<td>9</td>
<td>3</td>
<td>$7.50</td>
<td>$10</td>
<td>gal</td>
<td>HA 98</td>
<td>49%</td>
<td>$15</td>
<td>50</td>
<td>1.0</td>
<td>Y</td>
</tr>
<tr>
<td>7</td>
<td>Humate International</td>
<td>Humate AS</td>
<td>100% soluble solid</td>
<td>75</td>
<td>25</td>
<td>$8.64</td>
<td>$12</td>
<td>lb</td>
<td>HA 57</td>
<td>28%</td>
<td>$30</td>
<td>NT</td>
<td>0.6</td>
<td>N</td>
</tr>
<tr>
<td>8</td>
<td>Humus Products of America</td>
<td>15% Concentrated Liquid Humus</td>
<td>15% Liquid</td>
<td>NA</td>
<td>NA</td>
<td>$11.00</td>
<td>NA</td>
<td>gal</td>
<td>NA</td>
<td>HA 51</td>
<td>25%</td>
<td>$35</td>
<td>66</td>
<td>1.0</td>
</tr>
<tr>
<td>9</td>
<td>LignoTech</td>
<td>BorréGro HA-2</td>
<td>100% soluble solid</td>
<td>50</td>
<td>20</td>
<td>$3.26</td>
<td>$6</td>
<td>lb</td>
<td>HA 55</td>
<td>28%</td>
<td>$12</td>
<td>96</td>
<td>1.2</td>
<td>N</td>
</tr>
<tr>
<td>10</td>
<td>LignoTech</td>
<td>BorréGro HA-1</td>
<td>100% soluble solid</td>
<td>50</td>
<td>20</td>
<td>$2.88</td>
<td>$7</td>
<td>lb</td>
<td>HA 27</td>
<td>14%</td>
<td>$21</td>
<td>5.8</td>
<td>1.1</td>
<td>Y</td>
</tr>
<tr>
<td>11</td>
<td>Horizon Ag Products</td>
<td>Quantum H</td>
<td>7% Liquid</td>
<td>6.9</td>
<td>0.1</td>
<td>$3.75</td>
<td>$6</td>
<td>gal</td>
<td>HA 68</td>
<td>34%</td>
<td>$19</td>
<td>NT</td>
<td>0.5</td>
<td>N</td>
</tr>
<tr>
<td>12</td>
<td>UAS America</td>
<td>Super Hume</td>
<td>17% Liquid</td>
<td>3</td>
<td>14</td>
<td>$3.90</td>
<td>$3</td>
<td>gal</td>
<td>FA 36</td>
<td>18%</td>
<td>$15</td>
<td>76</td>
<td>0.5</td>
<td>Y</td>
</tr>
<tr>
<td>13</td>
<td>Northwest Agricultural Products</td>
<td>Ful-Vac 3</td>
<td>3% Liquid</td>
<td>0</td>
<td>3</td>
<td>$3.00</td>
<td>$11</td>
<td>gal</td>
<td>FA 16</td>
<td>8%</td>
<td>$153</td>
<td>NT</td>
<td>0.5</td>
<td>Y</td>
</tr>
<tr>
<td>14</td>
<td>Natural Resources Group</td>
<td>F Power 10%</td>
<td>10% Liquid</td>
<td>0</td>
<td>10</td>
<td>$7.75</td>
<td>$9</td>
<td>gal</td>
<td>FA 12</td>
<td>6%</td>
<td>$159</td>
<td>NT</td>
<td>1.0</td>
<td>N</td>
</tr>
<tr>
<td>15</td>
<td>Humatech</td>
<td>Aqua F</td>
<td>Liquid</td>
<td>0</td>
<td>3</td>
<td>NA</td>
<td>NA</td>
<td>NA</td>
<td>HA 17</td>
<td>9%</td>
<td>NA</td>
<td>NT</td>
<td>NA</td>
<td>NA</td>
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<tr>
<td>16</td>
<td>Henna Global Wholesale</td>
<td>Henna Paste</td>
<td>Powder</td>
<td>NA</td>
<td>NA</td>
<td>$17.27</td>
<td>$17</td>
<td>lb</td>
<td>NA</td>
<td>19%</td>
<td>$91</td>
<td>NT</td>
<td>0.6</td>
<td>N</td>
</tr>
<tr>
<td>17</td>
<td>Spectrum</td>
<td>Menadione Sodium Bisulfite</td>
<td>Powder</td>
<td>NA</td>
<td>NA</td>
<td>$181.36</td>
<td>$181</td>
<td>Chemical</td>
<td>NA</td>
<td>0%</td>
<td>NA</td>
<td>NT</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>18</td>
<td>Acros Fisher</td>
<td>AQDS</td>
<td>Powder</td>
<td>NA</td>
<td>NA</td>
<td>$127.12</td>
<td>$127</td>
<td>Chemical</td>
<td>NA</td>
<td>0%</td>
<td>NA</td>
<td>NT</td>
<td>NA</td>
<td>NA</td>
</tr>
<tr>
<td>19</td>
<td>Spectrum</td>
<td>AQS</td>
<td>Powder</td>
<td>NA</td>
<td>NA</td>
<td>$10.53</td>
<td>$11</td>
<td>Chemical</td>
<td>NA</td>
<td>20%</td>
<td>$53</td>
<td>NT</td>
<td>0.6</td>
<td>N</td>
</tr>
<tr>
<td>20</td>
<td>Spectrum</td>
<td>Indigo Disulfonate</td>
<td>Powder</td>
<td>NA</td>
<td>NA</td>
<td>$217.92</td>
<td>$218</td>
<td>Chemical</td>
<td>NA</td>
<td>399%</td>
<td>$55</td>
<td>3.5</td>
<td>0.6</td>
<td>Y</td>
</tr>
<tr>
<td>21</td>
<td>Acros Fisher</td>
<td>Lawsons</td>
<td>Powder</td>
<td>NA</td>
<td>NA</td>
<td>$175.26</td>
<td>$313</td>
<td>Chemical</td>
<td>NA</td>
<td>41%</td>
<td>$424</td>
<td>NT</td>
<td>NA</td>
<td>NA</td>
</tr>
</tbody>
</table>

**Notes:**
- NA - not applicable or available.
- NT - not tested.
- HA - humic acid.
- FA - fulvic acid.
- IHSS - International Humic Substance Society.
- Koc - organic carbon partition coefficient.
- g/L - grams per liter.
- lb - pounds.
- L/kg - liters per kilogram.
The electron shuttling capacity was used to calculate the unit cost of each product. These unit costs are reported in units of dollars per pound of electron shuttling compounds and is defined as the actual product cost divided by the relative activity (to IHSS humic acids). The results indicate humic acid Products 1 to 12 ranged in unit cost from $7 to $21/lb and the two fulvic acid products were $153 and $159/lb. Henna was $91/lb. The pure products AQDS, AQS, and indigo disulfonate were slightly more expensive than the humic acid products and ranged from $35 to $55/lb. Lawuese was much more expensive with a unit cost of $424/lb.

The humic acid products varied in unit price by a factor of three following normalization with respect to electron shuttling activity. AQDS and indigo disulfonate were more expensive than the humic acids even though they had a very high specific activity. Nevertheless, further evaluation of indigo disulfonate was warranted because of its registration as an FD&C food colorant. Additionally, its high specific activity relative to IHSS humic acids indicates that less compound would need to be injected into the ground.

The soil-water partitioning coefficient (KOC) is important with respect to transport of compounds through an aquifer. The soil-water partitioning coefficients and the qualitative solubility of these compounds were evaluated as part of Task 4 using soil and groundwater collected from the SSC-OTC Site as part of Task 3. The KOC values of the humic acid products ranged from 48 to 120 L/kg with the exception of Product 10 which is a sulfonated derivative of humic acids and had a KOC of 5.8 L/kg. Indigo disulfonate was similar with a Koc of 3.5 L/kg. Supporting data are presented in Appendix F.

While many of the humic acid products were reported to be 10% soluble, many were observed to partially precipitate in Site groundwater. This finding indicates that commercial products should be evaluated for compatibility with Site groundwater to ensure that precipitation does not occur following injection.

### 3.4 Electron Shuttle Selection

Based on the results presented in Section 3.3 in accordance with Task 5, Product 6 (Monterey Ag-Resources HA-12 humic acids), Product 10 (LignoTech Borregro HA-1 sulfonated humic acids), and Product 20 (indigo disulfonate) were selected for evaluation in the vinyl chloride microcosm study. Product 6 was selected as a standard humic acid that remained in solution in groundwater. Product 10 was selected because of its lower KOC value. Product 20 was selected because it is a non-humic acid product that has a high electron shuttling capacity. Product 18 (AQDS) was also used for comparison because of its previously demonstrated ability to shuttle electrons and promote vinyl chloride transformation (Bradley, et al. 1998).

### 3.5 Microcosm Results

Task 6 was conducted by USGS (Drs. Frank Chapelle and Paul Bradley) and demonstrated little to no effect of electron shuttles on oxidative or reductive VC
biodegradation. Analysis of select microcosms from each treatment was performed at approximately 30 days incubation to determine if a complete time-point assessment was appropriate. No evidence of $^{14}$C-VC loss or accumulation of $^{14}$C-products was observed in any treatment. It was concluded that the biodegradation activity was low or insignificant and a full time-point analysis was not appropriate.

A complete time point assessment was conducted after approximately 95 days of incubation. No evidence of significant $^{14}$C-VC loss or accumulation of $^{14}$C-products was observed. It was again concluded that oxidative or reductive biodegradation activity was either absent or low in this sediment under these incubation conditions. It was decided to conduct another full time-point at approximately 200 days and reassess the continuation of the microcosm study.

A complete time-point was conducted after approximately 190 days of incubation and results are summarized in Table 7. Figure 6 shows photographs of selected microcosm bottles. Significant degradation activity was observed in four of nine experimental (live treatment) groundwater control (no electron shuttle compounds added) microcosms after 190 days of incubation. $^{14}$C-VC loss in these four microcosms was 82±3 percent. The activity not recovered as $^{14}$C-VC was entirely associated with $^{14}$C-ethene. This indicated that reductive dechlorination of VC was the significant degradation process in these sediments in the absence of electron shuttle addition. It should be noted, however, that no significant loss of $^{14}$C-VC or accumulation of $^{14}$C-products was observed in five of the nine experimental water control microcosms. The mean $^{14}$C-VC loss for all nine experimental water control microcosms was 37±43 percent. No evidence of $^{14}$C-VC loss or accumulation of $^{14}$C-products was observed in autoclaved sediment control or sediment free control microcosms prepared with water only. Based on these results, it was concluded that a potential for VC biodegradation was present in Site sediments, but that the activity was low. Under these laboratory conditions, biodegradation activity was associated with reductive dechlorination to ethene.

Table 7
Microcosm Results

<table>
<thead>
<tr>
<th>Condition</th>
<th>VC loss</th>
<th>Ethene Prod.</th>
<th>CO$_2$ Prod.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Groundwater only control</td>
<td>82%</td>
<td>82%</td>
<td>0%</td>
</tr>
<tr>
<td>(4 out of 9 bottles showed activity)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AQDS – high conc. (1 out of 3 bottles showed activity)</td>
<td>100%</td>
<td>0%</td>
<td>100%</td>
</tr>
<tr>
<td>AQDS – medium conc.</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
</tr>
<tr>
<td>AQDS – low conc.</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
</tr>
<tr>
<td>Humic acid (all conc.)</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
</tr>
<tr>
<td>Sulfonated humate (all conc.)</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
</tr>
<tr>
<td>Indigo carmine (all conc.)</td>
<td>0%</td>
<td>0%</td>
<td>0%</td>
</tr>
</tbody>
</table>
No evidence of $^{14}$C-VC loss or accumulation of $^{14}$C-products was observed in experimental (live), autoclaved control or sediment free control microcosms at any shuttle concentration in those treatments prepared with Monterey humic acids, LignoTech sulfonated humic acids, or indigo carmine. This result is significant, as it suggests that addition of these electron shuttles inhibits the reduction of $^{14}$C-VC to $^{14}$C-ethene that was observed in some of the groundwater only control microcosms. This is consistent with the hypothesized oxidizing effect of the addition of oxidized electron shuttle compounds to otherwise reduced sediments. However, no evidence of biodegradation of $^{14}$C-VC to $^{14}$CO$_2$ was observed under these shuttle amended conditions after 190 days incubation.

No evidence of $^{14}$C-VC loss or accumulation of $^{14}$C-products was observed in experimental (live) microcosms prepared with AQDS at either low or medium concentrations. Complete $^{14}$C-VC loss was observed in one of three experimental (live) microcosms prepared with high concentrations of AQDS. In this treatment, $^{14}$C-radioactivity was recovered entirely as $^{14}$CO$_2$. The mean ± standard deviation (SD) for $^{14}$C-VC loss when considering all three experimental (live) microcosms in the high AQDS treatment was 36±56 percent. No evidence of $^{14}$C-VC loss or accumulation of $^{14}$C-products was observed in autoclaved or sediment free control treatments at any AQDS treatment level.

The AQDS results indicated that high concentrations of AQDS might eventually promote $^{14}$C-VC degradation to $^{14}$CO$_2$. The fact that $^{14}$C-VC biodegradation was dominated by reduction to $^{14}$C-ethene in the absence of AQDS addition, however, indicated that a significant reductive capacity existed in these sediments. This reductive capacity would need to be overcome before oxidative degradation of VC would be expected to become significant. If this is the case then the onset of oxidative degradation would be expected to occur first or, perhaps, only under relatively high concentrations of AQDS. The oxidation of $^{14}$C-VC to $^{14}$CO$_2$ in one of three experimental microcosms at high AQDS concentrations is consistent with this hypothetical relationship, but does not constitute statistically significant or compelling evidence for this process.
These data indicate that the known electron shuttle was not capable of consistently promoting vinyl chloride oxidation with SSC-OTC Site soil and groundwater after 190 days. Additionally, the commercially available electron shuttle products did not promote vinyl chloride oxidation. Whether these compounds are capable of promoting this activity at other sites is unknown. Nevertheless, use of electron shuttles for promoting oxidative transformation of VC does not appear to be warranted for the Site groundwater.
Section 4
Conclusions

Fifteen commercially available humic/fulvic acid products were tested, shown to be capable of being microbically reduced by iron-reducing bacteria, and in turn being able to transfer these electrons to ferric citrate. The relative electron shuttling capacity of these compounds varied from 6 to 49 percent of the electron shuttling capacity for a humic acids standard. Fulvic acids had a lower electron shuttling capacity than humic acids as expected. Humic acid compounds varied with respect to adsorption to soil and had KOC values that ranged from 5.8 to 120 L/kg. The product having the lowest KOC was a sulfonated humic acid derivative. These compounds varied with respect to remaining in solution in groundwater – only four of fourteen products tested did not precipitate when added to site groundwater. Therefore, commercial humic acid products should always be tested with site-specific groundwater prior to use because of the potential for precipitation.

Six non-humic acid products were also tested and indigo disulfonate, also known as indigo carmine or FD&C Blue number 2 was found to be particularly effective with respect to electron shuttling. This relative electron shuttling capacity of this compound was 400 percent of the humic acid standard. The other five compounds ranged in relative electron shuttling capacity from 0 to 360 percent.

Three products were tested for their ability to promote anaerobic oxidation of radiolabeled vinyl chloride. These products included a commercial humic acid product (Monterey Ag Resources HA-12), a sulfonated humic acid (LignoTech BorreGro HA-1), and indigo disulfonate. The products were selected based on the above criteria and cost. Cost was normalized per unit of electron shuttling capacity. The normalized costs ranged from $7 to $420 per pound of electron shuttling compounds. The normalized costs of the three selected products were $15, $21, $55 per pound of electron shuttling compounds, respectively. The least expensive products were not selected for testing because they precipitated when mixed with groundwater.

None of these three products were capable of promoting vinyl chloride oxidation to carbon dioxide or reduction to ethene in groundwater and soil from the Space and Naval Warfare Systems Old Town Campus (SSC-OTC) Site in San Diego, California. The positive control with the known electron shuttling compound AQDS was also only able to promote vinyl chloride oxidation to carbon dioxide in one of three microcosms and only at the highest concentration of AQDS. Four out of nine microcosms containing soil and groundwater only demonstrated reductive dechlorination of vinyl chloride to ethane. These data indicate that electron shuttles, whether they are known compounds that have been tested in the laboratory (i.e., AQDS) or products with demonstrated electron shuttling capacity, are not necessarily capable of promoting consistent anaerobic oxidation of vinyl chloride to carbon dioxide.
Sampling and analysis of the Altus AFB biowall was conducted; however, the project was cancelled prior to completion of any data evaluation and interpretation. Reductions in total molar chlorinated ethene concentrations were somewhat greater in along the northern flow path relative to the southern flow path (i.e., 41 versus 26 percent). Biowall samples collected from the northern flow path also had greater electron shuttling capacity and bioavailable ferric iron concentrations relative to those collected from the southern flow path. Whether these biowall characteristics caused the difference in contaminant reduction cannot be determined at this time and may warrant additional research.

Other destruction mechanisms in biowalls including biogeochemical transformation are also relevant and warrant additional research. A workshop on *in situ* biogeochemical transformation of chlorinated solvents was held in 2008 (AFCEE 2008). Based on the key issues identified in the workshop, research and demonstration needs were identified. In total, seven research and two demonstration needs were identified, with most of them focused on improving the fundamental understanding of mechanisms and processes that contribute to chlorinated solvent degradation. These research and demonstration need topics include:

- Biogeochemical mechanisms of chlorinated solvent degradation.
- Geochemical and microbiological requirements for formation of active mineral phases.
- Sampling and analysis requirements and protocols for characterization and monitoring of *in situ* biogeochemical transformation.
- Geochemical modeling for predicting the development, effectiveness, and sustainability of *in situ* biogeochemical transformation.
- Data mining, sampling, analysis, and geochemical modeling of existing sites to facilitate understanding of *in situ* biogeochemical transformation in the field.
- Methods for selection of *in situ* amendments to promote formation of biogeochemically active mineral phases.
- Applicability of *in situ* biogeochemical transformation to other contaminants.
- Demonstration of a sampling and analysis protocol for characterization and monitoring of *in situ* biogeochemical transformation in the field.
- Pilot-scale demonstrations with selected amendments that promote development of *in situ* biogeochemical transformation.

This research and development will facilitate application of *in situ* biogeochemical transformation processes and result in more cost-effective ways of mitigating the risks that are associated with inorganic and organic contamination of soil, groundwater, and sediment.
Section 5
Acknowledgements

This report was written and edited by Dr. Patrick Evans of CDM and Carmen Lebrón of NAVFAC’s Engineering Service Center respectively. Ms. Carmen Lebrón of NAVFAC ESC was the Project Manager. Funding from the Department of Defense Environmental Security Technology Certification Program (ESTCP) is gratefully acknowledged and appreciated. The support of Mr. Alan Vancil at SSC-OTC is also appreciated. Ms. Kelly Nevin conducted electron shuttle assays in the laboratory of Dr. Derek Lovley. Drs. Paul Bradley and Frank Chapelle of the U.S. Geological Survey conducted the radiolabeled vinyl chloride microcosm study. Parsons conducted sampling and analysis at Altus AFB.
Section 6
References


CDM. 2005. Final Treatability Study Work Plan for ESTCP Project No. CU-0316: Enhanced Oxidative Bioremediation of cis-Dichloroethene (cis-DCE) and Vinyl Chloride (VC) using Electron Shuttles at Installation Restoration Site 11, Space and
Naval Warfare Systems Center, Old Town Campus, San Diego, California. April 29, 2005.


Appendix A
Electron Shuttle Toxicology
Appendix A
Electron Shuttle Toxicology

Electron shuttles are chemicals that assist in electron transfer from recalcitrant environmental chemicals to electron acceptors. They can be used to encourage the oxidation of chemicals such as vinyl chloride as part of subsurface bioremediation efforts. Since these chemicals must be released into the subsurface and could migrate to and with groundwater, they may, in theory, represent a potential environmental hazard. This white paper discusses the toxicology of three common electron shuttles, humic acids, indigo carmine and 2,6-anthraquinone disulfonic acid (AQDS). The discussions are intended to provide insight into the hazards, if any, that release of these chemicals during remediation may represent.

Humic Acids
The following assessment of toxicology of humic acids was produced using information obtained from a number of sources including the open literature, regulatory evaluations, toxicity test reports and others. The information retrieved is not exhaustive, but should be representative of the types of studies available and the basic toxicity of humic acids.

Humic acids are a group of polymeric polyhydroxy acids that occur naturally as degradation products of plant materials. They occur in most surface waters, including rivers and streams, ponds and lakes and estuaries and oceans. Humic acids vary depending on source and their chemical composition and structure are only generally known. They are characterized by molecular weights between 2,000 and 50,000 Daltons and solubility in weak alkali but not weak acid. Much commercially available humic acid is extracted from bituminous (brown) coal as the alkali soluble organic fraction (EPA 2000, Federal Register, July 18, Vol 65, Number 138).

Toxicity of humic acids has not been well defined in typical toxicity studies. Thus, toxicity must be addressed by considering chemical properties and toxicokinetics, limited in vitro and animal studies, and information from environmental exposures and agricultural uses.

Chemical Properties and Toxicokinetics
Humic acids are characteristically high molecular weight, polar chemicals. These characteristics suggest that these acids will be very poorly absorbed from the GI tract. Studies in which humic acids were administered to rodents indicated that stools turned black after a single high dose, suggesting that much of the chemical had passed through the digestive tract. Lack of absorption following oral exposure would limit any direct toxicity of humic acids to the gastrointestinal tract. However, as discussed below, even high doses of humic acids did not appear to cause any GI distress after short-term exposures.
Humic acids may act as chelators of metals, probably through binding to acid groups, although both N and S containing groups might also be involved. Chelation could, in theory, either increase or ameliorate toxicity due to heavy metals. In several studies, as summarized below, humic acids have decreased metals toxicity. Lack of long term exposure studies, however, makes definitive conclusions difficult.

**In Vitro and Animal Toxicity Testing**

Toxicity information on humic acids is limited, but does suggest that significant toxicity is not expected. In single dose toxicity tests, administration of 5 g of humic acids per kilogram to rats did not cause any noticeable toxicity and necropsy after 14 days did not identify any gross anatomical changes (LignoTech USA, Inc 1996, Product Safety Laboratories 2002). Administration of this single dose did not produce any noticeable GI distress (e.g., diarrhea). Similar results were observed in studies in which animals were fed for up to 90 days (Daniel, et al. 1992). No obvious toxicity was observed and no gross pathology was reported. Acute to subchronic exposure, even at doses in the range of a few to several grams per kilogram, does not appear to produce adverse effects.

Several studies have reported a protective effect of humic acids for aquatic and terrestrial plant test organisms and for earthworms exposed to toxic metals. For example, Cu toxicity to Zea mays was decreased when humic acids were added to test water (Ullah and Gerzabek 1991). This effect is likely due to the ability of humic acid to chelate metals and thereby make them less bioavailable.

In vitro, humic acids can produce toxicity to cultured cells that is consistent with production of free radicals (Cheng, et al. 2003). Some similar toxicity has been reported in animals following intraperitoneal injection. Free radical production may be associated with quinones and/or semi-quinones present in humic acids, but mechanisms are not known with any certainty. This in vitro or IP toxicity may not be relevant for environmental exposures, since humic acids are unlikely to be absorbed from the GI tract to any significant extent.

Tests for mutagenicity with humic acids have been negative in almost all cases (Bernacchi, et al. 1996; Ribs, et al. 1997). The only positive results reported were explained by authors as due to residual chlorine used in an extraction process (Cozzi, et al. 1993). [An exception is discussed along with information on environmental exposures in Taiwan (see below)]. Lack of mutagenicity is consistent with lack of carcinogenicity, but in vitro tests can never be conclusive. A carcinogenic response seems unlikely, however, given the size of these molecules and their expected lack of absorption from the GI tract.

Toxicity information on humic acids is limited, especially for longer exposure durations. These chemicals are of low acute toxicity, lack mutagenicity in almost all cases, and often ameliorate toxicity to aquatic organisms due to heavy metals and some organic chemicals (Xu, et al. 1996; Goodrich, et al. 1991; Kim, et al. 1999; Florence, et al. 1992; Gundersen 1994; Fent and Looser 1995). Overall, little in
available evidence suggests that humic acids would represent a significant human health risk. However, many gaps in available toxicity information exist.

**Environmental Exposures**

Humic acids are ubiquitous in surfaces waters and many human populations are continuously exposed to these chemicals (EPA 2000, Federal Register, July 18, Vol 65, Number 138). Several literature searches have failed, with one possible exception, to provide any indication of health problems associated with exposure to humic acids in drinking water. In some parts of the US and the world, the amount of humic acids in drinking water is substantial and one would expect significant health effects to be notable if humic acids were significantly toxic. Thus, lack of reported toxicity from continuous drinking water exposures does provide some indication that exposure to humic acids does not produce chronic toxicity. Of course, this conclusion cannot be made with certainty since it is at least theoretically possible that some more subtle and/or common health effects could go unnoticed. For example, chelation of metals by humic acids could alter human responses to intake by making them either more or less bioavailable. Such effects might be less likely to be identified without actual epidemiological studies.

Humic acids have been suggested by some authors as a factor in the etiology of Blackfoot disease in Taiwan (Lu, et al. 1988). Areas where Blackfoot is endemic are characterized by high levels of arsenic and humic acids in drinking water. Since Blackfoot disease is not characteristic of all areas where arsenic levels in drinking water are elevated, factors other than arsenic exposure has been considered as factors in the development of the disease in Taiwan. Besides being found in high concentrations in drinking water, humic acids in Blackfoot areas have been shown to be mutagenic in short-term assays. The basis for this mutagenicity is not known and is inconsistent with lack of mutagenic response observed for humic acids from other sources. Further, a casual association between humic acids concentrations and Blackfoot disease does not address the issue of the size and associated lack of GI absorption of these chemicals. Some unusual aspect of humic acids from the Blackfoot disease area, or some site-related chemical associated with these acids may well be responsible for the mutagenicity and, perhaps, may be a factor in disease etiology. In any event, the association of disease with humic acids in drinking water appears to be specific for the Blackfoot disease region of Taiwan, and is likely not relevant to toxicity of humic acids in general.

**Agricultural Use**

Humic acids have been used for decades as soil amendments and adjuvants for pesticide formulations for various food crops. Manufacturers, users and field workers have not reported adverse health effects of humic acids extracted, packaged and applied to fields. Moreover, EPA found that "humic acid, sodium salt is expected to be practically non-toxic to mammals" and that "a cumulative risk assessment is not necessary" to support an exception tolerance for pesticide formulations (EPA 2000, Federal Register, July 18, Vol 65, Number 138). Further, materials safety data sheets
for humic acids list few if any toxicity concerns, report no important toxic constituents or trace contaminants, and suggest few if any cautions for clean-up of spilled material (Ligno-Tech, 2004). Very wide-spread use of humic acids as soil amendments without report of any toxicity is consistent with low toxicity. Since agricultural use would go on for a number of years, agricultural use provides a glimpse of potential for human toxicity (or lack thereof) after chronic exposure.

Summary
Toxicity of humic acids has not been characterized using standard test methods used for most chemicals. However, several lines of evidence suggest, overall, that humic acids are minimally toxic.

- Humic acids are large polar compounds and are unlikely to be absorbed for the GI tract.
- No acute toxicity is observed in laboratory animals even when very large doses (g/kg) are administered.
- Humic acids may reduce metal toxicity probably by chelating metals and making them less bioavailable.
- Except for humic acids from the area where Blackfoot disease is found in Taiwan, humic acids are not mutagenic in short-term bioassays.
- Humic acids are ubiquitous in the environment, and many communities which obtain water from surface resources have high amounts of humic acids in drinking water. Despite chronic exposure to relatively high concentrations, no reports are available that suggest any long term health consequences for people drinking these waters.
- Humic acids have been used for decades as soil amendments in agricultural settings. Workers handling these materials have not reported signs of toxicity that could be traced to humic acids.

By themselves, none of the above lines of evidence would serve to characterize chronic toxicity of humic acids. However, taken together the weight of evidence suggests that even relatively high chronic exposure to humic acids is not associated with any adverse health effects. Use of humic acids as electron shuttles for subsurface remediation would appear to pose a significant environmental risk.

Indigo Carmine
Indigo carmine is a large (MW > 400) disulfonic acid that is used primarily as a food coloring. Synonyms include indigotine, CI Food Blue 1, CI Acid Blue 74, CI Natural blue 2, FD&C Blue No. 2 and many others. Because of its use as an additive to food, the chemical has been studied fairly extensively and some basic aspects of its toxicology are known with some confidence. Overall, indigo carmine is relatively
non-toxic and seem unlikely to represent a significant threat when released to the environment during remediation efforts. Much of the following description of the toxicity of indigo carmine is taken from a previous review (BIBRA Working Group, 1996).

**Acute Toxicity**

Acute toxicity of indigo carmine is low, with rodent oral LD₅₀s reported in the range of 2 g/kg. Probably, this low toxicity is due, at least in part, to low bioavailability in the GI tract. The chemical is large and polar and unlikely to be efficiently absorbed. Reported LD₅₀s are 1 to 2 orders of magnitude lower when indigo carmine is administered s.c. or i.v. (Graham & Allmark 1959). Some reports do suggest poor GI absorption, but provide few details (Reports from the Scientific Committee for Food (1983).

**Subchronic Toxicity**

No clinical signs, gross lesions or histopathological changes were reported in dogs fed indigo carmine at up to 2 percent in the diet subchronically for 2 years (WHO 1975). Similar results were reported in pigs administered up to 1.3 g/kg-d for 90 days. In a special study of the major metabolite of indigo carmine, isatine-5-sulfonic acid, no gross or histopathological changes were noted in rats fed a diet that contained up to 2 percent of the chemical for 13 weeks (WHO 1975).

A final study using rats did suggest some effects on body weight and some biochemical parameters (e.g., kidney and liver functions) (Aboel-Zahab, et al. 1997). Doses were high and similar to those used in the subchronic studies cited above. The authors conclude that the changes seen were not serious effects.

**Chronic Toxicity**

Two chronic bioassays have been reported for mice. In the first, a 2.5 mg/kg dose of indigo carmine was administered s.c. daily for 104. No treatment related tumors were reported. In the second, indigo carmine was added to feed at up to 1.6 percent for 80 weeks. Aside from slight anemia at feed concentrations of 0.8 and 1.6 percent, no treatment related changes were reported in biological, gross or histopathological parameters or tumor incidence (WHO 1975).

Four chronic bioassays have been reported using rats. In the two feeding studies, indigo carmine in the diet at concentrations up to 1 percent induced no treatment related biological or gross or histopathological changes and no increased tumor incidence was observed (Hanson, et al. 1966; Oettel, et al. 1965). Growth of male rats was inhibited at feed concentration of 2 and 5 percent. but even in these animals no other treatment related changes were observed.

Two other rat studies are reported, both involving injection of indigo carmine. In the first, a two percent solution of chemical was injected (route not specified) for 2 years. Total or daily doses were not provided. No treatment related changes were observed,
except for fibrosarcomas at the site of injection. In a shorter (7 month) study, daily s.c. injection of 1 ml of a 0.5 percent solution of indigo carmine produce no treatment related changes, including no tumors at the site of injection (WHO 1975).

**Reproductive Toxicity and Teratogenicity**
In a study of potential reproductive effects, rats were administered up to 250 mg/kg-d of indigo carmine daily by gavage (Oettel, et al. 1965). No signs of either maternal or fetal toxicity were observed. Similar results were observed in rabbits administered the same doses of indigo carmine by gavage. Not surprisingly, studies using these same daily doses, but only during specific days during gestation, showed no signs of teratogenicity in either rats or rabbits (WHO 1975).

**Human Toxicity**
The only reports of indigo carmine toxicity in humans come from episodes where patients were administered the chemical i.v. during surgery. The chemical was used as a vasography agent, but also caused a drop in blood pressure.

**Summary**
Data appear to be sufficient to characterize to toxicity of indigo carmine as low for acute, subchronic and chronic exposure durations. Even very high doses (e.g., up to 1 to 2 percent in animal diets) appear to cause no observable changes as measured by common biochemical and gross or histopathology parameters. The chemical is large and is unlikely to be absorbed from the GI tract following ingestion. Its common use as a food coloring is consistent with a conclusion of low toxicity. Overall, releases of indigo carmine during remediation efforts should pose little if any environmental risk.

**2,6-Anthraquinone disulfonic acid (AQDS)**
The toxicity of AQDS has apparently not been characterized. A search of common toxicological databases (e.g., HSDB, Toxline, Toxnet, Medline) and of on the internet recovered very little of any use in describing the toxicological properties of this chemical. Thus, overall, the environmental hazard that might be associated with release of this chemical remains largely unknown.

AQDS is a large (MW> 300) polar molecule. It would not be expected to be well absorbed from the GI tract following ingestion because of these properties, and considering the apparently low GI bioavailability of indigo carmine. Both AQDS and indigo carmines are large disulfonic acids and might be expected to behave similarly in the GI tract.

Unlike indigo carmine, AQDS is a planar molecule and can intercalate between bases in nucleic acids. Chemicals that can intercalate, such as the azo dyes and acridines, are effective mutagens and/or animal carcinogens. Thus, it is possible that AQDS has some mutagenic or carcinogenic potential.
The scant data available to describe the toxicity of AQDS make any conclusions about potential health threats associated with its release during remediation problematic.

References


EPA 2000 Humic Acid, Sodium Salt, Exemption Tolerance, Fed Reg, July 18 (Volume 65, Number 138), Page 44469-44472


Goodrich, MS, LH Dulak, MA Friedman and JJ Lech. 1991. Acute and long-term toxicity of water-soluble cationic polymers to rainbow trout (Oncorhynchus mykiss) and the modification of toxicity by humic acid. Environ Toxicol Chem 10:509-516.


Appendix B
Altus AFB Reports
FINAL REPORT

SUBSURFACE INVESTIGATION OF A MULCH BIOWALL AT LANDFILL 03, ALTUS AIR FORCE BASE, OKLAHOMA

In support of:
Environmental Security Technology Certification Program Project CU-0316:
Enhanced Bioremediation of cis-Dichloroethene (cis-DCE) and Vinyl Chloride (VC)
Using Electron Shuttles

Prepared for:
Naval Facilities Engineering Command
Port Hueneme, California
Contract No. N47408-99-C-7022

July 2005

Prepared by:
PARSONS
1700 Broadway, Suite 900
Denver, Colorado 80290
1.0 INTRODUCTION

On 16 December 2004, the Navy modified Contract N47408-99-C-7022 with Parsons Infrastructure & Technology Group, Inc. (Parsons) to conduct soil and groundwater sampling at Altus Air Force Base (AFB), Oklahoma, in support of Environmental Security Technology Certification Program (ESTCP) Project CU-0316, Enhanced Bioremediation of cis-Dichloroethene (cis-DCE) and Vinyl Chloride (VC) Using Electron Shuttles. The overall ESTCP project objectives and scope of work, along with a site description, are provided in the work plan for ESTCP Project CU-0316, titled “Draft Work Plan, Subsurface Investigation and Treatability Study Site 1 Alameda Point Alameda, California and Existing Mulch Biowall Altus Air Force Base Altus, Oklahoma” (CDM, 2004).

The objective of the work described in this Final Report is to collect groundwater and soil/mulch substrate samples to evaluate the role of electron shuttles in the enhanced biodegradation of cis-1,2-dichlorethene (cis-DCE) and vinyl chloride (VC) in groundwater in the vicinity of an existing mulch biowall at Landfill 03 (LF03), Altus AFB. The biowall was constructed in June 2002 by Parsons for the Air Force Center for Environmental Excellence (AFCEE) and Altus AFB. This report summarizes the field activities for sample collection and presents the analytical results for the groundwater and soil/mulch samples collected at the Altus AFB LF03 mulch biowall site.

2.0 SCOPE OF WORK

The location of the mulch biowall at LF-03 is shown on Figure 1 (attached). Sample collection was conducted over a period from 18 April 2005 to 22 April 2005 by drilling and sampling soil borings within the biowall, and by collecting groundwater samples from existing monitoring wells. Site activities included the following:

- Drilling and sampling of two soil borings within the biowall to a total depth of 25 feet below ground surface (bgs),
- Collection of three soil/mulch substrate samples from each boring,
- Abandoning the two soil borings,
- Collection of six groundwater samples from existing groundwater monitoring wells, and
- Disposal of investigation-derived waste (IDW).

Site activities are described in the following sections and are summarized in Table 1 (attached). Soil and groundwater samples were submitted for laboratory analyses as described in Section 3 and Section 4. In addition to the procedures described in this report, work were conducted in accordance with the Work Plan for the Bark Mulch Trench Interim Corrective Action, In-Situ Anaerobic Bioremediation of Contaminated Groundwater at Altus Air Force Base, Oklahoma (Parsons, 2004).
3.0 DRILLING AND SOIL SAMPLING

Prior to drilling, Parsons coordinated with Altus AFB and the local public utility locator to locate underground utilities. Two borings (SB-1 and SB-2) were advanced into the mulch biowall at LF03 using a truck mounted hollow stem auger rig. The soil borings were located adjacent to monitoring wells PES-MP-1 and PES-MP-6, shown on Figure 2 (attached). Boreholes were drilled using 4-1/4-inch inside-diameter hollow-stem augers, and soil samples were collected through the augers using standard split-spoon samplers. The location, number, and sample analytes are provided on Table 1. Soil/mulch matrix samples were sent to fixed-based laboratories and analyzed in accordance with the sampling protocol listed in Table 2 (attached).

Soil/mulch samples for electron shuttle bioassays and other analyses listed on Table 1 were collected and packaged to ensure sample integrity prior to analysis by the analytical laboratories. Immediately after extracting and opening the sampler, a portion of each sample was transferred to the appropriate sample container. Samples for electron shuttle bioassays were placed into 4-ounce glass jars, and groundwater from the adjacent monitoring well was added to saturate the sample and eliminate headspace. Samples for analysis of volatile organic compounds (VOCs) and total organic carbon (TOC) were placed into 4-ounce jars but were not saturated. The jars were then sealed with a Teflon®-lined cap, packaged in a cooler with ice, then shipped for overnight delivery to the analytical laboratory.

Each sample for analysis of bioavailable iron, total iron, sulfide, iron sulfides (FeS and FeS₂) was placed into two 40-milliliter (ml) glass vials, sealed with a Teflon® septa cap, then purged with nitrogen gas to eliminate oxygen. The vials were then wrapped in bubble wrap, packaged in a cooler with dry ice, then shipped for overnight delivery to the analytical laboratory.

After each soil boring was completed and samples collected, the augers were retracted and the biowall material removed during drilling was placed back into the borehole.

4.0 GROUNDWATER SAMPLING

Groundwater samples were collected from six existing monitoring wells (OU-1-04, PES-MP-01, PES-MP-02, WL019, PES-MP-06, and PES-MP-07), located along two transects oriented perpendicular to the mulch biowall. Although the work plan indicated an upgradient sample would be collected from well OU-1-05, well OU-1-04 was selected for the upgradient sample for the northern transect because a tracer test was being performed by the U.S. Environmental Protection Agency (USEPA) at OU-1-05 during the sampling event. Each transect is comprised of one upgradient well, one well placed within the mulch biowall, and one downgradient well (Figure 2).

Additional downgradient monitoring wells located along the biowall monitoring transects (PES-MP-03, PES-MP-04, PES-MP-05, PES-MP-08, PES-MP-09, and PES-MP-10) were sampled under a contract with AFCEE. To provide a more complete description of site conditions at the time of the sampling event, Parsons has included these data in the tables attached to this report.

Prior to collection of groundwater samples, each well was purged to remove stagnant water from the well and to allow its replacement by groundwater from the adjacent formation, which is more representative of actual aquifer conditions. A peristaltic pump was used to purge the monitoring wells and to collect groundwater samples. A dedicated
length of clean, disposable high-density polyethylene (HDPE) tubing was used for each well. Groundwater samples were both analyzed in the field and sent to fixed-based laboratories for analysis in accordance with the sampling protocol provided in Table 2.

5.0 ANALYTICAL RESULTS

Analytical results for soil/mulch and groundwater samples are provided in Appendix A, and are summarized on Tables 3 through 9. Data provided in Table 3 through Table 9 are as follows:

- Table 3 lists groundwater elevation data for April 2005.
- Table 4 is a summary of chlorinated aliphatic hydrocarbons (CAHs) detected in groundwater.
- Table 5 lists groundwater geochemical data.
- Table 6 lists concentrations of total organic carbon and volatile fatty acids in groundwater.
- Table 7 lists concentrations of dissolved metals (arsenic, selenium, and manganese) in groundwater for select wells (OU1-04, WL-019, PES-MP2, and PES-MP7).
- Table 8 is a summary of analytical results for CAHs in samples of the sand/mulch mixture from the biowall.
- Table 9 is a summary of the shuttling capacity of soil/mulch samples determined by bioassays conducted at the University of Massachusetts.
- Table 10 is a summary of the soil mineralogical analyses of the soil mulch mixtures.

6.0 INVESTIGATION-DERIVED WASTE

Solid IDW waste such as personal protective equipment (PPE), disposable sample tubing, disposable gloves, etc. were double-bagged and disposed to the Base solid waste handling system. Liquid IDW and decontamination fluids generated at the site were containerized and discharged to the Base groundwater treatment system. Drill cuttings were used to backfill the soil borings immediately following soil/mulch sampling activities.

7.0 REFERENCES


Figure 1. Location of Mulch Biowall Relative to TCE Plume (isoconcentration contours in micrograms per liter of TCE in April 1999. (CDM, 2004)
Figure 2. Monitoring Well Sampling Locations Relative to Mulch Biowall. (CDM, 2004)
TABLES
### TABLE 1
**SUMMARY OF PROPOSED SAMPLING ACTIVITIES**

**BIOWALL SAMPLING AND ANALYSIS PLAN**

**ALTUS AFB, OKLAHOMA**

<table>
<thead>
<tr>
<th>Location Identifier</th>
<th>Location Description</th>
<th>Soil/Mulch Analyses</th>
<th>Groundwater Analyses</th>
</tr>
</thead>
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<td></td>
<td></td>
<td>Soil/Mulch Analyses</td>
<td>Groundwater Analyses</td>
</tr>
<tr>
<td></td>
<td></td>
<td>VOCs(^a)</td>
<td>Total Organic Carbon (SW8260B)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Total Iron, Sulfide, FeS, and FeS(_2) (Microseeps SOP(^b))</td>
<td>Total Iron, Sulfide, FeS, and FeS(_2) (Microseeps SOP(^b))</td>
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<tr>
<td></td>
<td></td>
<td>Bioavailable Iron (New Horizons)</td>
<td>Bioavailable Iron (New Horizons)</td>
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<td></td>
<td></td>
<td>Electron Shuttle Biosensor (UMASS(^c))</td>
<td>Electron Shuttle Biosensor (UMASS(^c))</td>
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<td></td>
<td></td>
<td>8 RCRA Metals (SW8260B)</td>
<td>8 RCRA Metals (SW8260B)</td>
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<tr>
<td></td>
<td></td>
<td>Methane, Ethane, Ethene (AM-20GAX)</td>
<td>Methane, Ethane, Ethene (AM-20GAX)</td>
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<tr>
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<td></td>
<td>Nitrate + Nitrite (E353.1)</td>
<td>Nitrate + Nitrite (E353.1)</td>
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<td>Total Organic Carbon (SW9060M)</td>
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<td></td>
<td>Volatile Fatty Acids (Microseeps SOP)</td>
<td>Volatile Fatty Acids (Microseeps SOP)</td>
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<td>Hardness (E150.2)</td>
<td>Hardness (E150.2)</td>
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<td></td>
<td></td>
<td>Well Head Analyses(^d)</td>
<td>Well Head Analyses(^d)</td>
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<tr>
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<td></td>
<td>Mobile Lab Analyses(^e)</td>
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### Soil Boreholes

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<td>SB-1</td>
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### Groundwater Monitoring Wells

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<td>Within Biowall</td>
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<td>10' Downgradient of Biowall</td>
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<td>WL-019</td>
<td>20' Upgradient of Biowall</td>
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<td>Within Biowall</td>
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<tr>
<td>PES-MP07</td>
<td>5' Downgradient of Biowall</td>
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</table>

**SUBTOTALS** |

|                      |                      | 66     | 6                  |

### Quality Assurance/Quality Control (QA/QC)

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<tr>
<td>Matrix Spike Duplicate</td>
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<td>Trip Blanks</td>
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### Investigation-Derived Waste (IDW) Characterization

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<th>Groundwater Analyses</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

\(^a\) Volatile organic compounds (VOCs) to include aromatic and chlorinated aliphatic hydrocarbons.
\(^b\) Microseeps analytical laboratory internal standard operating procedures (SOP).
\(^c\) University of Massachusetts analytical laboratory internal SOP.
\(^d\) Well head analyses include dissolved oxygen, oxidation-reduction potential, pH, temperature, and conductivity.
\(^e\) Mobile lab analyses include carbon dioxide, alkalinity, ferrous iron, hydrogen sulfide, and manganese.
TABLE 2
ANALYTICAL PROTOCOLS FOR GROUNDWATER, SOIL, AND MULCH/SAND SAMPLES
LF03 MULCH BIOWALL
ALTUS AFB BIOWALL, OKLAHOMA

<table>
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<th>MATRIX</th>
<th>FIELD (F) OR ANALYTICAL LABORATORY (L)</th>
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<td><strong>WATER</strong></td>
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<tr>
<td>Redox Potential</td>
<td>Direct-reading meter</td>
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<tr>
<td>Dissolved Oxygen</td>
<td>Direct-reading meter</td>
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<tr>
<td>pH</td>
<td>Direct-reading meter</td>
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<tr>
<td>Specific Conductance</td>
<td>Direct-reading meter</td>
</tr>
<tr>
<td>Temperature</td>
<td>Direct-reading meter</td>
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<tr>
<td>Ferrous Iron</td>
<td>Colorimetric, Hach Method 8146 (or similar)</td>
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<tr>
<td>Manganese</td>
<td>Colorimetric, Hach Method 8034 (or similar)</td>
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<tr>
<td>Alkalinity (Carbonate [CO$_3^{-2}$])</td>
<td>Titrimetric, Hach Method 8221 (or similar)</td>
</tr>
<tr>
<td>Hydrogen Sulfide</td>
<td>Colorimetric, Hach Method 8131 or HS-C</td>
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<tr>
<td>Carbon Dioxide</td>
<td>Titrimetric, CHEMetrics Method 4500 (or similar)</td>
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<td>Nitrate + Nitrite [as Nitrogen (N)]</td>
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<tr>
<td>Sulfate</td>
<td>E300.1</td>
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<td>Chloride</td>
<td>E300.1</td>
</tr>
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<td>Methane, Ethane, Ethene</td>
<td>AM-20GAX$^a$/</td>
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<tr>
<td>Total Organic Carbon</td>
<td>SW9060</td>
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<tr>
<td>VOCs$^b$</td>
<td>SW8260B</td>
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<tr>
<td>Volatile Fatty Acids</td>
<td>Microbial Insights SOP</td>
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<td>Total Dissolved Solids</td>
<td>E160.3</td>
</tr>
<tr>
<td>Hardness</td>
<td>E130.2</td>
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</table>

| MULCH/SAND                     |                                         |
| VOCS$^b$                       | SW8260B                                | L |
| Total Organic Carbon           | Walkley Black modified                 | L |
| Bioavailable Iron              | New Horizons Test Kit                  | L |
| Total Iron, Sulfide, FeS, and FeS$_2$ | AMIBA Protocol (Rowan University SOP)  | L |
| Electron Shuttles Bioassays    | University of Massachusetts            | L |
| RCRA 8 Metals                  | SW6010B                                | L |

$^a$ AM-20GAX = Microseeps, Inc. laboratory standard operating procedure.
$^b$ VOCs = volatile organic compounds.
# TABLE 3
## SUMMARY OF GROUNDWATER ELEVATIONS
### LF03 MULCH BIO_WALL
#### ALTUS AFB, OKLAHOMA

<table>
<thead>
<tr>
<th>Well/Borehole Identification</th>
<th>Date</th>
<th>Screened Interval (feet bgs)</th>
<th>Northing</th>
<th>Easting</th>
<th>Ground Surface Datum (feet amsl)</th>
<th>Elevation Water Depth to (feet btoc)</th>
<th>Groundwater Elevation (feet amsl)</th>
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</thead>
<tbody>
<tr>
<td><strong>Northern Flow Path</strong></td>
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<td>1354.51</td>
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<td>14 - 24</td>
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<td>1356.6</td>
<td>1356.38</td>
<td>10.39</td>
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</table>

*a/ feet bgs indicates feet below ground surface.

*b/ feet amsl indicates elevation in feet above mean sea level.

*c/ feet btoc indicates depth in feet below top of casing.
### TABLE 4
SUMMARY OF CHLORINATED ALIPHATIC HYDROCARBONS DETECTED IN GROUNDWATER
LF03 MULCH BIOWALL
ALTUS AFB, OKLAHOMA

<table>
<thead>
<tr>
<th>Sample Identification</th>
<th>Sampling Location</th>
<th>Sample Date</th>
<th>Dilution</th>
<th>PCEa/ (µg/L)b/</th>
<th>TCEa/ (µg/L)</th>
<th>1,1-DCEa/ (µg/L)</th>
<th>cis-1,2-DCE (µg/L)</th>
<th>trans-1,2-DCE (µg/L)</th>
<th>VCa/ (µg/L)</th>
<th>Methylene Chloride (µg/L)</th>
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<td><strong>Northern Flow Path</strong></td>
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<tr>
<td>OU1-04</td>
<td>Upgradient</td>
<td>19-Apr-05</td>
<td>100</td>
<td>&lt;50c/</td>
<td>1,500</td>
<td>&lt;50</td>
<td>550</td>
<td>64</td>
<td>&lt;100</td>
<td>23Jd/</td>
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<tr>
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<td>19-Apr-05</td>
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<td>&lt;12</td>
<td>&lt;12</td>
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<td>590</td>
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<td>&lt;12</td>
<td>&lt;12</td>
<td>300</td>
<td>11J</td>
<td>380</td>
<td>3.5J</td>
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<td>10.1J</td>
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<td>2,095</td>
<td>31</td>
<td>259</td>
<td>5.79J</td>
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<td>174</td>
<td>&lt;50</td>
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<td>71</td>
<td>120</td>
<td>&lt;50</td>
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<td>581</td>
<td>&lt;100</td>
<td>2,461</td>
<td>76.5J</td>
<td>&lt;100</td>
<td>18.9J</td>
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<td>WL019</td>
<td>Upgradient</td>
<td>18-Apr-05</td>
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<td>&lt;1.2</td>
<td>74</td>
<td>0.46J</td>
<td>73</td>
<td>16</td>
<td>&lt;2.5</td>
<td>&lt;5.0</td>
</tr>
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<td>PES-MP06 (0')</td>
<td>Within Biowall</td>
<td>18-Apr-05</td>
<td>5</td>
<td>&lt;2.5</td>
<td>5.8</td>
<td>&lt;2.5</td>
<td>85</td>
<td>24</td>
<td>3.0J</td>
<td>&lt;10</td>
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<td>PES-MP07 (5')</td>
<td>5' Downgradient</td>
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<td>&lt;5.0</td>
<td>&lt;5.0</td>
<td>190</td>
<td>28</td>
<td>4.6J</td>
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<td>PES-MP08 (10')</td>
<td>10' Downgradient</td>
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<td>3.32J</td>
<td>&lt;10</td>
<td>272</td>
<td>32</td>
<td>5.39J</td>
<td>&lt;20</td>
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<td>PES-MP18 (duplicate)</td>
<td>10' Downgradient</td>
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<td>3.24J</td>
<td>&lt;10</td>
<td>275</td>
<td>31.82</td>
<td>5.58J</td>
<td>&lt;20</td>
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<td>PES-MP09 (30')</td>
<td>30' Downgradient</td>
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<td>20</td>
<td>&lt;20</td>
<td>7.67J</td>
<td>&lt;20</td>
<td>314</td>
<td>80</td>
<td>3.65J</td>
<td>&lt;40</td>
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<td>PES-MP10 (100')</td>
<td>100' Downgradient</td>
<td>20-Apr-05</td>
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<td>&lt;20</td>
<td>407</td>
<td>&lt;20</td>
<td>79</td>
<td>26</td>
<td>&lt;20</td>
<td>&lt;40</td>
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</tbody>
</table>

a/ PCE = tetrachloroethene, TCE = trichloroethene, DCE = dichloroethene, VC = vinyl chloride.
b/ µg/L = micrograms per liter.
c/ <50 indicates the concentration is below the practical quantification limit indicated.
d/ J-flag indicates the concentration is below the quantification limit but above the method detection limit, and the concentration is estimated.
<table>
<thead>
<tr>
<th>Sample Location</th>
<th>Sample Date</th>
<th>Temp (°C)</th>
<th>pH</th>
<th>Conductivity (mS/cm)</th>
<th>Redox Potential (mV)</th>
<th>Dissolved Oxygen (mg/L)</th>
<th>Dissolved Nitrate (as N) (mg/L)</th>
<th>Ferrous Iron (mg/L)</th>
<th>Carbon Dioxide (mg/L)</th>
<th>Chloride (mg/L)</th>
<th>Sulfate (mg/L)</th>
<th>Sulfide (mg/L)</th>
<th>Hydrogen Sulfide (mg/L)</th>
<th>Dioxide Alkalinity (µg/L)</th>
<th>Methane (µg/L)</th>
<th>Ethane (µg/L)</th>
<th>Ethene (µg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Northern Flow Path</strong></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>OU-1-04</td>
<td>19-Apr-05</td>
<td>17.1</td>
<td>6.75</td>
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<td>8.20</td>
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<td>12.0</td>
<td>0.24</td>
<td>0.9</td>
<td>0.16</td>
<td>1,600</td>
<td>0.25</td>
<td>250</td>
<td>148</td>
<td>308</td>
<td>13</td>
<td>0.055</td>
</tr>
<tr>
<td>PES-MP01 (0')</td>
<td>19-Apr-05</td>
<td>16.0</td>
<td>6.23</td>
<td>4.02</td>
<td>0.60</td>
<td>-332</td>
<td>79</td>
<td>0.020</td>
<td>21.2</td>
<td>0.14</td>
<td>190</td>
<td>0.16</td>
<td>330</td>
<td>928</td>
<td>1,512</td>
<td>12,000</td>
<td>0.006</td>
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<tr>
<td>PES-MP11 (Duplicate)</td>
<td>19-Apr-05</td>
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<td>6.23</td>
<td>4.02</td>
<td>0.60</td>
<td>-332</td>
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<td>0.033</td>
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<td>NA</td>
<td>180</td>
<td>NA</td>
<td>350</td>
<td>NA</td>
<td>NA</td>
<td>11,000</td>
<td>&lt;0.005</td>
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<tr>
<td>PES-MP02 (5')</td>
<td>19-Apr-05</td>
<td>17.4</td>
<td>6.30</td>
<td>5.07</td>
<td>0.40</td>
<td>-340</td>
<td>100</td>
<td>-0.050</td>
<td>22.0</td>
<td>0.07</td>
<td>930</td>
<td>0.72</td>
<td>290</td>
<td>1,160</td>
<td>1,780</td>
<td>14,000</td>
<td>0.005</td>
</tr>
<tr>
<td>PES-MP03 (10')</td>
<td>19-Apr-05</td>
<td>17.0</td>
<td>6.36</td>
<td>5.05</td>
<td>0.60</td>
<td>-206</td>
<td>36</td>
<td>-0.050</td>
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<td>1.0</td>
<td>1,100</td>
<td>0.23</td>
<td>250</td>
<td>688</td>
<td>984</td>
<td>14,000</td>
<td>&lt;0.005</td>
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<tr>
<td>PES-MP04 (30')</td>
<td>19-Apr-05</td>
<td>16.2</td>
<td>6.35</td>
<td>5.53</td>
<td>0.50</td>
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<td>J</td>
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<td>1,300</td>
<td>0.05</td>
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<td>836</td>
<td>8,200</td>
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<td>PES-MP05 (100')</td>
<td>20-Apr-05</td>
<td>17.0</td>
<td>6.57</td>
<td>3.49</td>
<td>0.50</td>
<td>25</td>
<td>10.0</td>
<td>0.24</td>
<td>0.80</td>
<td>0.11</td>
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<td>300</td>
<td>304</td>
<td>560</td>
<td>1,700</td>
<td>&lt;0.005</td>
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<tr>
<td><strong>Southern Flow Path</strong></td>
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<tr>
<td>WL019</td>
<td>18-Apr-05</td>
<td>16.8</td>
<td>6.72</td>
<td>4.21</td>
<td>0.60</td>
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<td>10</td>
<td>0.019</td>
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<td>1,900</td>
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<td>340</td>
<td>160</td>
<td>394</td>
<td>20</td>
<td>0.032</td>
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<tr>
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<td>18-Apr-05</td>
<td>18.4</td>
<td>6.34</td>
<td>4.08</td>
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<td>25</td>
<td>0.038</td>
<td>12.8</td>
<td>0.04</td>
<td>2200</td>
<td>0.10</td>
<td>380</td>
<td>492</td>
<td>685</td>
<td>13,000</td>
<td>&lt;0.005</td>
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<tr>
<td>PES-MP07 (5')</td>
<td>18-Apr-05</td>
<td>16.6</td>
<td>6.45</td>
<td>4.85</td>
<td>0.40</td>
<td>-152</td>
<td>51</td>
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<td>2.1</td>
<td>1,300</td>
<td>0.22</td>
<td>370</td>
<td>1,143</td>
<td>1,492</td>
<td>13,000</td>
<td>0.031</td>
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<td>PES-MP08 (10')</td>
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<td>16.9</td>
<td>6.37</td>
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<td>8.2</td>
<td>1.7</td>
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<td>1,360</td>
<td>13,000</td>
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<td>PES-MP09 (30')</td>
<td>20-Apr-05</td>
<td>17.3</td>
<td>6.39</td>
<td>3.82</td>
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<td>NA</td>
<td>1,100</td>
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<td>330</td>
<td>NA</td>
<td>NA</td>
<td>1,800</td>
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<tr>
<td>PES-MP10 (100')</td>
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<td>18.5</td>
<td>6.71</td>
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<td>1.00</td>
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<td>10</td>
<td>0.38</td>
<td>17.8</td>
<td>&lt;0.2</td>
<td>3,200</td>
<td>&lt;0.10</td>
<td>410</td>
<td>150</td>
<td>292</td>
<td>12</td>
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</table>

*a/ °C = degrees Centigrade.
*b/ su = standard pH units.
*c/ mV = millivolts.
*d/ mS/cm = millisiemens per centimeter.
*e/ µg/L = micrograms per liter.
*f/ J-flag indicates the concentration is below the quantification limit but above the method detection limit, and the concentration is estimated.
*g/ NA = not analyzed.
### TABLE 6
TOTAL ORGANIC CARBON AND VOLATILE FATTY ACIDS IN GROUNDWATER
LF03 MULCH BIOWALL
ALTUS AFB, OKLAHOMA

<table>
<thead>
<tr>
<th>Sample Location</th>
<th>Sample Date</th>
<th>Total Organic Carbon (mg/L)^a/</th>
<th>Total VFAs (mg/L)</th>
<th>Volatile Fatty (Metabolic) Acids</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Pyruvic (mg/L)</td>
</tr>
<tr>
<td><strong>MONITORING WELLS</strong></td>
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</tr>
<tr>
<td>Northern Flow Path</td>
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<td></td>
</tr>
<tr>
<td>OU-1-04 19-Apr-05</td>
<td>12</td>
<td>ND</td>
<td>&lt;4^c/ &lt;1</td>
<td>&lt;1</td>
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<td>PES-MP01 19-Apr-05</td>
<td>79</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
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<tr>
<td>PES-MP11 (Dup) 19-Apr-05</td>
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<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
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<tr>
<td>PES-MP02 19-Apr-05</td>
<td>100</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
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<td>PES-MP03 19-Apr-05</td>
<td>36</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
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<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
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<td>10</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Southern Flow Path</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>WL019 18-Apr-05</td>
<td>10</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
<tr>
<td>PES-MP06 18-Apr-05</td>
<td>25</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
<tr>
<td>PES-MP07 18-Apr-05</td>
<td>51</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
<tr>
<td>PES-MP08 20-Apr-05</td>
<td>57</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
<tr>
<td>PES-MP09 20-Apr-05</td>
<td>26</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
<tr>
<td>PES-MP10 20-Apr-05</td>
<td>10</td>
<td>ND</td>
<td>&lt;4</td>
<td>&lt;1</td>
</tr>
</tbody>
</table>

^a/ mg/L = milligrams per liter.
^b/ ND = not detected.
^c/ "<" indicates that the analyte was below the limit of quantitation.
TABLE 7
DISSOLVED METALS IN GROUNDWATER
LF03 MULCH BIOWALL
ALTUS AFB, OKLAHOMA

<table>
<thead>
<tr>
<th>Sample Identification</th>
<th>Location</th>
<th>Sample Date</th>
<th>Arsenic (mg/L)a/</th>
<th>Selenium (mg/L)</th>
<th>Manganese (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OU1-04</td>
<td>Upgradient of Biowall</td>
<td>19-Apr-05</td>
<td>&lt;0.01b/</td>
<td>0.015Jc/</td>
<td>0.22</td>
</tr>
<tr>
<td>WL-019</td>
<td>Upgradient of Biowall</td>
<td>18-Apr-05</td>
<td>&lt;0.01</td>
<td>0.011J</td>
<td>0.37</td>
</tr>
<tr>
<td>PES-MP2</td>
<td>5 Feet Downgradient of Biowall</td>
<td>19-Apr-05</td>
<td>0.03</td>
<td>0.013J</td>
<td>1.5</td>
</tr>
<tr>
<td>PES-MP7</td>
<td>5 Feet Downgradient of Biowall</td>
<td>18-Apr-05</td>
<td>&lt;0.01</td>
<td>0.016J</td>
<td>1.3</td>
</tr>
</tbody>
</table>

a/ mg/L = milligrams per liter.

b/ < indicates that the analyte was below the limit of quantitation.

c/ J-flag indicates the concentration is below the quantification limit but above the method detection limit, and the concentration is estimated.
## TABLE 8

**SUMMARY OF ANALYTICAL RESULTS FOR MULCH MIXTURE**

**LF03 MULCH BIOWALL**

**ALTUS AFB, OKLAHOMA**

<table>
<thead>
<tr>
<th>Sample Location</th>
<th>Sample Date</th>
<th>Sample Depth (feet bgs)a</th>
<th>Moisture (percent)</th>
<th>TCEb</th>
<th>cis-1,2-DCEb</th>
<th>trans-1,2-DCEb</th>
<th>VCb</th>
<th>Methylene Chloride (µg/kg)</th>
<th>Benzene (µg/kg)</th>
<th>Toluene (µg/kg)</th>
<th>2-butanone (µg/kg)</th>
<th>Acetone (µg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SB1-5</td>
<td>22-Apr-05</td>
<td>5</td>
<td>32.2%</td>
<td>&lt;3.6d</td>
<td>43</td>
<td>0.90J</td>
<td>&lt;7.2</td>
<td>&lt;7.2</td>
<td>&lt;3.6</td>
<td>16</td>
<td>0.89J</td>
<td>42</td>
</tr>
<tr>
<td>SB1-15</td>
<td>22-Apr-05</td>
<td>15</td>
<td>38.2%</td>
<td>12</td>
<td>440</td>
<td>17</td>
<td>130</td>
<td>1.1J</td>
<td>&lt;4.0</td>
<td>8.8</td>
<td>54</td>
<td>56</td>
</tr>
<tr>
<td>SB1-20</td>
<td>22-Apr-05</td>
<td>20</td>
<td>45.1%</td>
<td>25</td>
<td>760</td>
<td>18</td>
<td>210</td>
<td>&lt;8.3</td>
<td>&lt;4.1</td>
<td>11</td>
<td>49</td>
<td>17</td>
</tr>
<tr>
<td>SB1-7</td>
<td>22-Apr-05</td>
<td>7</td>
<td>21.5%</td>
<td>&lt;3.1</td>
<td>3.0J</td>
<td>&lt;3.1</td>
<td>&lt;6.1</td>
<td>0.66J</td>
<td>&lt;3.1</td>
<td>22</td>
<td>0.64J</td>
<td>37</td>
</tr>
<tr>
<td>SB12-7 (duplicate)</td>
<td>22-Apr-05</td>
<td>7</td>
<td>35.4%</td>
<td>&lt;3.7</td>
<td>9.2</td>
<td>1.7J</td>
<td>&lt;7.5</td>
<td>0.89J</td>
<td>0.81J</td>
<td>40</td>
<td>2.7J</td>
<td>33</td>
</tr>
<tr>
<td>SB2-15</td>
<td>22-Apr-05</td>
<td>15</td>
<td>29.0%</td>
<td>1.5J</td>
<td>330</td>
<td>20</td>
<td>1.4J</td>
<td>&lt;6.8</td>
<td>&lt;3.4</td>
<td>36</td>
<td>260</td>
<td>48</td>
</tr>
<tr>
<td>SB2-20</td>
<td>22-Apr-05</td>
<td>20</td>
<td>19.9%</td>
<td>&lt;3.1</td>
<td>130</td>
<td>6.7</td>
<td>&lt;6.1</td>
<td>0.65J</td>
<td>&lt;3.1</td>
<td>8.6</td>
<td>92</td>
<td>54</td>
</tr>
</tbody>
</table>

a/ feet bgs = feet below ground surface.
b/ TCE = trichloroethene; DCE = dichloroethene; VC = vinyl chloride.
c/ µg/kg = micrograms per kilogram.
d/ <3.6 indicates that the analyte was not detected above the indicated method detection limit.
e/ J flag indicates that the analyte was detected at a concentration above the method detection limit but below the reporting limit resulting in an estimated value.
f/ B flag indicates that the analyte was detected in the sample blank.
### TABLE 9
HUMICS IN SOIL
LF03 MULCH BIOWALL
ALTUS AFB, OKLAHOMA

<table>
<thead>
<tr>
<th>Sample Identification (Borehole ID-sample depth)</th>
<th>Sample Date</th>
<th>Test Concentration (Dry Weight) (g/L) (^a)</th>
<th>Shuttling Capacity (% of 0.5 g/L IHSS Soil Humic Acid standard)</th>
<th>Shuttling Capacity 0.2475 mM Fe Reduced (Average of duplicates)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SB1-5</td>
<td>22-Apr-05</td>
<td>1.0</td>
<td>78.79</td>
<td>0.195</td>
</tr>
<tr>
<td>SB1-15</td>
<td>22-Apr-05</td>
<td>1.0</td>
<td>63.03</td>
<td>0.156</td>
</tr>
<tr>
<td>SB1-20</td>
<td>22-Apr-05</td>
<td>1.0</td>
<td>46.67</td>
<td>0.1155</td>
</tr>
<tr>
<td>SB2-7</td>
<td>22-Apr-05</td>
<td>1.0</td>
<td>29.70</td>
<td>0.0735</td>
</tr>
<tr>
<td>SB12-7 (Duplicate)</td>
<td>22-Apr-05</td>
<td>1.0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>SB2-15</td>
<td>22-Apr-05</td>
<td>1.0</td>
<td>32.73</td>
<td>0.081</td>
</tr>
<tr>
<td>SB2-20</td>
<td>22-Apr-05</td>
<td>1.0</td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

\(^a\) g/L = gram per liter.
### TABLE 10
**SUMMARY OF SOIL/MULCH MINERALOGICAL RESULTS**

**LF03 MULCH BIOWALL**

**ALTUS AFB, OKLAHOMA**

<table>
<thead>
<tr>
<th>Sample Location</th>
<th>Sample Date</th>
<th>Sample Depth (feet bgs)</th>
<th>Percent Solids</th>
<th>Organic Carbon (mg/kg)</th>
<th>WAEFe$^{3+}$ c/ (mg/kg)</th>
<th>SAEFe$^{3+}$ c/ (mg/kg)</th>
<th>WAEFe$^{2+}$ c/ (mg/kg)</th>
<th>SAEFe$^{2+}$ c/ (mg/kg)</th>
<th>WAEMn c/ (mg/kg)</th>
<th>SAEMn c/ (mg/kg)</th>
<th>AVS c/ (mg/kg)</th>
<th>CES b/ (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SB1-5</td>
<td>22-Apr-05</td>
<td>5</td>
<td>60%</td>
<td>29,000</td>
<td>&lt;200&lt;sup&gt;d&lt;/sup&gt;</td>
<td>&lt;300</td>
<td>1,300</td>
<td>3,700</td>
<td>&lt;200</td>
<td>&lt;300</td>
<td>13,000</td>
<td>19,000</td>
</tr>
<tr>
<td>SB1-15</td>
<td>22-Apr-05</td>
<td>15</td>
<td>63%</td>
<td>41,000</td>
<td>&lt;200</td>
<td>&lt;300</td>
<td>1,900</td>
<td>5,200</td>
<td>&lt;200</td>
<td>&lt;300</td>
<td>13,000</td>
<td>9,800</td>
</tr>
<tr>
<td>SB1-20</td>
<td>22-Apr-05</td>
<td>20</td>
<td>64%</td>
<td>21,000</td>
<td>&lt;200</td>
<td>&lt;300</td>
<td>&lt;200</td>
<td>500</td>
<td>&lt;200</td>
<td>&lt;300</td>
<td>6,900</td>
<td>7,800</td>
</tr>
<tr>
<td>SB2-7</td>
<td>22-Apr-05</td>
<td>7</td>
<td>86%</td>
<td>15,000</td>
<td>&lt;100</td>
<td>&lt;200</td>
<td>300</td>
<td>1,000</td>
<td>&lt;100</td>
<td>&lt;200</td>
<td>9,000</td>
<td>6,400</td>
</tr>
<tr>
<td>SB12-7 (duplicate)</td>
<td>22-Apr-05</td>
<td>7</td>
<td>67%</td>
<td>23,000</td>
<td>&lt;100</td>
<td>400</td>
<td>600</td>
<td>1,800</td>
<td>&lt;100</td>
<td>&lt;300</td>
<td>14,000</td>
<td>12,000</td>
</tr>
<tr>
<td>SB2-15</td>
<td>22-Apr-05</td>
<td>15</td>
<td>78%</td>
<td>18,000</td>
<td>&lt;100</td>
<td>&lt;200</td>
<td>1,200</td>
<td>3,100</td>
<td>&lt;100</td>
<td>&lt;200</td>
<td>9,400</td>
<td>2,400</td>
</tr>
<tr>
<td>SB2-20</td>
<td>22-Apr-05</td>
<td>20</td>
<td>78%</td>
<td>20,000</td>
<td>&lt;100</td>
<td>900</td>
<td>3,100</td>
<td>5,400</td>
<td>&lt;100</td>
<td>&lt;200</td>
<td>7,400</td>
<td>8,200</td>
</tr>
</tbody>
</table>

<sup>a</sup> feet bgs = feet below ground surface.

<sup>b</sup> mg/kg = micrograms per kilogram dry weight.

<sup>c</sup> WAEFe$^{3+}$ = weak acid extractable ferric iron; SAEFe$^{3+}$ = strong acid extractable ferric iron; WAEFe$^{2+}$ = weak acid extractable ferrous iron; SAEFe$^{2+}$ = strong acid extractable ferrous iron; WAEMn = weak acid divalent manganese; SAEMn = strong acid extractable divalent manganese; AVS = acid volatile sulfide; CES = chromium extractable sulfide.

<sup>d</sup> <200 indicates that the analyte was not detected above the indicated method detection limit.
APPENDIX A
ANALYTICAL RESULTS
## Analytical Results

**Method:** 8260

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dichlorodifluoromethane</td>
<td>&lt;100u</td>
<td>U</td>
<td>-</td>
<td>100</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Chloroform</td>
<td>&lt;100u</td>
<td>U</td>
<td>-</td>
<td>100</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>&lt;100u</td>
<td>U</td>
<td>-</td>
<td>100</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Bromonaphthalene</td>
<td>&lt;100u</td>
<td>U</td>
<td>-</td>
<td>100</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Chloroform</td>
<td>&lt;100u</td>
<td>U</td>
<td>-</td>
<td>100</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td>&lt;100u</td>
<td>U</td>
<td>-</td>
<td>100</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Acetone</td>
<td>&lt;1000u</td>
<td>U</td>
<td>25</td>
<td>1000</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>&lt;50u</td>
<td>U</td>
<td>2.2</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>23</td>
<td>J</td>
<td>8.9</td>
<td>200</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2-Dichloroethene</td>
<td>64</td>
<td></td>
<td>3.9</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Methyl tert-butyl ether</td>
<td>&lt;50u</td>
<td>U</td>
<td>3.2</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1,1-Trichloroethene</td>
<td>&lt;50u</td>
<td>U</td>
<td>2.2</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>2-Butoxone</td>
<td>&lt;1000u</td>
<td>U</td>
<td>68</td>
<td>1000</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>cis-1,2-Dichloroethene</td>
<td>550</td>
<td></td>
<td>3.9</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Bromochloromethane</td>
<td>&lt;50u</td>
<td>U</td>
<td>7.4</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Chloroform</td>
<td>&lt;500u</td>
<td>U</td>
<td>2.3</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>2,2-Dichloropropane</td>
<td>&lt;50u</td>
<td>U</td>
<td>2.9</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2-Dichloroethene</td>
<td>&lt;50u</td>
<td>U</td>
<td>1.8</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1,1-Trichloroethene</td>
<td>&lt;50u</td>
<td>U</td>
<td>4.1</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>&lt;500u</td>
<td>U</td>
<td>2.6</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>&lt;50u</td>
<td>U</td>
<td>3.3</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Benzene</td>
<td>&lt;50u</td>
<td>U</td>
<td>1.7</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Dibromomethane</td>
<td>&lt;50u</td>
<td>U</td>
<td>2.5</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>&lt;50u</td>
<td>U</td>
<td>4.7</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Trichloroethene</td>
<td>1500</td>
<td></td>
<td>3.1</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Bromodichloromethane</td>
<td>&lt;50u</td>
<td>U</td>
<td>2.3</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>cis-1,3-Dichloropropane</td>
<td>&lt;50u</td>
<td>U</td>
<td>2.6</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>4-Methyl-2-pentanone</td>
<td>&lt;500u</td>
<td>U</td>
<td>12.6</td>
<td>500</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>trans-1,3-Dichloropropene</td>
<td>&lt;50u</td>
<td>U</td>
<td>3.1</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1,2-Trichloroethene</td>
<td>&lt;50u</td>
<td>U</td>
<td>4.3</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Toluene</td>
<td>&lt;50u</td>
<td>U</td>
<td>1.6</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,3-Dichloropropane</td>
<td>&lt;50u</td>
<td>U</td>
<td>2.8</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Dibromochloromethane</td>
<td>&lt;50u</td>
<td>U</td>
<td>1.7</td>
<td>50</td>
<td>04/28/05</td>
<td></td>
</tr>
</tbody>
</table>

- Analyte detected above the PQL in the associated Prep Block.
- Outside control limits
- U - Undetected at the reported level.
- * - reported value is estimated
- D - Result is diluted.
- B - concentration exceeded the calibration range and is estimated.

**Additional Notes:**

- **Matrix:** Water
- **Sample Size:** 10 mL
- **Dilution:** 100
- **Prep Block:** HP5973 GC/MS#1
- **Sample Description:** OUI-04
- **Job No.:** 0025. 008.32306
- **Certification NY No.:** 101S0R

**Collection:** 04/19/05
**Received:** 04/23/05
**Prepared:** 04/28/05
**QC Batch:** 042805W1

**Author:** Monika Santucci
**Date:** May 3, 2005

500B Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (515) 437-0200
## Analytical Results

**Method: 8260**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2-Dibromoethane</td>
<td>&lt;50.</td>
<td>U</td>
<td>3.2</td>
<td>50.</td>
<td>04/20/05</td>
<td></td>
</tr>
<tr>
<td>Tetrachloroethene</td>
<td>&lt;50.</td>
<td>U</td>
<td>4.6</td>
<td>50.</td>
<td>04/20/05</td>
<td></td>
</tr>
<tr>
<td>1,1,1,2-Tetrachloroethane</td>
<td>&lt;50.</td>
<td>U</td>
<td>5.3</td>
<td>50.</td>
<td>04/20/05</td>
<td></td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>1.7</td>
<td>50.</td>
<td>04/20/05</td>
<td></td>
</tr>
<tr>
<td>1-Chlorohexane</td>
<td>&lt;50.</td>
<td>U</td>
<td>5.4</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>2.5</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Bromoform</td>
<td>&lt;50.</td>
<td>U</td>
<td>13.</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Xylene (total)</td>
<td>&lt;50.</td>
<td>U</td>
<td>6.1</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Styrene</td>
<td>&lt;50.</td>
<td>U</td>
<td>1.7</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>&lt;50.</td>
<td>U</td>
<td>4.6</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2,3-Trichloropropane</td>
<td>&lt;50.</td>
<td>U</td>
<td>36.</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Isopropylbenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>1.5</td>
<td>50.</td>
<td>04/20/05</td>
<td></td>
</tr>
<tr>
<td>Bromobenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>2.4</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>m-Xylenes</td>
<td>&lt;50.</td>
<td>U</td>
<td>1.2</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>2-Chlorotoluene</td>
<td>&lt;50.</td>
<td>U</td>
<td>2.8</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>o-Chlorotoluene</td>
<td>&lt;50.</td>
<td>U</td>
<td>1.4</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,3,5-Trimethylbenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>3.2</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>tert-Butylbenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>1.7</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>n-Butylbenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>6.8</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2,4-Trimethylbenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>2.4</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>sec-Butylbenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>1.9</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>3.1</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>3.5</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>p-Isopropyltoluene</td>
<td>&lt;50.</td>
<td>U</td>
<td>2.2</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>&lt;50.</td>
<td>U</td>
<td>6.7</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2-Dibromo-3-chloropropane</td>
<td>&lt;100.</td>
<td>U</td>
<td>22.</td>
<td>100.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>&lt;100.</td>
<td>U</td>
<td>13.</td>
<td>100.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Naphthalene</td>
<td>&lt;100.</td>
<td>U</td>
<td>13.</td>
<td>100.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>&lt;100.</td>
<td>U</td>
<td>15.</td>
<td>100.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2,3-Trichlorobenzene</td>
<td>&lt;100.</td>
<td>U</td>
<td>14.</td>
<td>100.</td>
<td>04/28/05</td>
<td></td>
</tr>
</tbody>
</table>

- B: Analyte detected above the PQL in the associated Prep Blank
- O: Outside control limits
- U: Undetected at the reported level
- I: Reported value is estimated
- D: Result is dilute
- E: Concentration exceeded the calibration range and is estimated.

**Job No.:** 0055  008-32206  **Certification NY No.:** 10155B

**Collected:** 04/19/05  **Matrix:** Water

**Received:** 04/22/05  **Sample Site:** 10 mL

**Prepared:** 04/28/05  **Dilution:** 100

**Sample #:** P4054  **Sample Description:** GUL-04

**Project:** OUL-Bowll-Altus AFB  **Instrument:** HP5973 GC/MS#1

**Units:** ug/L  **Number of analytes:** 63

**Author:** Monika Santucci  **Date:** May 3, 2005
O'Brien & Gere Laboratories, Inc.

Analytical Results
Method: 8260

Job No.: 0055 - 008 22206
Certification NY No.: 10155R

Collected: 04/19/05
Received: 04/23/05
Prepared: 04/28/05
Matrix: Water
QC Batch: 042805W1

% Solids: Sample Size: 10 mL
Dilution: 100

<table>
<thead>
<tr>
<th>Surrogate</th>
<th>%R</th>
<th>Qual Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,1-Dioctanolfluoromethane (surrogate)</td>
<td>1.03</td>
<td>75 - 127</td>
</tr>
<tr>
<td>1,2-Dichloroethane-d4 (surrogate)</td>
<td>99</td>
<td>75 - 134</td>
</tr>
<tr>
<td>Toluene-d8 (surrogate)</td>
<td>1.01</td>
<td>75 - 125</td>
</tr>
<tr>
<td>Bromomethane (surrogate)</td>
<td>94</td>
<td>75 - 125</td>
</tr>
</tbody>
</table>

Notes:

B - Analyte detected above the PQL in the associated Prep Blank.
# - Outside control limits
U - Undetected at the reported level.
I - reported value is estimated.
D - Result is diluted.
E - concentration exceeded the calibration range and is estimated.

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-6200

Authorized: Monika Santucci
Date: May 3, 2005

[Signature]
<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dichlorodifluoromethane</td>
<td>&lt;25. U</td>
<td>.85</td>
<td>25.</td>
<td>0.4/28/05</td>
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<tr>
<td>Chloromethane</td>
<td>&lt;25. U</td>
<td>.85</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>&lt;550. U</td>
<td>.78</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Bromomethane</td>
<td>&lt;25. U</td>
<td>2.4</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Chloroethane</td>
<td>&lt;25. U</td>
<td>.3</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td>&lt;25. U</td>
<td>.45</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Acetone</td>
<td>&lt;250. U</td>
<td>5.8</td>
<td>250.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>&lt;12. U</td>
<td>.55</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>J 5.9 J</td>
<td>1.3</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>trans-1,2-Dichloroethene</td>
<td>J 5.5 J</td>
<td>.98</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Methyl tert-butyl ether</td>
<td>&lt;12. U</td>
<td>.80</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>cis-1,1-Dichloroethane</td>
<td>&lt;12. U</td>
<td>.55</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>2-Hexanone</td>
<td>&lt;250. U</td>
<td>1.7</td>
<td>250.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>cis-1,2-Dichloroethene</td>
<td>69.</td>
<td>.98</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Bromochloromethane</td>
<td>&lt;12. U</td>
<td>1.8</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Chloroform</td>
<td>&lt;12. U</td>
<td>.55</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2-Dichloropropane</td>
<td>&lt;12. U</td>
<td>.72</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,2-Dichloroethene</td>
<td>&lt;12. U</td>
<td>.45</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1,1-Trichloroethene</td>
<td>&lt;12. U</td>
<td>1.0</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1-Dichloroethene</td>
<td>&lt;12. U</td>
<td>.65</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Carbon tetrachloride</td>
<td>&lt;12. U</td>
<td>.92</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Benzene</td>
<td>&lt;12. U</td>
<td>.62</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Dichloromethane</td>
<td>&lt;12. U</td>
<td>.62</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>2,2-Dichloropropane</td>
<td>&lt;12. U</td>
<td>1.2</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Trichloroethene</td>
<td>&lt;12. U</td>
<td>.78</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Bromodichloromethane</td>
<td>&lt;12. U</td>
<td>.58</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>cis-1,3-Dichloropropene</td>
<td>&lt;12. U</td>
<td>.55</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>4-Methyl-2-pentanone</td>
<td>&lt;200. U</td>
<td>3.0</td>
<td>120.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>trans-1,3-Dichloropropene</td>
<td>&lt;12. U</td>
<td>.78</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1,2-Trichloroethene</td>
<td>&lt;12. U</td>
<td>1.1</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Toluene</td>
<td>J 5.8 J</td>
<td>.40</td>
<td>12.</td>
<td>04/28/05</td>
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<tr>
<td>1,3-Dichloropropene</td>
<td>&lt;12. U</td>
<td>.70</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Dichloromethane</td>
<td>&lt;12. U</td>
<td>.62</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
</tbody>
</table>
## Analytical Results

**Method:** 8260  
**Job No.:** 0055_J08_12206  
**Certification NY No.:** 10155R  
**Matrix:** Water  
**Batch:** 042803W1

**Collected:** 04/19/05  
**Received:** 04/23/05  
**Prepared:** 04/28/05  
**Sample Size:** 10 mL  
**Dilution:** 23

### Results

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2-Dibromoethane</td>
<td>&lt;12. U</td>
<td>0.80</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>Tetrachloroethylene</td>
<td>&lt;12. U</td>
<td>1.2</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>1,1,1,1-Tetrachloroethane</td>
<td>&lt;12. U</td>
<td>1.3</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>&lt;12. U</td>
<td>0.42</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>1-Chlorobenzene</td>
<td>&lt;12. U</td>
<td>1.4</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>Ethylbenzene</td>
<td>&lt;12. U</td>
<td>0.62</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>Bromoform</td>
<td>&lt;12. U</td>
<td>3.4</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>Xylene (total)</td>
<td>&lt;12. U</td>
<td>1.0</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>Styrene</td>
<td>&lt;12. U</td>
<td>0.42</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>1,1,2,2-Tetrachloroethane</td>
<td>&lt;12. U</td>
<td>1.2</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>1,2,3-Trichloropropane</td>
<td>&lt;12. U</td>
<td>5.6</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>Isopropylbenzene</td>
<td>&lt;12. U</td>
<td>0.38</td>
<td>11.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>Bromobenzene</td>
<td>&lt;12. U</td>
<td>0.60</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>n-Propylbenzene</td>
<td>&lt;22. U</td>
<td>0.30</td>
<td>12.</td>
<td></td>
<td>04/28/05</td>
</tr>
<tr>
<td>2-Chlorotoluene</td>
<td>&lt;12. U</td>
<td>0.70</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>4-Chlorotoluene</td>
<td>&lt;12. U</td>
<td>0.35</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>1,3,5-Trimethylbenzene</td>
<td>&lt;12. U</td>
<td>0.80</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>tert-Butylbenzene</td>
<td>&lt;12. U</td>
<td>0.42</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>n-Butylbenzene</td>
<td>&lt;12. U</td>
<td>2.2</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>1,2,4-Trimethylbenzene</td>
<td>&lt;12. U</td>
<td>0.60</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>sec-Butylbenzene</td>
<td>&lt;12. U</td>
<td>0.49</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>1,3-Dichlorobenzene</td>
<td>&lt;12. U</td>
<td>0.52</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>1,4-Dichlorobenzene</td>
<td>&lt;12. U</td>
<td>0.98</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>p-Isopropyltoluene</td>
<td>&lt;12. U</td>
<td>0.55</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>1,2-Dichlorobenzene</td>
<td>&lt;12. U</td>
<td>1.7</td>
<td>12.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>1,2-Dibromo-3-chloropropane</td>
<td>&lt;25. U</td>
<td>5.3</td>
<td>25.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>1,2,4-Trichlorobenzene</td>
<td>&lt;25. U</td>
<td>1.6</td>
<td>25.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>&lt;25. U</td>
<td>1.6</td>
<td>25.</td>
<td></td>
<td>06/24/05</td>
</tr>
<tr>
<td>1,2,3-Trichlorobenzene</td>
<td>&lt;25. U</td>
<td>3.5</td>
<td>25.</td>
<td></td>
<td>06/24/05</td>
</tr>
</tbody>
</table>

B - Analyte detected above the PQL in the associated Prep Blank  
O - Outside control limits U - Undetected at the reported level  
D - Result is dilute  
- concentration exceeded the calibration range and it is estimated.

**Authorized:** [Signature]  
**Date:** May 3, 2005  
**Monika Samucci**

5000 Brittenfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-6200
**O'Brien & Gere Laboratories, Inc.**

**Analytical Results**

**Method: 8260**

**Client:** Parsons  
**Project:** OUI-Biwowall-Alhus AFB  
**Proj. Desc:** N47408-99-C-7022  
**Package #:** 134  
**Sample #:** F4055  
**Sample Description:** FES-MP1  
**Instrument:** HP5973 GC/MS#1  
**Units:** ug/L  
**Number of analyses:** 63

<table>
<thead>
<tr>
<th>Surrogate</th>
<th>%R</th>
<th>Qual</th>
<th>%R Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethyl-o-fluorocarbon (surrogate)</td>
<td>103</td>
<td>75 - 127</td>
<td></td>
</tr>
<tr>
<td>1,2-Dichloroethylene (surrogate)</td>
<td>99</td>
<td>75 - 134</td>
<td></td>
</tr>
<tr>
<td>Toluene-d8 (surrogate)</td>
<td>102</td>
<td>75 - 125</td>
<td></td>
</tr>
<tr>
<td>Bromofluorobenzene (surrogate)</td>
<td>95</td>
<td>75 - 125</td>
<td></td>
</tr>
</tbody>
</table>

**Notes:**

- B - Analyte detected above the PQL in the associated Prep Blank.
- # - Outside control limits  
- U - Undetected at the reported level.  
- J - Reported value is estimated.  
- D - Result is diluted.  
- E - Concentration exceeded the calibration range and is estimated.

**Authorized:**  
**Date:** May 1, 2005  
**Monsu Sanucci**

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
### Analytical Results

**Method:** 8260

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dichlorodifluoromethane</td>
<td>&lt;25.</td>
<td>U</td>
<td>25.</td>
<td>125.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Chloromethane</td>
<td>&lt;25.</td>
<td>U</td>
<td>25.</td>
<td>125.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>380.</td>
<td></td>
<td>70</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Bromomethane</td>
<td>&lt;25.</td>
<td>U</td>
<td>2.4</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Chloroform</td>
<td>&lt;25.</td>
<td>U</td>
<td>2.1</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Trichlorofluoromethane</td>
<td>&lt;25.</td>
<td>U</td>
<td>4.5</td>
<td>25.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Acetone</td>
<td>&lt;250.</td>
<td>U</td>
<td>5.8</td>
<td>250.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>1,1-Dichloroethane</td>
<td>&lt;12.</td>
<td>U</td>
<td>55</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>J 3.3</td>
<td>J</td>
<td>2.2</td>
<td>50.</td>
<td>04/28/05</td>
<td></td>
</tr>
<tr>
<td>trans-1,2-Dichloroethane</td>
<td>J 11.</td>
<td>J</td>
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<td>0.42</td>
<td>12.</td>
<td>04/28/05</td>
<td></td>
</tr>
</tbody>
</table>

B - Analyte detected above the PQL in the associated Prep Blank.
F - Outside control limits U - Undetected at the reported level.
J - reported value is estimated. D - Result is diluted.
E - concentration exceeded the calibration range and is estimated.

**Certification NYS No:** 10155R

Job No.: 0025. 008. 32206

**Sample:** P4066

**Sample Description:** PES-KP2

**Instrument:** HP5973 GC/MS/F1

**Units:** µG/L

**Number of analyses:** 63

**Acknowledged:** [Signature]

**Date:** May 3, 2005

**Monika Santucci**

---

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
### Analytical Results

**Method:** 8260

**Client:** O'Brien & Gere Laboratories, Inc.
**Job No.:** 0055 008-32206
**Certification NY No.:** 10155R

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MQL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
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<td>1,2-Dichloroethane</td>
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<td>12.</td>
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<td>04/28/05</td>
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<td>1-Chlorohexane</td>
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<td>Benzene</td>
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<td>0.4</td>
<td>12.</td>
<td>04/28/05</td>
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<td>1,2,2,2-Tetrachloroethane</td>
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<td>U</td>
<td>1.2</td>
<td>12.</td>
<td>04/28/05</td>
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<td>12.</td>
<td>04/28/05</td>
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<tr>
<td>Bromobenzene</td>
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<td>U</td>
<td>0.6</td>
<td>12.</td>
<td>04/28/05</td>
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<td>n-Propylbenzene</td>
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<td>0.3</td>
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<td>04/28/05</td>
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<td>2-Chlorotoluene</td>
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<td>12.</td>
<td>04/28/05</td>
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<td>04/28/05</td>
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<td>0.5</td>
<td>12.</td>
<td>04/28/05</td>
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<td>1,4-Dichlorobenzene</td>
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<td>1,2-Dichlorobenzene</td>
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<td>U</td>
<td>1.7</td>
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<td>04/28/05</td>
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<td>04/28/05</td>
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<td>U</td>
<td>5.5</td>
<td>25.</td>
<td>04/28/05</td>
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</table>

- B - Analyte detected above the PQI in the associated Prep blank.
- # - Outside control limits
- O - Undetected at the reported level
- 1 - reported value is estimated
- D - Result is diluted
- E - concentration exceeded the calibration range and is estimated.

**Authorized:**

Date: May 3, 2005

Monika Santucci
Surrogate | %R | Qual | %R Limits |
--- | --- | --- | --- |
Styrene (surrogate) | 101 | | 75 - 127 |
1,2-Dichloroethane-24 (surrogate) | 97 | | 75 - 124 |
Toluene-88 (surrogate) | 102 | | 75 - 125 |
Bromonofluorobenzene (surrogate) | 95 | | 75 - 125 |

Notes:

B = Analyte detected above the PQL in the associated Prep Blank.
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J = reported value is estimated. D = Result is diluted.
E = concentration exceeded the calibration range and is estimated.

Authorized: [Signature]
Date: May 3, 2005
<table>
<thead>
<tr>
<th>Parameter</th>
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<th>Qual</th>
<th>MCL</th>
<th>PQL</th>
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<td>Vinyl chloride</td>
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<td>5.0</td>
<td>04/29/05</td>
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<td>Carbon tetrachloride</td>
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<td>Benzene</td>
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<td>0.085</td>
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</table>

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Authorized: [Signature]
Date: May 3, 2005
Monika Santucci
<table>
<thead>
<tr>
<th>Parameter</th>
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<th>Qual</th>
<th>PQL</th>
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Authorized: [Signature]
Date: May 3, 2005
Monika Santucci

5000 Britonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-6200
O'Brien & Gere Laboratories, Inc.

Analytical Results
Method: 8260

Client: Parsons
Project: OUI-Blewett-Almt AFB
Proj. Desc: N47408-95-C-7022
Package: 134
Sample: F4057
Sample Description: PES-MP6
Instrument: HP5973 GCMSFI
Units: ug/L
Number of analytes: 63

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Membrane: [Signature]  Date: May 3, 2005
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<td>&lt;10</td>
<td>U</td>
<td>1.3</td>
<td>10.0</td>
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<td>Hexachlorobutadiene</td>
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<td>04/29/05</td>
</tr>
<tr>
<td>1,2,3-Trichlorobenzene</td>
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<td>U</td>
<td>1.3</td>
<td>10.0</td>
<td>04/29/05</td>
</tr>
</tbody>
</table>

B - Analyte detected above the PQL in the associated Prep Blank.
# - Outside control limits U - Undetected at the reported level.
J - reported value is estimated  D - Result is diluted.
E - concentration exceeded the calibration range and is estimated.

Authorized: Monika Santucci
Date: May 3, 2005
5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
**O'Brien & Gere Laboratories, Inc.**

**Analytical Results**

**Method: 8260**

<table>
<thead>
<tr>
<th>Surrogate</th>
<th>%R</th>
<th>Qual. Limits</th>
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<tbody>
<tr>
<td>Giboow, fluoromethane (surrogate)</td>
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<td>75 - 127</td>
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<tr>
<td>1,2-Dichloroethane-84 (surrogate)</td>
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<td>75 - 134</td>
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<tr>
<td>Toluene-d8 (surrogate)</td>
<td>101</td>
<td>75 - 125</td>
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<tr>
<td>Bromofluorobenzene (surrogate)</td>
<td>93</td>
<td>75 - 125</td>
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</table>

**Notes:**

- Analyte detected above the PQL in the associated Prep Blank.
- Outside control limits. U - Undetected at the reported level.
- J - reported value is estimated. D - Result is diluted.
- E - concentration exceeded the calibration range and is estimated.

---

**Certificate No. 10155R.**

**Job No.: 0055.008.32206**

**Certification:**

- **Matrix:** Water
- **QC Batch:** 042905W1
- **Sample Size:** 10 ml
- **Dilution:** 10

**Prepared:** 04/29/05

**Received:** 04/23/05

**Collected:** 04/15/05

**Sample:** F4058

**Package:** 134

**Sample Description:** PSS-MP?

**Instrument:** HP5973 GC/MS

---

**Authorized:**

[Signature]

**Date:** May 5, 2005

Menika Santucci

---

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200

19
### Analytical Results

**Method:** 8260

**Client:** Parsons
**Project:** OUI-Biowall-Alta AEF
**Proj. Desc:** N47408-99-C-7022
**Package:** 154
**Sample:** P4059
**Sample Description:** WL1019
**Instrument:** HP5973 GC/MS#1
**Units:** ug/L
**Sample Site:** 10 mL

<table>
<thead>
<tr>
<th>Parameter</th>
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<th>Qul</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
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<td>Chloromethane</td>
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<td>Vinyl chloride</td>
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<td>0.42</td>
<td>1.2</td>
<td>04/29/05</td>
<td></td>
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</tbody>
</table>

- B = Analyte detected above the PQL in the associated Prep. Blank.
- O = Outside control limits
- U = Undetected at the reported level

**Authored by:** Manisha Sanmucci
**Date:** May 3, 2005

5000 Brittonfield Parkway / Suite 300, Box 4942, Syracuse, NY 13221 / (315) 437-0200
<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MT</th>
<th>PQI</th>
<th>Analyzed</th>
<th>Notes</th>
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<td>n-Butylbenzene</td>
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<td>.12</td>
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<td>.35</td>
<td>2.5</td>
<td>04/29/05</td>
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</tbody>
</table>

B - Analyte detected above the PQI in the associated Prep Blank
- Outside control limits U - Undetected at the reported level
# - reported value is estimated. D - Result is diluted
E - concentration exceeded the calibration range and is estimated.

Date: May 3, 2005
Monika Sandesi
Surrogate &% Qual %R Limits

Dibromofluoromethane (surrogate) 105 75-125
1,2-Dichloroethane-d4 (surrogate) 99 75-125
Trifluoroane-d8 (surrogate) 101 75-125
Pentfluoroanisole (surrogate) 93 75-125

Notes:

B - Analyte detected above the PQL is the associated Prep Blank
# - Outside control limits U - Undetermined at the reported level
- reported value is estimated. D - Result is diluted
E - concentration exceeded the calibration range and is estimated.

3000 Brittmenfield Parkway / Suite 300, Box 4942 / Syracusz, NY 13221 / (315) 437-0200
### Analytical Results

**Method:** 8260

**Job No.:** 0055.069.32206  
**Certification NY No.:** 19153R

- **Sample:** 40106  
  **Sample Description:** FES-MP1  
  **Instrument:** H55973 GC/MS/I  
  **Units:** ug/L  
  **Number of analyses:** 63

<table>
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<tr>
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<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
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<td>Methyl tert-butyl ether</td>
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<tr>
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<td>Carbon tetrachloride</td>
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<td>Benzene</td>
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<td>0.24</td>
<td>0.24</td>
<td>4/29/05</td>
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</tbody>
</table>

B - Analyte detected above the PQL in the associated Prep Blank  
# - Outside control limits  
U - Undetected at the reported level  
D - Reporting value is censored  
E - Concentration exceeded the calibration range and is estimated

- **Sample Size:** 10 mL  
- **Dilution:** 20

**Collected:** 04/19/05  
**Received:** 04/23/05  
**Prepared:** 04/29/05  
**Matrix:** Water  
**QC Batch:** 043905W1

**Sample Size:** 10 mL  
**Dilution:** 20  
**Job No.:** 0055.069.32206  
**Certification NY No.:** 19153R

**Authorized:**  
Date: May 3, 2005  
Monika Samori

5000 Britfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-6200

23
**Analytical Results**

**Method:** 8260

**Client:** O'Brien & Gere Laboratories, Inc.

**Project:** OJ-3017 - Alus AFB

**Job No.:** 0655-003-32206

**Certification:** NY No.: 10119B

**Sample:** 0460

**Sample Description:** FES-MP11

**Instrument:** HP5973 GC/MS/NI

**Units:** ug/L

**Number of analyses:** 63

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MQL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
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<td>1,2-Dichloroethane</td>
<td>&lt;10.</td>
<td>U</td>
<td>.64</td>
<td>10</td>
<td>04/23/05</td>
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<tr>
<td>Tetrahydrofuran</td>
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<td>U</td>
<td>.92</td>
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<td>04/23/05</td>
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<td>U</td>
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<td>04/23/05</td>
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<td>Acenaphthene</td>
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<td>10</td>
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<td>Xylene (total)</td>
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<td>10</td>
<td>04/23/05</td>
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<td>Styrene</td>
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<td>.34</td>
<td>10</td>
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<td>U</td>
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<td>10</td>
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<td>Isopropylbenzene</td>
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<td>.30</td>
<td>10</td>
<td>04/23/05</td>
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<td>2-Chlorotoluene</td>
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<td>10</td>
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<td>4-Chlorotoluene</td>
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<td>10</td>
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<td>1,3,5-Trimethylbenzene</td>
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<td>04/23/05</td>
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<td>tert-Butylbenzene</td>
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<td>10</td>
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<td>n-Butylbenzene</td>
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<td>1,2,4-Trimethylbenzene</td>
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<td>sec-Nitrobenzene</td>
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<tr>
<td>1,3-Dichlorobenzene</td>
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<td>U</td>
<td>.42</td>
<td>10</td>
<td>04/23/05</td>
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<td>1,4-Dichlorobenzene</td>
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<td>.78</td>
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<td>3-Butyltoluene</td>
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<td>1,2-Dichlorobenzene</td>
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<td>U</td>
<td>1.3</td>
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<td>1,2,4-Trichlorobenzene</td>
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<td>U</td>
<td>2.7</td>
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<td>Hexachlorobenzene</td>
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<td>1,2,3-Trichloroform</td>
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<td>U</td>
<td>2.8</td>
<td>20</td>
<td>04/29/05</td>
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</tbody>
</table>

**B - Analyte detected above the PQL in the associated Prep Blank.**

- C - Outside control limits
- U - Undetected at the reported level.
- J - Reported value is estimated.
- D - Result is diluted.
- E - Concentration exceeded the calibration range and is estimated.

**Author: (Signature)**

**Date:** May 3, 2005

**Monica Santucci**

**500 Britonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200**
Analytical Results
Method: 8260

Job No.: 0055.008.32206
Certification NY No.: 10153R

Collected: 04/19/05
Reconciled: 04/23/05
Prepared: 04/20/05
QC Batch: 043905W1

Sample: Water
Sample Size: 10 mL
Dilution: 20

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<th>Surrogate</th>
<th>SP</th>
<th>Qual</th>
<th>LR Limits</th>
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<tr>
<td>Dichlorofluoromethane (surrogate)</td>
<td>108</td>
<td>75-127</td>
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<tr>
<td>1,2-Dichloroethane-d4 (surrogate)</td>
<td>101</td>
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<td>Toluene-d8 (surrogate)</td>
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<td>75-125</td>
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<tr>
<td>Bromofluorobenzene (surrogate)</td>
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<td>75-125</td>
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Notes:

B - Analyte detected above the PQL in the associated Prep Blank.
O - Outside control limits
U - Undetected at the reported level.
- reported value is estimated. D - Result is diluted.
E - concentration exceeded the calibrator range and is estimated.

Authorized: [Signature]
Date: May 3, 2005
Monika Santucci

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
### Analytical Results

**Method:** 8260  
**Job No.:** 0055.008.3220  
**Certification NY No.:** 10155R  
**Received:** 06/22/05  
**Prepared:** 06/27/05  
**%Solid:** 47.8  
**Sample Size:** 5.12 g  
**Dilution:** 1

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<th>Parameter</th>
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<th>Qual</th>
<th>MDL</th>
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<th>Analyzed</th>
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<td>.12</td>
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<td>1,1-Dichloroethane</td>
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<td>cis,1,2-Dichloroethane</td>
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<td>.36</td>
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<td>Chloroform</td>
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<tr>
<td>2,2-Dichloropropane</td>
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<td>.23</td>
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<td>06/21/05</td>
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<tr>
<td>1,2-Dichloroethane</td>
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<td>.34</td>
<td>3.6</td>
<td>06/21/05</td>
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<td>1,1,1-Trichloroethane</td>
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<td>.14</td>
<td>3.6</td>
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<td>Carbon tetrachloride</td>
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<tr>
<td>Benzene</td>
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<td>.33</td>
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<tr>
<td>Dichloromethane</td>
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<td>.36</td>
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<td>06/21/05</td>
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<tr>
<td>1,2-Dichloroethane</td>
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<tr>
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<tr>
<td>Bromochloromethane</td>
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<td>.12</td>
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<td>06/21/05</td>
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<tr>
<td>cis-1,3-Dichloropropene</td>
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<td>.13</td>
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<td>cis-1,3-Dimethylcyclopropane</td>
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<td>.35</td>
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<tr>
<td>1,1,2-Trichloroethane</td>
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<td>.16</td>
<td>3.6</td>
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<tr>
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<td>3.6</td>
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<tr>
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<td>.12</td>
<td>3.6</td>
<td>06/21/05</td>
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<td>.19</td>
<td>3.6</td>
<td>06/21/05</td>
<td></td>
</tr>
</tbody>
</table>

A - Analyte detected above the PQL or the associated Prop. Blank.  
# - Outside control limits  
U - Undetected at the reported level.  
I - reported value is estimated.  
D - Result is diluted.  
E - Concentration exceeded the calibration range and is estimated.

Authorised:  
Date: May 16, 2005  
Monika Sammozi

5000 Brusnell Parkway, Suite 300, Box 4942 / Synectics, NY 11221 / (315) 437-0200
## Analytical Results

**Method:** 8260

**Client:** Pason  
**Project:** OIL-Buwell-Alber AFB  
**Proc. Date:** 04/26/05  
**Sample:** F0061  
**Sample Description:** SL1-5  
**Instrument:** HP973 GC/MS#3  
**Units:** US/UK g/Dry weight  
**Number of studies:** 63

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<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>NDL</th>
<th>PQL</th>
<th>Analyzed Notes</th>
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<td>Bromobenzene</td>
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<td>1,3,5-Trimethylbenzene</td>
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<tr>
<td>1,4-Dichlorobenzene</td>
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<td>3.6</td>
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<td>p-Isopropyltoluene</td>
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<tr>
<td>1,2-Dichlorobenzene</td>
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<td>1,2-Dibromo-3-chloropropane</td>
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</tbody>
</table>

B. Analyte detected above the PQL in the associated Prep Blank.  
4. Outside control limits 'U' - Undetected at the reported level.  
J. Reported value is estimated.  
D. Result is deleted.  
E. Concentration exceeded the calibration range and is estimated.

**Certification NY No.:** 10155R  
**Matrix:** Soil  
**Sample Size:** 5.12 g  
**QC Batch:** 0423US3  
**Sample:** 1.29 g  

**Date:** May 16, 2005  
**Mona Santoski**

**5000 Brinonfield Parkway / Suite 300, Box 4942 / Smyrna, NY 13221 / (315) 437-8500**

---

27
**O'Brien & Gere Laboratories, Inc.**

**Analytical Results**

**Method: 8260**

<table>
<thead>
<tr>
<th>Client: Part #:</th>
<th>Project: OTU-Blowah-Alton AFB</th>
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<tr>
<td>Prod. Desc.:</td>
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<td>Package #:</td>
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<td>Sample #:</td>
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<td>Instrument:</td>
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<td>Units:</td>
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<th>Surrogate</th>
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<th>Qual</th>
<th>%R Limits</th>
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<tr>
<td>Dibromofluoromethane (surrogate)</td>
<td>108</td>
<td>10 - 156</td>
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<td>1,2-Dichloroethane-d5 (surrogate)</td>
<td>222</td>
<td>75 - 125</td>
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<td>Toluene-d8 (surrogate)</td>
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<td>75 - 125</td>
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<td>Bromofluoromethane (surrogate)</td>
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<td>59 - 125</td>
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</tbody>
</table>

Notes:

B - Analyte detected above the PQL in the associated Prep Elut. 
# - Outside control limits. U - Undetected at the reported level. 
J - reported value is estimated. D - Result is dilute. 
E - concentration exceeded the calibration range and is estimated.

Authorized: [Signature]  
Date: May 16, 2005  
Monika Saunter

5000 Brittonfield Parkway / Suite 300, Box 4042 / Syracuse, NY 13221 / (315) 437-0200
# Analytical Results

Method: 8260

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MQL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
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<td>Methylene chloride</td>
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<td>.16</td>
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<tr>
<td>1,1-Dichloroethane</td>
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<td>2,2-Dichloropropane</td>
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<td>1,2-Dichloroethane</td>
<td>&lt;4.0 U</td>
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<td>1,1,1-Trichloroethene</td>
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<td>1,1-Dichloropropane</td>
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<td>Dibromochloromethane</td>
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<td>2,2-Dichloropropane</td>
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<td>Trichloroethene</td>
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<td>cis-1,3-Dichloropropane</td>
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<tr>
<td>trans-1,3-Dichloropropane</td>
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<td>.14</td>
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<td>1,3-Dichloropropane</td>
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<td>Dibromochloromethane</td>
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<td>.21</td>
<td>4.0</td>
<td></td>
<td>04/27/05</td>
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</tbody>
</table>

D - Analyte detected above the PQL in the associated Prep Blank.
# - Outside control limits U - Undetected at the reported level.
J - reported value is estimated.  D - Result is diluted.
E - concentration exceeded the calibration curve and is estimated.

Date: May 16, 2005
Monika Sanucci
5000 Brittonfield Parkway Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
### Analytical Results

Client: O'Brien & Gere Laboratories, Inc.
Project: OUI-Boswah-Ahius AFB
Job No.: 0055-0202-32206
Proj. Desc.: 5047406.99-C-7022
Packaged: 314
Sample: F4062
Sample Description: SB1-5
Instruments: HPLC 5973 GC/MS/MS
Units: ug/Kg Dry weight
Number of analyses: 63

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<tr>
<th>Parameter</th>
<th>Result (ug)</th>
<th>Qual</th>
<th>MDL (ug)</th>
<th>PQL (ug)</th>
<th>Analyzed</th>
<th>Notes</th>
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<td>U</td>
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<td>Tetrachloroethane</td>
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<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
<td></td>
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<tr>
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<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
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<td>Styrene</td>
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<tr>
<td>1,1,2,2-Tetrachloroethane</td>
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<tr>
<td>1,2,3-Trichloropropylene</td>
<td>&lt;4.0</td>
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</tr>
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<td>Trimethylbenzene</td>
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<td>04/27/05</td>
<td></td>
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<tr>
<td>Bromobenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
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<tr>
<td>n-Propylbenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
<td></td>
</tr>
<tr>
<td>2-Chlorobenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
<td></td>
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<tr>
<td>4-Chlorobenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
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<tr>
<td>2,5-Dimethylbenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
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<tr>
<td>tert-Butylbenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
<td></td>
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<tr>
<td>n-Butylbenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
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<tr>
<td>1,2,4-Trimethylbenzene</td>
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<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
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<tr>
<td>sec-Rutylbenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
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<tr>
<td>1,3-Dichlorobenzene</td>
<td>&lt;4.0</td>
<td>U</td>
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<td>0.1</td>
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<tr>
<td>1,4-Dichlorobenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
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<td>5-Propyltoluene</td>
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<td>1,2-Dichlorobenzene</td>
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<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
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<td>1,2-Dibromo-3-Chloropropane</td>
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<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
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<td></td>
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<tr>
<td>1,2,4-Trimethylbenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
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<td></td>
</tr>
<tr>
<td>Naphthalene</td>
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<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
<td></td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
<td></td>
</tr>
<tr>
<td>1,2,3-Trichlorobenzene</td>
<td>&lt;4.0</td>
<td>U</td>
<td>&lt;0.1</td>
<td>0.1</td>
<td>04/27/05</td>
<td></td>
</tr>
</tbody>
</table>

B - Analyte detected above the PQL in the associated Prep Blank.
# - Outside control limit. U - Undetected at the reported level.
J - reported value is estimated. D - Result is diluted.
K - concentration exceeded the calibration range and is estimated.

Authorized: [Signature]
Date: May 16, 2005

Meesa Samucci
5000 Brintonfield Parkway / Suite 200, Box 4942 / Syracuse, NY 13221 / (315) 437-6500

30
## Analytical Results

**Method:** 8260

**Client:** Parsons

**Project:** OUI-Bellows-Falls AFB

**Proj Desc:** N47468-09-C-7022

**Sample:** 94062

**Sample Description:** SBI-15

**Instrument:** HP5973 GC/MS

**Units:** ug/Kg

**Dry weight**

**Number of analytes:** 63

<table>
<thead>
<tr>
<th>Surrogate</th>
<th>%R</th>
<th>Qval</th>
<th>%R Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>o-Dichlorodifluoromethane (surrogate)</td>
<td>111</td>
<td>40-156</td>
<td></td>
</tr>
<tr>
<td>1,1,1-Trichloroethane (surrogate)</td>
<td>119</td>
<td>1-120</td>
<td></td>
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<tr>
<td>Toluene-d8 (surrogate)</td>
<td>57</td>
<td>75-125</td>
<td></td>
</tr>
<tr>
<td>Bromocarbophene (surrogate)</td>
<td>46</td>
<td>99-128</td>
<td></td>
</tr>
</tbody>
</table>

**Notes:**

B - Analyte detected above the PQL in the associated Prep Blank
# - Outside control limits
U - Undetected at the reported level
J - reported value is estimated
D - Result is diluted
E - concentration exceeded the calibration curve and is estimated

**Certification NY No.:** 0155R

**Job No.:** 0055. 008. 32206

**Collect.** 24/22/05

**Prep.** 04/27/05

**Sample Size:** 0.05 g

**Dilution:** 1

**%Solids:** 61.8

**QC Batch:** 04270153

**Authorization:**

**Date:** May 16, 2005

**Monica Sanucci**

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5000 Brittianfield Parkway / Suite 300, Brea 92822 / Riverside, NY 13221 / (315) 437-5200

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31
## Analytical Results

**Method:** 8260

**Client:** Parsons  
**Project:** OUI-Browell-Alta AFB  
**Proj Desc:** NAV489-86-C-7022  
**Package:** 134  
**Sample:** F402 DL  
**Sample Description:** SE115  
**Instrument:** HS5973 GC/MS3  
**Units:** mg/Kg Dry weight  
**Number of analytes:** 63  

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dichlorodifluoromethane</td>
<td>&lt;36  U</td>
<td>.58</td>
<td>36</td>
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<td>04/27/05</td>
</tr>
<tr>
<td>Chloroform</td>
<td>&lt;36  U</td>
<td>.74</td>
<td>36</td>
<td></td>
<td>04/27/05</td>
</tr>
<tr>
<td>Vinyl chloride</td>
<td>&lt;36  U</td>
<td>.58</td>
<td>36</td>
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<td>04/27/05</td>
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<tr>
<td>Bromomethane</td>
<td>&lt;36  U</td>
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<td>36</td>
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<td>04/27/05</td>
</tr>
<tr>
<td>Chloroethane</td>
<td>&lt;36  U</td>
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<td>Trichlorofluoromethane</td>
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<td>04/27/05</td>
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<tr>
<td>Acetone</td>
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<tr>
<td>1,1-Dichloroethane</td>
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<tr>
<td>Methylyne chloride</td>
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<td>trans-1,2-Dichloroethane</td>
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<td>.72</td>
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<tr>
<td>Methyl tert-butyl ether</td>
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<td>1,1-Dichloroethene</td>
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<tr>
<td>2-Butoxane</td>
<td>J 63 J</td>
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<td>cis-1,2-Dichloroethene</td>
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<td>Bromochloromethane</td>
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<td>04/27/05</td>
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<tr>
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<td>1,2-Dichloroethane</td>
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<tr>
<td>1,1,1-Trichloroethane</td>
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<td>.72</td>
<td>18</td>
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<tr>
<td>1,1-Dichloroethylene</td>
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<td>Carbon tetrachloride</td>
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<td>Dichloroethene</td>
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<td>.79</td>
<td>18</td>
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<tr>
<td>1,2-Dichloropropene</td>
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<td>Trichloroethene</td>
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<tr>
<td>Bromochloromethane</td>
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<tr>
<td>cis-1,3-Dichloropropene</td>
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<tr>
<td>trans-1,3-Dichloropropene</td>
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<td>2,1,2-Trichloroethene</td>
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</tbody>
</table>

B - Analyte detected above the PQL in the associated Prep Blank.  
# - Outside control limits  
U - Undetected at the reported level.  
J - reported value is estimated.  
D - Result is diluted.  
E - concentration exceeded the calibration range and is estimated.
<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>Unit(s)</th>
<th>PQL</th>
<th>Analyzed Notes</th>
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<tr>
<td>1,2-Dichloroethane</td>
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<tr>
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<td>15</td>
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<tr>
<td>1,1,1,2-Tetrachloroethane</td>
<td>&lt;18</td>
<td>U</td>
<td>79</td>
<td>04/27/05</td>
<td></td>
</tr>
<tr>
<td>Chlorobenzene</td>
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<td>U</td>
<td>65</td>
<td>04/27/05</td>
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<tr>
<td>1-Chlorobenzene</td>
<td>&lt;18</td>
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<td>Ethylbenzene</td>
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<tr>
<td>Xylenes (total)</td>
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<td>Styrene</td>
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<td>U</td>
<td>72</td>
<td>01/27/05</td>
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<td>1,2,4,1-Tetrachloroethane</td>
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<td>1.2</td>
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<tr>
<td>1,2,3-Trichloropropane</td>
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<td>1.2</td>
<td>06/27/05</td>
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<tr>
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<td>U</td>
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<td>06/27/05</td>
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<tr>
<td>Bromobenzene</td>
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<td>n-Hexylbenzene</td>
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<td>U</td>
<td>65</td>
<td>04/27/05</td>
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<tr>
<td>2-Chloro-4-Chlorobenzene</td>
<td>&lt;18</td>
<td>U</td>
<td>1.2</td>
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<tr>
<td>1,3,5-Trimethylbenzene</td>
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<td>U</td>
<td>65</td>
<td>04/27/05</td>
<td></td>
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<tr>
<td>tert-Butylbenzene</td>
<td>&lt;18</td>
<td>U</td>
<td>18</td>
<td>06/27/05</td>
<td></td>
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<tr>
<td>n-Butylbenzene</td>
<td>&lt;18</td>
<td>U</td>
<td>18</td>
<td>06/27/05</td>
<td></td>
</tr>
<tr>
<td>1,3,4-Trimethylbenzene</td>
<td>&lt;18</td>
<td>U</td>
<td>18</td>
<td>04/27/05</td>
<td></td>
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<tr>
<td>sec-Butylbenzene</td>
<td>&lt;18</td>
<td>U</td>
<td>18</td>
<td>04/27/05</td>
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<td>1,3-Dichlorobenzene</td>
<td>&lt;18</td>
<td>U</td>
<td>18</td>
<td>04/27/05</td>
<td></td>
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<td>p-Isopropylbenzene</td>
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<td>U</td>
<td>18</td>
<td>04/27/05</td>
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<td>1,2-Dichlorobenzene</td>
<td>&lt;18</td>
<td>U</td>
<td>18</td>
<td>04/27/05</td>
<td></td>
</tr>
<tr>
<td>1,2,3-Naphthalene</td>
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<td>U</td>
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<td>04/27/05</td>
<td></td>
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<tr>
<td>1,3,4-Naphthalene</td>
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<td>U</td>
<td>18</td>
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<td></td>
</tr>
<tr>
<td>Naphthalene</td>
<td>&lt;15</td>
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<td>04/27/05</td>
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</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>&lt;15</td>
<td>U</td>
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</tr>
<tr>
<td>1,2,3-Trichlorobenzene</td>
<td>&lt;16</td>
<td>U</td>
<td>18</td>
<td>04/27/05</td>
<td></td>
</tr>
</tbody>
</table>

B - Analyte detected above the PQL in the associated Prep Blank.

# - Outside control limits: U - Underdected at the reported level.
J - reported value is estimated. D - Result is diluted.
E - contamination exceeded the calibration range and is estimated.

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0300

Author: [Signature]
Date: May 16, 2005

Monteith Sainucci
<table>
<thead>
<tr>
<th>Surrogate</th>
<th>%R</th>
<th>Quin</th>
<th>%R limits</th>
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</thead>
<tbody>
<tr>
<td>Dichlorofluoromethane (surrogate)</td>
<td>106</td>
<td>40 - 156</td>
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</tr>
<tr>
<td>1,2-Dichloroethane-14C (surrogate)</td>
<td>114</td>
<td>71 - 128</td>
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<tr>
<td>Toluene-13C6 (surrogate)</td>
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<td>70 - 125</td>
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<tr>
<td>Bromofluorobenzene (surrogate)</td>
<td>91</td>
<td>59 - 126</td>
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</tr>
</tbody>
</table>

Notes:

B - Analyte detected above the PQL in the associated Prep Blank.
# - Ovatile control limits U - Undetected at the reported level.
J - reported value is estimated. D - Result is diluted.
E - concentration exceeded the calibration range and is estimated.

Authorized: [Signature]  Date: May 16, 2005  Monica Santucci

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-9200
## Analytical Results

**Method**: 8260  
**Job No.**: 0055.008.3205  
**Certification NY No.**: 1015SR  
**Sample**: F4063  
**Sample Description**: SB-10  
**Instrument**: HP-5973 GC/MS3  
**Units**: ug/Kg Dry weight  
**Number of analytes**: 63

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
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B - Analyte detected above the PQL in the associated Prep Blank.  
# - Outside control limits  
U - Undetected at the reported level.  
J - reported value is estimated.  
D - Result is dimidil.  
E - concentration exceeded the calibration range and is estimated.
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B - Analyte detected above the PQL in the associated Prep Blank.  
# - Outside control limits  U - Undetected at the reported level. 
J - reported value is estimated.  D - Result is diluted.  
E - Demonstration exceeded the calibration range and is estimated. 

Authorized: [Signature]  
Date: May 16, 2005  
Monika Santucci
**O'Brien & Gere Laboratories, Inc.**

**Analytical Results**

**Method:** 8260

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<td>79 - 125</td>
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**Notes:**

- B - Analyte detected above the PQL in the associated Prep Blank
- # - Outside control limits
- U - Undetected at the reported level
- ? - Reported value is estimated
- D - Result is dilution
- E - Concentration exceeded the calibration range and is estimated

**Client:** Parsons  
**Project:** OUS-Knowl-Abus AFB  
**Proj. Date:** N7406-99-C-7022  
**Package #:** J4  
**Sample #:** F4063  
**Sample Description:** SB1-20  
**Instruments:** HP5973 GC/MSK3  
**Units:** wt/kg, Dry weight  
**Number of analyses:** 63  
**Job No.:** 0055, 008, 32206  
**Certification NY No.:** 10153R  
**Collected:** 04/23/05  
**Received:** 04/23/05  
**Prepared:** 04/27/05  
**Matrix:** Solid  
**QC Date:** 04/27/05  
**Sample Size:** 5.5 g  
**Dilution:** 1  
**Authorised:** (Signature)

**Date:** May 16, 2005  
**Manika Santucci**

5000 Edgewood Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 417-0200

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<table>
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<th>Parameter</th>
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<td>0.31</td>
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B - Analyte detected above the PQL in the associated Prep Blank.
# - Outside of control limits.
U - Undetected at the reported level.
F - reported value is estimated.
D - Result is diluted.
E - concentration exceeded the calibration range and is estimated.

Job No.: 0053 908.37206
Certification No.: 10155R
Collected: 04/27/05
Prepared: 04/27/05
QC Batch: 04/27/053
%Solid: 54.9
Sample Size: 1.1 g
Division: i
### Analytical Results

**Client:** Parsons  
**Project:** OUI-Biwali-Alta AFB  
**Program:** NA4008-99-C-7022  
**Sample:** F063 DL  
**Sample Description:** SP1-26  
**Instrument:** HP5973 GCMS63  
**Units:** ug/Kg, Dry weight  
**Number of analyses:** 63

#### Parameters

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**Job No.:** 0055. 008. 32206  
**Certification NY No.:** 10155R

**Collected:** 04/23/05  
**Received:** 04/23/06  
**Prepared:** 04/27/05  
**Q C Batch:** 0427G53  
**% Solids:** 54.9  
**Sample Size:** 1.1 g  
** Dilution:** 1

B - Analyte detected above the PQL in the associated Prep Filtrate.  
# - Outside control limits  
U - Undetected at the reported level.  
J - Reported value re-investigated.  
D - Result is diluted.  
2 - Concentration exceeded the calibration range and is estimated.

Authorized: [Signature]

Date: May 16, 2005

Monika Santorri

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syosset, NY 11771 / (315) 437-0700

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<table>
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<td>1,2-Dichloroethene-d4 (surrogate)</td>
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Notes:
- B - Analyte detected above the PQL in the associated Prep Blank.
- # - Outside control limits U - Undetected at the reported level.
- J - Reported value is estimated. D - Result is diluted.
- E - Concentration exceeded the calibration range and is estimated.

5000 Buxtonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-8010
### Analytical Results

**Method:** 8260  
**Job No.:** 0055. OIR. 12206  
**Certification NY No.:** 10155R

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<th>Parameter</th>
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<td>1,2-Dichloroethane</td>
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- **P**: Analyte detected above the PQL in the associated Prep Blank.  
- **d**: Outside control limits  
- **U**: Undetected at the reported level  
- **J**: reported value is estimated  
- **D**: Result is diluted  
- **R**: Concentration exceeded the calibration range and is estimated.

**Authorized:** [Signature]

**Date:** May 15, 2005  
**Monika Stanocci**

5000 Brintnield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 477-0200

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## Analytical Results

**Method:** 8260

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<th>Parameter</th>
<th>Result</th>
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<th>mpp</th>
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**B** - Analyte detected above the PQL in the associated Prep Blank.

**#** - Outside control limits. **U** - Undetected at the reported level.

**J** - Reported value is estimated. **D** - Result is diluted.

**E** - Concentration exceeded the calibration range and is estimated.

**Date:** May 16, 2005

**Author:** F. Santucci

**Company:** O'Brien & Gere Laboratories, Inc.

**Project:** GUE-Blewitz-Aber AFB

**Lot No.:** 0555 - 058-32206

**Certification NY No.:** 101858

**Sample Description:** SSU-2.1

**Sample Size:** 5.22 g

**Instrument:** HP5973 GCMS903

**Units:** ug/Kg Dry weight

**Number of analytes:** 63
O'Brien & Gere Laboratories, Inc.

Analytical Results
Method: 8260

Job No.: 0055 008 32206
Certification NY No.: 10151R

Collected: 04/22/05
Matrix: Solid

Received: 04/23/05
QC Batch: 042705S3

Prepared: 04/27/05
%Solids: 78.5

Sample Size: 5.22 g
Dilution: 1

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<th>Qual</th>
<th>%R Limits</th>
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<td>Toluene-d8 (surrogate)</td>
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<tr>
<td>Bromofluorobenzene (surrogate)</td>
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<td>1,3-Dibromobenzene (surrogate)</td>
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Notes:

B - Analyte detected above the POL in the associated Prep Blank.
θ - Outside control limits
U - Undetected at the reported level.
J - Reported value is estimated. D - Result is diluted.
E - Concentration exceeded the calibration curve and is estimated.

5000 Baumfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-2200

Authorized: <Signature>  M. Sannucci  Date: May 16, 2005
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</tbody>
</table>

B - Analyte detected above the PQL in the associated Prep Blank
# - Outside control limits U - Undetected at the reported level)
J - reports value is estimated  D - Result is diluted
E - concentration exceeded the calibration curve and is estimated

Job No: D05, 005-32206
Certificate No: 10153
Sample: 10153
Sample Description: SR3-15
Instrument: HP6973 GC/MS/MS
Precision: 04/27/05
Units: mg/Kg, Dry weight
Number of analyses: 63

Prepared: 04/27/05
MQS: 71.0
Analytical Results Method: 8260
Client: Parsons
Project: OUI-Bioass-Alhos AFB
Prep Desc: H-N18-06-59-C-7022
Sample: 14015
Sample Description: SR3-15
Instrument: HP6973 GC/MS/MS
Units: mg/Kg, Dry weight
Number of analyses: 63

Collected: 04/22/05
MQS: 71.0
Sample Size: 5.18 g
Dilution: 1

Authorized: 
Date: May 16, 2005
Memika Samisch

5000 Brintonfield Parkway, Suite 10, Doral, FL 33069/ Sarasota, NY 12222/ (315) 437-5200
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<td>6.8</td>
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</table>

B: Analyte detected above the PQL in the associated Prep Blends.
S: Outside control limits U: Undetected as the reported level
J: Reported value is estimated, D: Result is diluted.
E: Concentration exceeded the calibration range and is estimated.

Date: May 16, 2005
Mona Santucci
**Analytical Results**

**Method:** 8260

**Job No.:** 0055-008-32206
**Certification No.:** 10155R

<table>
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<tr>
<th>Surrogate</th>
<th>%R</th>
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<th>Limits</th>
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<td>1,2-Dichloroethane-d4 (surrogate)</td>
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<td>Toluene-d8 (surrogate)</td>
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<td>Bromofluorobenzene (surrogate)</td>
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<td>59 - 129</td>
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**Notes:**

B - Analyte detected above the NOL in the associated Prep Blank.
# - Outside control limits. U - Undetected at the reported level.
J - Reported value is estimated. D - Result is dilute.

Authorized: [Signature]
Date: May 16, 2005

Monica Santucci

---

5000 Brintonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 677-0200
### Analytical Results

**Method:** 8260

**Job No.:** 0055. 008.32206  
**Certification NY No.:** 1015R

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<tr>
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<tr>
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</table>

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- # - Outside control limits U - Undetected at the reported level.
- J - reported value estimated. D - Result is diluted.
- E - concentration exceeded the calibration range and is estimated.

**Authorised:** [Signature]  
**Date:** May 16, 2005  
**Monika Samuoli**

5000 Brittenfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13212 / (315) 437-0200
### Analytical Results

**Method:** 8260

**Client:** O'Brien & Gere Laboratories, Inc.

**Project:** BU-TiO2-Alum AF3

**Proj Desc:** NA7468-95-C-7022

**Package #:** J14

**Sample:** F4065 DL

**Sample Description:** SB2-15

**Instrument:** HP5973 GC/MSM3

**Units:** up/Kg/Dry weight

**Number of analyses:** 63

**Collected:** 04/22/05

**Received:** 04/23/05

**Prepared:** 04/27/05

**Matrix:** Solid

**QC Batch:** 040740M53

**Sample Size:** 1.9 g

**Dilution:** 1

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<th>Qual.</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed</th>
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<tr>
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<td>9.3</td>
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<td>2-Bromoform</td>
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<td>2.9</td>
<td>9.3</td>
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<td>9.3</td>
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</table>

B - Analyte detected above the PQL. In the associated Prop Blank.
# - Outside control limits U - Undetected at the reported level.
J - reported value is estimated. D - Result is diluted.
E - concentration exceeded the calibration range and is estimated.

**Date:** May 16, 2005

**Authorised:** Monica Sanzoni

**5000 Britfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200**
O'Brien & Gere Laboratories, Inc.

Analytical Results
Method: 8260

Job No.: 0055.008.32206
Certification NY No.: X015R

Collected: 04/22/05
Received: 04/23/05
Prepared: 04/27/05

Client: Parsons
Project: OUI-Bioxell-Almes AFS
Proj. Desc: N47458-99-C-7022
Package: 134
Sample: P4065 DL
Sample Description: SB2-15
Instrument: HP5973 GCM/MS
Units: ug/kg (Dry weight)
Number of analyses: 63

Matrix: Solid
QC Batch: 042705S3
%Solid: 71.0
Sample Size: 1.9 g
Dilution: 1

### Surrogate

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<tr>
<th>Surrogate</th>
<th>%</th>
<th>Qual</th>
<th>%R Limits</th>
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<tr>
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<td>40 - 156</td>
</tr>
<tr>
<td>1,2-Dichloroethane-d8 (surrogate)</td>
<td>114</td>
<td></td>
<td>71 - 126</td>
</tr>
<tr>
<td>Toluene-d8 (surrogate)</td>
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<td>95 - 125</td>
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<td>Benzo[a]fluoranthene (surrogate)</td>
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<td>69 - 128</td>
</tr>
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Notes:

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Authorized: [Signature]
Date: May 16, 2005
Manaska Santocci

5000 Brittania Parkway / Suite 100, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
**Analytical Results**

**Method:** 8260

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
<th>Analyzed Notes</th>
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<tr>
<td>Methylene chloride</td>
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<td>1,1,1-Trichloroethane</td>
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<td>2-Butanone</td>
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</table>

- **B**: Analyte detected above the PQL in the associated Prep Blank.
- **B#**: Outside control limits
- **U**: Undetected at the reported level
- **J**: Reported value is estimated.
- **D**: Results diluted.
- **E**: Concentration exceeded the calibration range and is estimated.

**Client:** Parsons  
**Project:** OU1-Barnwell-Almus AFB  
**Proj. Desc:** N47406-9F-C-7022  
**Package:** 156  
**Sample:** F4066  
**Sample Description:** SB2-20  
**Instrument:** HP5973 GC/MS/S3  
**Units:** ug/Kg Dry weight  
**Number of analyses:** 63  
**Job No.:** 0055 . 008 . 32206  
**Certification NY No.:** 10115R  
**Collected:** 04/22/05  
**Received:** 04/23/05  
**Prepared:** 04/27/05  
**Matrix:** Solid  
**QC Batch:** 04/27/05S3  
**Ns/Id:** 80.1  
**Sample Size:** 511 g  
**Dilution:** 1  

**Authorizing:** MParc  
**Date:** May 16, 2005  
5000 Brightonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13211 / (315) 437-0200
<table>
<thead>
<tr>
<th>Parameter</th>
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<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
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B - Analyte detected above the PQL in the associated Prep Blank.
# - Outside control limits  U - Undetected at the reported level.
I - reported value is estimated  D - Result is diluted.
E - Concentration exceeded the calibration range and is estimated.
Client: Persons
Project: OUI-Bipwell-Aims APB
Proj. Date: 11740-08-99-C-322
Package: 134
Sample: P4066
Sample Description: SBC-20
Instrument: HP5973 GCMSQX
Units: ug/kg
Dry weight
Number of analytes: 63

Notes:

Analytical Results
Method: 8260
Job No.: 0265-08.32296
Certification NY No.: 10155R
Collected: 04/22/05
Received: 04/23/05
Prepared: 04/27/05
Sample Size: 5.11 g
Matrix: Solid
QC Batch: 042705S3
%Recovery: 90.3
Dilution: 1

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<th>% Rel. Limits</th>
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<td>69 - 156</td>
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<td>1,2-Dichloroethane-d4 (surrogate)</td>
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<td>Toluene-d8 (surrogate)</td>
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<tr>
<td>Bromofluorobenzene (surrogate)</td>
<td>82</td>
<td>69 - 126</td>
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</table>

B - Analyte detected above the PQL in the associated Prep Blank.
# - Outside control limits U - Undetected at the reported level.
J - Reported value is estimated. D - Blank is diluted.
E - Concentration exceeded the calibration range and is estimated.

Authorized: [Signature]
Date: May 16, 2005

5000 Brittenfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
### Analytical Results
**Method:** 8260

**Client:** O'Brien & Gere Laboratories, Inc.

**Project:** OU1-Browa-Aillis AF6

**Prop. Desc:** NA7405-99-C-7022

**Package#:** 134

**Sample:** F4067

**Sample Description:** SB12-7

**Instruments:** HP979 GC/MS#3

**Units:** ug/kg Dry weigh

**Number of analytes:** 63

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<th>Parameter</th>
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<th>Qual</th>
<th>MDL</th>
<th>PQL</th>
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<tr>
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<tr>
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<td>.19</td>
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<td></td>
<td>04/27/05</td>
<td></td>
</tr>
</tbody>
</table>

**B** - Analyte detected above the PQL in the associated Prep Blank.

**#** - Outside control limits U - Undetected at the reported level.

**J** - Reported value is estimated. **D** - Result is diluted.

**E** - Concentration exceeded the calibration range and is estimated.

**Job No.:** 0055 . 008 . 3206

**Certification NY No.:** 10:15R

**Sample Size:** 5.19 g

**Dilution:** 1

**Collected:** 04/22/05

**Prepared:** 04/27/05

**Received:** 04/23/05

**QC Batch:** 042705S3

**QA/Attd.:** 64.6

**Date:** May 16, 2005

**Author:** Monika Santoro

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13211 / (315) 437-0200

53
### Analytical Results

**Method:** 8260

**Client:** Parsons  
**Project:** OU1-Bioequ-Altus AFB  
**Proj. Date:** 07/14/96  
**Package: #1**  
**Sample #:** 94097  
**Sample Description:** SB4-7  
**Instrument:** HP5973 GC/MS/MS  
**Units:** ug/Kg Dry weight  
**Number of analyses:** 63  

**Collected:** 07/22/05  
**Prepared:** 07/22/05  
**Matrix:** Solid  
**QC Rank:** 04270553  
**Sample Size:** 5.19 g  
**Dilution:** 1

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<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>PDL</th>
<th>Analyzed Notes</th>
</tr>
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<tr>
<td>1,2-Dichloroethane</td>
<td>&lt;3.7</td>
<td>U</td>
<td>.13</td>
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<td>U</td>
<td>.12</td>
<td>3.7</td>
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<td>1,2,3-Trichlorobenzene</td>
<td>&lt;7.5</td>
<td>U</td>
<td>.78</td>
<td>7.5</td>
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</tbody>
</table>

B - Analyte detected above the PDL in the associated Prep Blank.  
# - Outside control limits  
U - Undetected at the reported level.  
J - reported value is estimated.  
D - Result is dilute.  
E - Concentration exceeded the calibration range and is estimated.

**Authorized:**  
**Date:** May 16, 2005  
**Monika Stankov**

5000 Brintenfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200

---

54
<table>
<thead>
<tr>
<th>Surrogate</th>
<th>%R</th>
<th>Quel</th>
<th>%R Limits</th>
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<td>1,2-Dichloromethane-d4 (surrogate)</td>
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<tr>
<td>Dichloromethane (surrogate)</td>
<td>107</td>
<td>40 - 156</td>
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</tbody>
</table>

Notes:

B - Analyte detected above the PQL in the associated Prep Blanks.
# - Oxide control limits U - Undetected at the reported level.
I - reported value is estimated. D - Result is diluted.
E - concentration exceeded the calibration range and is estimated.

Authorized: Monika Santucci
Date: May 18, 2005

2000 Briarfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
## Analytical Results

**Method:** 8260  

**Client:** Parsons  
**Project:** O&U-Below-Ash FAB  
**Proj. Desc:** N47048-95-C-7022  
**Package:** 135  
**Sample:** E4068  
**Sample Discription:** QC Trip Blank  
**Instrument:** HP5973 GCM/SIF  
**Unit:** mg/L  
**Number of analytes:** 63  

### Parameter Results

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MDL</th>
<th>POL</th>
<th>Analyzed</th>
<th>Notes</th>
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<td>1.0</td>
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<tr>
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<td>Methylene chloride</td>
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<tr>
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<td>.023</td>
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<td>.074</td>
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<td>U</td>
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**Job No.:** 0005.008.32206  
**Certification No.:** 1015-SR  
**Collected:** 04/18/05  
**Received:** 04/23/05  
**Prepared:** 04/28/05  
**Matrix:** Water  
**QC Batch:** 04205071  
**Sample Size:** 10 mL  
**Dilution:** 1  

**B:** Analyte detected above the PQL.  
**F:** Outside control limits  
**U:** Undetected at the reported level  
**-** Reported value is estimated.  
**D:** Result is diluted.  
**-** Concentration exceeded the calibration range and is estimated.

Authorized: [Signature]

Date: May 3, 2003

Monika Santucci

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syosset, NY 11791 / (315) 437-0200
### Analytical Results
**Method:** 8260

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Result</th>
<th>Qual</th>
<th>MQL</th>
<th>PQL</th>
<th>Analyzed</th>
<th>Notes</th>
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<td>1,3,5-Trimethylbenzene</td>
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</tbody>
</table>

- B - Analyte detected above the PQL in the associated Prep Blank.
- # - Outside control limits
- U - Undetected at the reported level.
- *(J)* - reported value is estimated.
- D - Result is diluted.
- E - concentration exceeded the calibration range and is estimated.

**Client:** Parsons  
**Project:** OUJ-Blisswell-Alhus AFB  
**Proj. Desc.:** N47408-99-C-7022  
**Package:** 135  
**Sample:** P4008  
**Sample Description:** QC Trip Blank  
**Instrument:** HP5973 GC/MS#1  
**Units:** ug/L  
**Number of analytes:** 63

**Job No.:** 0055, 006, 32206  
**Certification NY No.:** 1015-SR  
**Collected:** 04/18/05  
**Received:** 04/23/05  
**Prepared:** 04/28/05  
**Matrix:** Water  
**Sample Size:** 10 mL  
**QC Batch:** 042805W1  
**%Solids:** 1

**Reported by:** [Signature]  
**Date:** May 5, 2005  
**Manager:** Stanucci

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200

57
O’Brien & Gere Laboratories, Inc.

Client: Parsons
Project: OII-Bowd-Arms AFB
Proj. Date: N47408-99-C-7022
Package#: 153
Sample: P4068
Sample Description: QC Trip Blank
Instrument: HP5973 GC/MS#1
Units: ug/L
Number of analytes: 63

Analytical Results
Method: 8260

Job-No.: 0035.008.32205
Certification NY No.: 10158R

Collected: 04/18/05
Received: 04/23/05
Prepared: 04/28/05

Matrix: Water
QC Batch: 042805W1
%Solids:
Sample Size: 10 mL
Dilution: 1

Surrogate                 %R    Qual% Limits
1,1-Dichloroethane (surrogate)  101  75 - 127
1,2-Dichloroethane-d4 (surrogate)  99  75 - 134
Toluene-d8 (surrogate)            100  75 - 125
Bromofluorobenzene (surrogate)    91  75 - 125

Notes:

B - Analyte detected above the PQL in the associated Prep Blank
# - Outside control limits U - Undetected at the reported level.
- reported value is estimated. D - Result is diluted.
E - concentration exceeded the calibration range and is estimated.

Authorized: [Signature]
Date: May 3, 2005
Monika Sanucci

5000 Brittonfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13221 / (315) 437-0200
### Analytical Results

**Wet Chemistry**

<table>
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<th>Parameter</th>
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<th>PQL Analyzed</th>
<th>QC Batch</th>
<th>Dil</th>
<th>Note</th>
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**Notes:**

- D: Analyte detected above the PQL in the associated Prep Blank
- U: Undetected at the reported level
- R: Result is diluted
- E: Concentration exceeded the calibration range and is estimated

**Job No.:** 0055.008.32206  
**Certification NY No.:** 10155R

Authorized: [Signature]  
Date: May 16, 2006  
Monika Santucci

5000 Brittainfield Parkway / Suite 300, Box 4942 / Syracuse, NY 13232 / (315) 437-0000
### Analytical Results

**Wet Chemistry**

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B - Analyte detected above the PQL in the associated Prep Blank  
H - Undetected at the reported level.  
J - Reported value is estimated.  
D - Result is diluted.  
E - Concentration exceeded the calibration range and is estimated.

Authorized: [Signature]  
Date: May 16, 2005  
Monika Santucci

---

5000 Brittonfield Parkway, Suite 300, Box 6942, Syracuse, NY 13221 / (315) 437-0200
### Analytical Results

**Wet Chemistry**

**Client:** Parsons  
**Project:** OUI-Hipwall-Alza AFB  
**Prog Desc:** M47408-99-C-7022  
**Package:** J34  
**Sample:** F4067  
**Samp. Description:** SB12-7

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<th>pQL Analyzed</th>
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**Notes:**

- B - Analyte detected above the PQL in the associated Prep Blank
- U - Undetected at the reported level
- J - Reported value is estimated  
- D - Result is diluted
- E - Concentration exceeded the calibration range and is estimated

**Authorized:** Monica Savicci  
**Date:** May 16, 2005

5000 Britenfield Parkway / Suite 300, Box 4947 / Syracuse, NY 13222 / (315) 437-0200
Analytical Analysis Report

Client: Bruce Henry
Parsons Engineering Science
1700 Broadway
Suite 900
Denver, CO 80290

Phone: (303) 831-8100

Fax:

MI Identifier: 062CD

Date Rec: 04/23/2005

Report Date: 07/27/2005

Client Project #: 738863.04000

Client Project Name: OU1 Biowall - Altus AFB

Analysis Requested: VFA

Project: OU1 Biowall - Altus AFB

Comments:

All samples within this data package were analyzed under U.S. EPA Good Laboratory Practice Standards: Toxic Substances Control Act (40 CFR part 790). All samples were processed according to standard operating procedures. Test results submitted in this data package meet the quality assurance requirements established by Microbial Insights, Inc.

Reported By: 

Reviewed By: 

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## Analysis Summary Report

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<tr>
<th>Sample Name:</th>
<th>Date Sampled:</th>
<th>Date Received:</th>
<th>Arrival Condition:</th>
<th>Metabolic Acids (mg/L)</th>
<th>Pyruvic</th>
<th>Lactic</th>
<th>Formic</th>
<th>Acetic</th>
<th>Propionic</th>
<th>Butyric</th>
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## Quality Control Report

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<th>MS Recovery %</th>
<th>MSD Recovery %</th>
<th>RPD %</th>
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<td>Butyric</td>
<td>90.7</td>
<td>85.3</td>
<td>6.1</td>
<td>88.3</td>
</tr>
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</table>
Analytical Analysis Report

Client: Bruce Henry  
Parsons Engineering Science  
1700 Broadway  
Suite 900  
Denver, CO 80290  

Phone: (303) 831-8100  
Fax:  

MI Identifier: 063CD  
Date Rec: 04/23/2005  
Report Date: 07/27/2005  

Client Project #: 735833.08000  
Client Project Name: OU1 Altus AFB  
Analysis Requested: VFA  

Project: OU1 Altus AFB  
Comments: Subcontract #735833-30001-00

All samples within this data package were analyzed under U.S. EPA Good Laboratory Practice Standards: Toxic Substances Control Act (40 CFR part 790). All samples were processed according to standard operating procedures. Test results submitted in this data package meet the quality assurance requirements established by Microbial Insights, Inc.

Reported By:  
Reviewed By:  

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## Analysis Summary Report

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Date Sampled</th>
<th>Date Received</th>
<th>Arrival Condition</th>
<th>Pyruvic</th>
<th>Lactic</th>
<th>Formic</th>
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Quality Control Report

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Shuttling Capacity of control 0.5 g/L IHSS Soil Humic Acid Standard 0.2475 mM Fe reduced
Laboratory Results

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Microseps test results meet all the requirements of the NELAC standards.

Approved By

The analytical results reported here are reliable and usable to the precision expressed in this report. As required by some regulating authorities, a full discussion of the uncertainty in our analytical results can be obtained at our website or through customer service. Unless otherwise specified, all results are reported on a wet weight basis.

As a valued client we would appreciate your comments on our service. Please call customer service at (412)826-5245 or email bhans@microseps.com

Case Narrative

The percent recovery for the batch MS/MSD analyses for chloride and sulfate was outside of control limits. The unspiked sample concentration was greater than 4 times the spike added. All other QC analyses were acceptable. No further action taken. One of the percent RPDs for the batch duplicate analyses was outside of control limits. The original result (a non-Parsons sample) was just under the PQL and the duplicate value was just above the PQL. Differences at this level are within the expected method error.

220 William Pitt Way • Pittsburgh, PA 15238 • Tel 412-826-5245 • Fax 412-826-3433
website www.microseps.com email info@microseps.com
### Analyte(s)

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#### WetChem

- **N Chloride**
  - M 250.0 5.0 mg/L 9056 4/28/05 23:20 md
  - Nitrate-Nitrite as N
    - 0.240 0.050 mg/L HACH-8192 4/27/05 tld
  - SolubleOrganic Carbon
    - 12.000 5.000 mg/L 9060 4/28/05 md
- **N Sulfate**
  - M 1600.0 10.0 mg/L 9056 4/29/05 0:04 md
  - Total Dissolved Solids(TDS)
    - 2400 10 mg/L 160.1 4/25/05 tld
  - Total Hardness as CaCO3
    - 1300 000 100 000 mg/L 130.2 4/26/05 rh

#### RiskAnalysis

- Ethane
  - 55 000 5.000 ng/L AM20GAX 5/2/05 rw
- Ethene
  - 71 000 5.000 ng/L AM20GAX 5/2/05 rw
- Methane
  - 13 000 0.015 ug/L AM20GAX 5/2/05 rw

---

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
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Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
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| RiskAnalysis            |      |        |     |       |            |                |    |
| Ethane                  |      | 5.100  | 5.000| ng/L  | AM20GAX    | 5/2/05         | rw |
| Ethene                  |      | 1600.000| 5.000| ng/L  | AM20GAX    | 5/2/05         | rw |
| Methane                 |      | 14000.000| 0.015| ug/L  | AM20GAX    | 5/2/05         | rw |

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**Risk Analysis**

- Ethane: 32 000 ng/L AM20GAX 5/2/05 rw
- Ethene: 36 000 ng/L AM20GAX 5/2/05 rw
- Methane: 20 000 ug/L AM20GAX 5/2/05 rw

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| Risk Analysis                      |      |        |     |       |          |                |    |
| Ethane                             |      | 31.000 | 5000| ng/L  | AM20GAX  | 5/2/05         | rw |
| Ethene                             |      | 340.000| 5000| ng/L  | AM20GAX  | 5/2/05         | rw |
| Methane                            |      | 13000.00| 0.015| ug/L  | AM20GAX  | 5/2/05         | rw |

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NEAC sample acceptance criteria, L - Subcontracted Lab used, N - NEAC certified analysis
### Sample Description
- **PES-MP11**
- **Matrix**: Water

### Lab Sample #
- **P0504366-07**

### Sampled Date/Time
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### Received
- **25 Apr 05 9:51**

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<td>N Chloride</td>
<td>M</td>
<td>350.0</td>
<td>5</td>
<td>mg/L</td>
<td>9056</td>
<td>4/29/05</td>
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</tr>
<tr>
<td>Nitrate-Nitrite as N</td>
<td>J</td>
<td>0.033</td>
<td>0.050</td>
<td>mg/L</td>
<td>HACH-8192</td>
<td>4/27/05</td>
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<tr>
<td>Soluble Organic Carbon</td>
<td>M</td>
<td>320.000</td>
<td>50.000</td>
<td>mg/L</td>
<td>9060</td>
<td>4/30/05</td>
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<tr>
<td>N Sulfate</td>
<td>M</td>
<td>180.0</td>
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<td>mg/L</td>
<td>9056</td>
<td>4/29/05</td>
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<tr>
<td>Total Dissolved Solids(TDS)</td>
<td></td>
<td>2300</td>
<td>10</td>
<td>mg/L</td>
<td>160.1</td>
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<tr>
<td>Total Hardness as CaCO3</td>
<td></td>
<td>1600.00</td>
<td>100.00</td>
<td>mg/L</td>
<td>130.2</td>
<td>4/26/05</td>
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### Risk Analysis

<table>
<thead>
<tr>
<th>Analyte(s)</th>
<th>Flag</th>
<th>Result</th>
<th>PQL</th>
<th>Units</th>
<th>Method #</th>
<th>Analysis Date</th>
<th>By</th>
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<tbody>
<tr>
<td>Ethane</td>
<td>U</td>
<td>&lt; 5.000</td>
<td>5.000</td>
<td>ng/L</td>
<td>AM20GAX</td>
<td>5/2/05</td>
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<td>Ethene</td>
<td></td>
<td>3000.000</td>
<td>5.000</td>
<td>ng/L</td>
<td>AM20GAX</td>
<td>5/2/05</td>
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<tr>
<td>Methane</td>
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<td>ug/L</td>
<td>AM20GAX</td>
<td>5/2/05</td>
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</table>

**Data Qualifiers**: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
**Prep Method:** Filterable Residue Gravimetric Dried at 180 Degrees C

**Analysis Method:** Filterable Residue Gravimetric Dried at 180 Degrees C

<table>
<thead>
<tr>
<th>M050426010-MB</th>
<th>Result</th>
<th>True Spike Conc.</th>
<th>RDL</th>
<th>% Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Dissolved Solids (TDS)</td>
<td>&lt; 10 mg/L</td>
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<td>NA</td>
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</table>

<table>
<thead>
<tr>
<th>M050426010-LCS</th>
<th>Result</th>
<th>True Spike Conc.</th>
<th>% Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Dissolved Solids (TDS)</td>
<td>180 mg/L</td>
<td>177.00</td>
<td>102.00</td>
<td>79 - 116</td>
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</table>

<table>
<thead>
<tr>
<th>P0504366-07A-DUP</th>
<th>Result</th>
<th>True Spike Conc.</th>
<th>% Recovery</th>
<th>Ctl Limits</th>
<th>RPD</th>
<th>RPD Ctl Limits</th>
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</thead>
<tbody>
<tr>
<td>Total Dissolved Solids (TDS)</td>
<td>2500 mg/L</td>
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<td>NA</td>
<td>8.33</td>
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</table>

Outlined Results indicate results outside of Control limits.
**Prep Method:** Total Hardness Titrametric EDTA  
**Analysis Method:** Total Hardness Titrametric EDTA

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>RDL</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>M050427012-MB</td>
<td>&lt; 10.00 mg/L</td>
<td>10.00</td>
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<tr>
<td>M050427012-LCS</td>
<td>110.00 mg/L</td>
<td>117.00</td>
<td>94.00</td>
<td>70 - 130</td>
<td>-</td>
</tr>
<tr>
<td>P0504366-06A-DUP</td>
<td>1900.00 mg/L</td>
<td>-</td>
<td>-</td>
<td>5.41</td>
<td>0 - 20</td>
</tr>
</tbody>
</table>

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
### M050427038-MB

<table>
<thead>
<tr>
<th>Test</th>
<th>Result</th>
<th>Spike Conc.</th>
<th>RDL</th>
<th>%Recovery</th>
<th>Ctrl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrate-Nitrite as N</td>
<td>&lt; 0.050</td>
<td>mg/L</td>
<td>0.050</td>
<td>-</td>
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</table>

### M050427038-LCS

<table>
<thead>
<tr>
<th>Test</th>
<th>Result</th>
<th>Spike Conc.</th>
<th>%Recovery</th>
<th>Ctrl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrate-Nitrite as N</td>
<td>2.400</td>
<td>mg/L</td>
<td>80.00</td>
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### P0504366-04A-MS

<table>
<thead>
<tr>
<th>Test</th>
<th>Result</th>
<th>Spike Conc.</th>
<th>%Recovery</th>
<th>Ctrl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrate-Nitrite as N</td>
<td>0.140</td>
<td>mg/L</td>
<td>61.00</td>
<td>57 - 145</td>
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### P0504366-04A-MSD

<table>
<thead>
<tr>
<th>Test</th>
<th>Result</th>
<th>Spike Conc.</th>
<th>%Recovery</th>
<th>Ctrl Limits</th>
<th>RPD</th>
<th>RPD Ctrl Limits</th>
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</thead>
<tbody>
<tr>
<td>Nitrate-Nitrite as N</td>
<td>0.150</td>
<td>mg/L</td>
<td>66.00</td>
<td>57 - 145</td>
<td>6.90</td>
<td>0 - 18</td>
</tr>
</tbody>
</table>

Outlined Results indicate results outside of Control limits

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD. SAM/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis.
### Prep Method:
Soluble Organic Carbon

### Analysis Method:
Soluble Organic Carbon

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>RDL</th>
<th>%Recovery</th>
<th>Ctrl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>M050429011-MB</td>
<td>&lt; 5.000 mg/L</td>
<td>5 000</td>
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<td>M050429011-LCS</td>
<td>Soluble Organic Carbon</td>
<td>21.000 mg/L</td>
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<td>80 - 120</td>
</tr>
<tr>
<td>P0504366-01A-DUP</td>
<td>Soluble Organic Carbon</td>
<td>15 000 mg/L</td>
<td>- NA</td>
<td>19.0</td>
<td>0 - 20</td>
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<tr>
<td>P0504366-01A-MS</td>
<td>Soluble Organic Carbon</td>
<td>34 000 mg/L</td>
<td>20.00</td>
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<tr>
<td>P0504366-01A-MSD</td>
<td>Soluble Organic Carbon</td>
<td>35 000 mg/L</td>
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</table>

Outlined Results indicate results outside of Control limits

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAM/P/DUP, B - detected in blank, S - field sample as received did not meet NEIAC sample acceptance criteria, L - Subcontracted Lab used, N - NEIAC certified analysis
<table>
<thead>
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<th>Ctrl Limits</th>
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<th>RPD Ctrl Limits</th>
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<tbody>
<tr>
<td>M050430006-MB</td>
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<td>5000</td>
<td>-</td>
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<tr>
<td>P0504368-02A-DUP</td>
<td>48000</td>
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<td>-</td>
<td>NA</td>
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<tr>
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<tr>
<td>Soluble Organic Carbon</td>
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<td>80.00</td>
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<td>P0504375-03A-MS</td>
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<td>2000</td>
<td>105.0</td>
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<tr>
<td>P0504437-04A-MS</td>
<td>26000</td>
<td>2000</td>
<td>114.0</td>
<td>70 - 130</td>
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<td>70 - 130</td>
<td>3.17</td>
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<td>Soluble Organic Carbon</td>
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</table>

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMPI/DUP, B - detected in blank, S - field sample as received did not meet NEIAC sample acceptance criteria, L - Subcontracted Lab used, N - NEIAC certified analysis
### P0504375-03A-MSD

<table>
<thead>
<tr>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
<th>RPD</th>
<th>RPD Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soluble Organic Carbon</td>
<td>49 mg/L</td>
<td>20.00</td>
<td>105.00</td>
<td>70 - 130</td>
<td>0.00</td>
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</tbody>
</table>

### P0504437-04A-MSD

<table>
<thead>
<tr>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
<th>RPD</th>
<th>RPD Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soluble Organic Carbon</td>
<td>26.000 mg/L</td>
<td>20.00</td>
<td>114.00</td>
<td>70 - 130</td>
<td>0.00</td>
</tr>
</tbody>
</table>

Outlined Results indicate results outside of Control limits

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAM/P/DUP, B - detected in blank, S - Field sample as received did not meet NEIAC sample acceptance criteria, L - Subcontracted Lab used, N - NEIAC certified analysis
<table>
<thead>
<tr>
<th></th>
<th>Result</th>
<th>TrueSpikeConc</th>
<th>RDL</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soluble Organic Carbon</td>
<td>&lt; 5 000 mg/L</td>
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<td>5.000</td>
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<tr>
<td>M050502003-LCS</td>
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<tr>
<td>Soluble Organic Carbon</td>
<td>20.000 mg/L</td>
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<td>19.70</td>
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<td>80 - 120</td>
</tr>
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<td>P0504454-01A-DUP</td>
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<tr>
<td>Soluble Organic Carbon</td>
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</tr>
<tr>
<td>Soluble Organic Carbon</td>
<td>25.000 mg/L</td>
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<td>20.00</td>
<td>102.00</td>
<td>70 - 130</td>
</tr>
<tr>
<td>P0504454-01A-MS</td>
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</tr>
<tr>
<td>Soluble Organic Carbon</td>
<td>26.000 mg/L</td>
<td></td>
<td>20.00</td>
<td>107.00</td>
<td>70 - 130</td>
</tr>
</tbody>
</table>

Data Qualifiers: 1 - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis.
**Prep Method:** In House Dissolved Gas Sample Preparation

**Analysis Method:** Analysis of C1-C4 Hydrocarbons in Water

### M050502018-MB

<table>
<thead>
<tr>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>RDL</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethene</td>
<td>&lt; 0.025 ug/L</td>
<td>0.025</td>
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<tr>
<td>Ethane</td>
<td>&lt; 0.025 ug/L</td>
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<td>NA</td>
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</table>

### M050502018-LCS

<table>
<thead>
<tr>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethene</td>
<td>22000 000 ng/L</td>
<td>19,445.00</td>
<td>113.00</td>
</tr>
<tr>
<td>Ethane</td>
<td>23000 000 ng/L</td>
<td>20,843.00</td>
<td>110.00</td>
</tr>
</tbody>
</table>

### M050502018-LCSD

<table>
<thead>
<tr>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
<th>RPD</th>
<th>RPD Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethene</td>
<td>21000 000 ng/L</td>
<td>19,445.00</td>
<td>108.00</td>
<td>75 - 125</td>
<td>4.65</td>
</tr>
<tr>
<td>Ethane</td>
<td>23000 000 ng/L</td>
<td>20,843.00</td>
<td>110.00</td>
<td>75 - 125</td>
<td>0.00</td>
</tr>
</tbody>
</table>

### P0504329-02A-MS

<table>
<thead>
<tr>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethene</td>
<td>21000 000 ng/L</td>
<td>19,445.00</td>
<td>108.00</td>
</tr>
<tr>
<td>Ethane</td>
<td>22000 000 ng/L</td>
<td>20,843.00</td>
<td>105.00</td>
</tr>
</tbody>
</table>

### P0504329-02A-MSD

<table>
<thead>
<tr>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
<th>RPD</th>
<th>RPD Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ethene</td>
<td>21000 000 ng/L</td>
<td>19,445.00</td>
<td>108.00</td>
<td>70 - 130</td>
<td>0.00</td>
</tr>
<tr>
<td>Ethane</td>
<td>22000 000 ng/L</td>
<td>20,843.00</td>
<td>105.00</td>
<td>70 - 130</td>
<td>0.00</td>
</tr>
</tbody>
</table>

Data Qualifiers: I - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAM/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
### Prep Method: In House Dissolved Gas Sample Preparation
### Analysis Method: Analysis of Dissolved Methane in Water

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Result</th>
<th>TrueSpikeConc</th>
<th>RDL</th>
<th>%Recovery</th>
<th>Ctrl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>M050502019-MB</td>
<td>&lt; 0.100</td>
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- **Outlined Results** indicate results outside of Control limits.
### Prep Method:
Anions by ion chromatography

### Analysis Method:
Anions by ion chromatography

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Outlined Results indicate results outside of Control limits

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
Laboratory Results

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Microseeps test results meet all the requirements of the NELAC standards.

Approved By: __________________________________________

The analytical results reported here are reliable and usable to the precision expressed in this report. As required by some regulating authorities, a full discussion of the uncertainty in our analytical results can be obtained at our web site or through customer service. Unless otherwise specified, all results are reported on a wet weight basis.

As a valued client we would appreciate your comments on our service. Please call customer service at (412)826-5245 or email bhans@microseeps.com

Case Narrative
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| OrganicsPrep            |       |        |     |                  |          |               |    |
| Percent Solids          |       | 60    | 1.0 | %                | 160.3    | 4/27/05       | jb |

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
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| OrganicsPrep                |      |        |     |       |          |                |    |
| Percent Solids             | 63   | 1.0    | %   | 160.3 | 4/27/05 | jb             |    |

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<td>0.02</td>
<td>% dry</td>
<td>WC43</td>
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<td>md</td>
</tr>
<tr>
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<td>U</td>
<td>&lt; 0.01</td>
<td>0.01</td>
<td>% dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
<tr>
<td>Weak Acid Soluble Ferric Iron</td>
<td>U</td>
<td>&lt; 0.01</td>
<td>0.01</td>
<td>% dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
<tr>
<td>Weak Acid Soluble Ferrous Iron</td>
<td>U</td>
<td>0.03</td>
<td>0.01</td>
<td>% dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
</tbody>
</table>

**Organics Prep**

| Percent Solids | 86 | 1 | % | 160.3 | 4/27/05 | jb |

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
<table>
<thead>
<tr>
<th>Analyte(s)</th>
<th>Flag</th>
<th>Result</th>
<th>PQL</th>
<th>Units</th>
<th>Method #</th>
<th>Analysis Date</th>
<th>By</th>
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</thead>
<tbody>
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<td>0.24</td>
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<tr>
<td>Organic Carbon</td>
<td></td>
<td>18000</td>
<td>32</td>
<td>mg/Kg dry</td>
<td>3.2.13</td>
<td>4/26/05</td>
<td>rh</td>
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<td>U</td>
<td>&lt;0.02</td>
<td>0.02</td>
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<td>7/8/05</td>
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<tr>
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<td>&lt;0.02</td>
<td>0.02</td>
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<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
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<tr>
<td>Strong Acid Soluble Ferrous Iron</td>
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<td>0.02</td>
<td>% dry</td>
<td>WC43</td>
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<tr>
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<td>0.01</td>
<td>0.01</td>
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<td>0.01</td>
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<td>7/8/05</td>
<td>md</td>
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<tr>
<td>Weak Acid Soluble Ferrous Iron</td>
<td></td>
<td>0.12</td>
<td>0.01</td>
<td>% dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
<tr>
<td>OrganicsPrep</td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Percent Solids</td>
<td></td>
<td>78</td>
<td>1</td>
<td>%</td>
<td>160.3</td>
<td>4/27/05</td>
<td>jb</td>
</tr>
</tbody>
</table>

Data Qualifiers:  J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
<table>
<thead>
<tr>
<th>Sample Description</th>
<th>Matrix</th>
<th>Lab Sample #</th>
<th>Sample Date/Time</th>
<th>Received Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>SB2-20</td>
<td>Solid</td>
<td>P0504379-06</td>
<td>22 Apr. 05 9:55</td>
<td>25 Apr. 05 13:09</td>
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</tbody>
</table>

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<th>Result</th>
<th>PQL</th>
<th>Units</th>
<th>Method #</th>
<th>Analysis Date</th>
<th>By</th>
</tr>
</thead>
<tbody>
<tr>
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</tr>
<tr>
<td>Organic Carbon</td>
<td>20000</td>
<td>32</td>
<td>mg/Kg dry</td>
<td>3.2.13</td>
<td>4/26/05</td>
<td>rh</td>
<td></td>
</tr>
<tr>
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<td>0.02</td>
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<td>7/8/05</td>
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<td></td>
</tr>
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<td>0.02</td>
<td>% dry</td>
<td>WC43</td>
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<td>0.02</td>
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<td>7/8/05</td>
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</tr>
<tr>
<td>Weak Acid Soluble Divalent Manganese</td>
<td>&lt; 0.01</td>
<td>0.01</td>
<td>% dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
<td></td>
</tr>
<tr>
<td>Weak Acid Soluble Ferric Iron U</td>
<td>&lt; 0.01</td>
<td>0.01</td>
<td>% dry</td>
<td>WC43</td>
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<td></td>
</tr>
<tr>
<td>Weak Acid Soluble Ferrous Iron</td>
<td>0.31</td>
<td>0.01</td>
<td>% dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
<td></td>
</tr>
</tbody>
</table>

| Organics                  |      |        |     |       |          |                |    |
| Percent Solids            | 78   | 1      | %  | 160.3 | 4/27/05  | jb             |    |

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
<table>
<thead>
<tr>
<th>Sample Description</th>
<th>Matrix</th>
<th>Lab Sample #</th>
<th>Sampled Date/Time</th>
<th>Received Date/Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>SB12-7</td>
<td>Solid</td>
<td>P0504379-07</td>
<td>22 Apr. 05 9:00</td>
<td>25 Apr. 05 13:09</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Analyte(s)</th>
<th>Flag</th>
<th>Result</th>
<th>PQL</th>
<th>Units</th>
<th>Method #</th>
<th>Analysis Date</th>
<th>By</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid Volatile Sulfide</td>
<td>1.4</td>
<td>0.15</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>rh</td>
</tr>
<tr>
<td>Chromium Extractable Sulfide</td>
<td>1.2</td>
<td>0.15</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/9/05</td>
<td>tld</td>
</tr>
<tr>
<td>Organic Carbon</td>
<td>23000</td>
<td>37</td>
<td>mg/Kg</td>
<td>dry</td>
<td>3.2 13</td>
<td>4/26/05</td>
<td>rh</td>
</tr>
<tr>
<td>Strong Acid Divalent Manganese</td>
<td>&lt; 0.03</td>
<td>0.03</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
<tr>
<td>Strong Acid Ferric Iron</td>
<td>0.04</td>
<td>0.03</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
<tr>
<td>Strong Acid Soluble Ferrous Iron</td>
<td>0.18</td>
<td>0.03</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
<tr>
<td>Weak Acid Soluble Divalent Manganese</td>
<td>&lt; 0.01</td>
<td>0.01</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
<tr>
<td>Weak Acid Soluble Ferric Iron</td>
<td>&lt; 0.01</td>
<td>0.01</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
<tr>
<td>Weak Acid Soluble Ferrous Iron</td>
<td>0.06</td>
<td>0.01</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
</tr>
</tbody>
</table>

**OrganicsPrep**

| Percent Solids | 67 | 1 | % | 160.3 | 4/27/05 | jb |

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
**Prep Method:** Organic Carbon (Walkley-Black)  
**Analysis Method:** Organic Carbon (Walkley-Black)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>RDL</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>M050427011-MB</td>
<td>&lt; 25.00 mg/Kg</td>
<td>25.00</td>
<td></td>
<td>-</td>
<td>NA</td>
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<tr>
<td>P0504379-04A-DUP</td>
<td>15000.00 mg/Kg</td>
<td></td>
<td></td>
<td>-</td>
<td>14.29</td>
</tr>
</tbody>
</table>

Data Qualifiers:  
J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAM/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
Prep Method: Total Residue Gravimetric Dried at 103-105 degrees C
Analysis Method: Total Residue Gravimetric Dried at 103-105 degrees C

There are no QC Samples in this Batch

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
Prep Method: Total Residue Gravimetric Dried at 103-105 degrees C
Analysis Method: Total Residue Gravimetric Dried at 103-105 degrees C

There are no QC Samples in this Batch
Prep Method: Chromium Extractable Sulfide
Analysis Method: Chromium Extractable Sulfide

There are no QC Samples in this Batch

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAM/P/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
<table>
<thead>
<tr>
<th>M050713005-LCS</th>
<th>Result</th>
<th>TrueSpikeConc.</th>
<th>%Recovery</th>
<th>Ctl Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid Volatile Sulfide</td>
<td>28.60 %</td>
<td>32.90</td>
<td>87.00</td>
<td>50 - 150</td>
</tr>
</tbody>
</table>

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis.
Prep Method: Weak Acid Soluble Metals
Analysis Method: Weak Acid Soluble Metals

There are no QC Samples in this Batch
Prep Method: Strong Acid Soluble Metals
Analysis Method: Strong Acid Soluble Metals

There are no QC Samples in this Batch
August 31, 2005

Ms. Carmen Lebron
NFESC ESC411
1100 23rd Avenue
Port Hueneme, CA 93043

Subject: Technical Memorandum: Bioavailable Iron Analytical Results for Subsurface Investigation of A Mulch Biowall at LF03, Altus Air Force Base, Oklahoma.

Dear Ms. Lebron:

Please find enclosed two copies of the subject technical memorandum. This technical memorandum was prepared by Parsons for the Naval Facilities Engineering Command, Port Hueneme, California. An electronic copy of the technical memorandum has been sent to you via email.

If you have any questions or comments on this deliverable, please call me at (970) 244-8829 or Mr. Bruce Henry at (303) 831-8100.

Sincerely,
PARSONS

John F. Hall, P.E.
Project Manager

cc: Mr. Bruce Henry, Parsons – (1 copy)
    Mr. Pat Evans, CDM – (1 copy)
    File: 735833.08000
This technical memorandum provides analytical results for bioavailable ferric iron and manganese in mulch biowall samples collected at Landfill 3 (LF03), Altus Air Force Base (AFB), Oklahoma. These results are an amendment to the report prepared by Parsons titled *Subsurface Investigation of a Mulch Biowall at LF03, Altus Air Force Base, Oklahoma In Support of: Environmental Security Technology Certification Program Project CU-0316: Enhanced Bioremediation of cis-Dichloroethene (cis-DCE) and Vinyl Chloride (VC) Using Electron Shuttle, July 2005.*

The location of the mulch biowall and the sampling locations at LF-03 are shown on Figures 1 and 2, respectively (attached). Samples were collected on 22 April 2005. Sampling procedures are described in the above referenced report. Analytical results for bioavailable ferrous iron and bioavailable manganese are provided in the attached laboratory report and are summarized on Table 1 (attached).
Figure 1. Location of Mulch Biowall Relative to TCE Plume (isoconcentration contours in micrograms per liter of TCE in April 1999. (CDM, 2004)
Figure 2. Monitoring Well Sampling Locations Relative to Mulch Biowall. (CDM, 2004)
<table>
<thead>
<tr>
<th>Sample Location</th>
<th>Sample Date</th>
<th>Sample Depth (feet bgs) (a/)</th>
<th>Percent Solids</th>
<th>Bio-available Ferric Iron (b/) (mg/kg) (c/)</th>
<th>Bio-available Manganese (b/) (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SB1-5</td>
<td>22-Apr-05</td>
<td>5</td>
<td>60%</td>
<td>441</td>
<td>12.3</td>
</tr>
<tr>
<td>SB1-15</td>
<td>22-Apr-05</td>
<td>15</td>
<td>63%</td>
<td>622</td>
<td>10.0</td>
</tr>
<tr>
<td>SB1-20</td>
<td>22-Apr-05</td>
<td>20</td>
<td>64%</td>
<td>645</td>
<td>13.0</td>
</tr>
<tr>
<td>SB2-7</td>
<td>22-Apr-05</td>
<td>7</td>
<td>86%</td>
<td>290</td>
<td>&lt;5.0 (d/)</td>
</tr>
<tr>
<td>SB12-7 (duplicate)</td>
<td>22-Apr-05</td>
<td>7</td>
<td>67%</td>
<td>13.6</td>
<td>&lt;7.5</td>
</tr>
<tr>
<td>SB2-15</td>
<td>22-Apr-05</td>
<td>15</td>
<td>78%</td>
<td>182</td>
<td>7.2</td>
</tr>
<tr>
<td>SB2-20</td>
<td>22-Apr-05</td>
<td>20</td>
<td>78%</td>
<td>&lt;6.4</td>
<td>&lt;6.4</td>
</tr>
</tbody>
</table>

\(a/\) feet bgs = feet below ground surface.

\(b/\) B-AFe = bio-available ferric iron; B-AMn = bio-available manganese.

\(c/\) mg/kg = micrograms per kilogram dry weight.

\(d/\) <5.0 indicates that the analyte was not detected above the indicated method detection limit.
Lab Sample # | Client Sample ID
--- | ---
P0504379-01 | SB1-5
P0504379-02 | SB1-15
P0504379-03 | SB1-20
P0504379-04 | SB2-7
P0504379-05 | SB2-15
P0504379-06 | SB2-20
P0504379-07 | SB12-7

Microseps test results meet all the requirements of the NELAC standards.  

Approved By: [Signature]

The analytical results reported here are reliable and usable to the precision expressed in this report. As required by some regulating authorities, a full discussion of the uncertainty in our analytical results can be obtained at our web site or through customer service. Unless otherwise specified, all results are reported on a wet weight basis.

As a valued client we would appreciate your comments on our service. Please call customer service at (412) 826-5245 or email bhans@microseps.com

Case Narrative

220 William Pitt Way • Pittsburgh, PA 15238 • Tel 412-826-5245 • Fax 412-826-3433
website www.microseps.com email info@microseps.com
<table>
<thead>
<tr>
<th>Sample Description</th>
<th>Matrix</th>
<th>Lab Sample #</th>
<th>Sampled Date/Time</th>
<th>Received</th>
<th>Analyte(s)</th>
<th>Flag</th>
<th>Result</th>
<th>PQL</th>
<th>Units</th>
<th>Method #</th>
<th>Analysis Date</th>
<th>By</th>
</tr>
</thead>
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<td>SB1-5</td>
<td>Solid</td>
<td>P0504379-01</td>
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<td>25 Apr. 05 13:09</td>
<td>WetChem</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>1.3</td>
<td>0.2</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>rh</td>
</tr>
<tr>
<td>Bio-Available Ferric Iron</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>441</td>
<td>8.3</td>
<td>mg/Kg dry</td>
<td>dry</td>
<td>BAFelll</td>
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<td>md</td>
</tr>
<tr>
<td>Bio-Available Manganese</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>12.3</td>
<td>8.3</td>
<td>mg/Kg dry</td>
<td>dry</td>
<td>BAFelll</td>
<td>7/14/05</td>
<td>md</td>
</tr>
<tr>
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<td></td>
<td></td>
<td></td>
<td>1.9</td>
<td>0.2</td>
<td>%</td>
<td>dry</td>
<td>WC43</td>
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<td></td>
<td></td>
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<td>42</td>
<td>mg/Kg dry</td>
<td>3.2.13</td>
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<td>BAFelll</td>
<td>WC43</td>
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<td>md</td>
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<tr>
<td>Strong Acid Divalent Manganese</td>
<td>U</td>
<td>&lt; 0.03</td>
<td>0.03</td>
<td>% dry</td>
<td>WC43</td>
<td>7/8/05</td>
<td>md</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Strong Acid Ferric Iron</td>
<td>U</td>
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Data Qualifiers:  
J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAM/P/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
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Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
### Prep Method:
Organic Carbon (Walkley-Black)

### Analysis Method:
Organic Carbon (Walkley-Black)

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<tr>
<th>Sample Code</th>
<th>Result</th>
<th>True Spike Conc.</th>
<th>RDL</th>
<th>% Recovery</th>
<th>Ctrl Limits</th>
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<tbody>
<tr>
<td>M050427011-MB</td>
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<tr>
<td>P0504379-04A-DUP</td>
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<td>14.29</td>
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</table>

Outlined Results indicate results outside of Control limits

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAM/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
Prep Method: Total Residue Gravimetric Dried at 103-105 degrees C
Analysis Method: Total Residue Gravimetric Dried at 103-105 degrees C

There are no QC Samples in this Batch

Outlined Results indicate results outside of Control limits

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
Prep Method: Total Residue Gravimetric Dried at 103-105 degrees celsius
Analysis Method: Total Residue Gravimetric Dried at 103-105 degrees celsius

There are no QC Samples in this Batch
Prep Method: Chromium Extractable Sulfide
Analysis Method: Chromium Extractable Sulfide

There are no QC Samples in this Batch
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<td>Acid Volatile Sulfide</td>
<td>28.60%</td>
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<td>87.00%</td>
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Prep Method: Acid Volatile Sulfide
Analysis Method: Acid Volatile Sulfide

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAMP/DUP, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis
Prep Method: Weak Acid Soluble Metals
Analysis Method: Weak Acid Soluble Metals

There are no QC Samples in this Batch
There are no QC Samples in this Batch
M050817023-MB

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P0504379-04A-DUP

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<td>- NA</td>
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<td>0 - 30</td>
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</table>

Outlined Results indicate results outside of Control limits.

Data Qualifiers: J - estimated value, U - Non detect, R - Poor surrogate recovery, M - Recovery/RPD poor for MS/MSD, SAM/DEU, B - detected in blank, S - field sample as received did not meet NELAC sample acceptance criteria, L - Subcontracted Lab used, N - NELAC certified analysis.
**CHAIN OF CUSTODY RECORD**

**Microseeps, Inc. - 220 William Pitt Way - Pittsburgh, PA 15238**

<table>
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<th>Company</th>
<th>Parsons</th>
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<tbody>
<tr>
<td>Co. Address</td>
<td>1700 Broadway, Denver, CO 80205</td>
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<tr>
<td>Proj. Manager</td>
<td>John Hull / Bruce Henry</td>
</tr>
<tr>
<td>Proj. Location</td>
<td>Altus AFB</td>
</tr>
<tr>
<td>Proj. Number</td>
<td>736-883</td>
</tr>
<tr>
<td>Phone #</td>
<td>303 831 8100</td>
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**Sampler's Signature:**

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<th>Time</th>
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**Remarks**

**Relinquished by:**

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**Relinquished by:**

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**WHITE COPY** Accompany Samples  **YELLOW COPY** Laboratory File  **PINK COPY** Submitter
# Chain of Custody Record

**Microseeps, Inc.** - 220 William Pitt Way - Pittsburgh, PA 15238

**Company:** Parsons  
**Co. Address:** 1700 Broadway Ste 900, Denver CO 80206

**Proj. Manager:** John Hall (Bruce Henry)  
**Proj. Location:** Altus AFB

**Proj. Number:** 735882.080000  
**Phone #:** 970-844-8000  
**Fax #:** 970-844-8899

**Sampler's signature:** [Signature]

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<thead>
<tr>
<th>Sample ID</th>
<th>Sample Description</th>
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**Company:** Parsons

**Relinquished by:** [Signature]  
**Company:** Parsons

**Relinquished by:** [Signature]  
**Company:** Parsons

**Company:**  
**Date:** 04/05  
**Time:** 15:00  
**Received by:** [Signature]

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**WHITE COPY:** Accompany Samples  
**YELLOW COPY:** Laboratory File  
**PINK COPY:** Submitter
Appendix C
Commercial Sources of Electron Shuttles
## Appendix C

### Commercial Sources of Electron Shuttles

#### Table C1

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<tr>
<th>Supplier</th>
<th>Contact</th>
<th>Phone</th>
<th>Product</th>
<th>Form</th>
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<th>Fulvic Acid</th>
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<td>TeraVita</td>
<td>Erik Morgan/Eric Danese</td>
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<td>80</td>
<td>5</td>
<td>Leonardite</td>
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<tr>
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<td>717 291 7251 x202</td>
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Appendix D
Soil Boring Logs
Approximately 4-inches of asphalt at surface. Linear ripes in some locations may be indicative of former building foundation. Ridges not present at this location.

No soil cuttings were derived or observed for this interval. Description is based on historic land use and building information.

Silty sand, fine, micaceous, dark gray, loose, wet.

Sandy silt, trace clay, dark gray, soft, wet.

Sandy silt, with clay, dark gray, soft, wet.

Temporary piezometer installed for groundwater sample collection. Abandoned after sample collection with hydrated bentonite pellets and asphalt patch.

Collected composite soil sample 11GP24 (10-20') at 1155 for APCL analyses: 6010B/3050B (As, Fe, Mn, V) and SW960. Additional soil samples were submitted to CDM in Bellevue, WA and the USGS for analyses to be chosen.

REMARKS

Approximately 4-inches of asphalt at surface. Linear rilles in some locations may be indicative of former building foundation. Ridges not present at this location. No soil cuttings were derived or observed for this interval. Description is based on historic land use and building information.

Silty sand, fine, micaceous, dark gray, loose, wet.

Silt and sand, fine, micaceous, dark gray, loose, wet.

Silty sand, fine, micaceous, dark gray, soft, wet.

Sandy silt, micaceous, dark gray trace red, redoxamorphic iron mottling, soft, wet.

strong natural organic (sewage) odor

END OF BORING 25' BGS

REMARKS

Collected composite soil sample 11GP25 (10-22') at 1035 for APCL analyses: 6010B/3050B (As, Fe, Mn, V) and SW960. Additional soil samples were submitted to CDM in Bellevue, WA and the USGS for analyses to be chosen.

Collected groundwater sample 11GP25 at 1336 for APCL analyses: 8260B/5030B, 6010B/3010A, CA 8015 modified, 300.0, 160.1, 376.1, RSK-175, and 415.1.

Reviewed by: Holly Carter

Date:
approximately 4-inches of asphalt at surface. Linear rignes in some locations may be indicative of former building foundation. Ridges not present at this location.

No soil cuttings were derived or observed for this interval. Description is based on historic land use and building information.

Sand, well-graded, fine to medium, trace clay (lenses <5mm), grayish brown, trace red oxidizable iron mottles in clay, loose, wet.

Sand, well-graded, fine to medium, trace clay, brownish gray, loose, wet.

Sand, well-graded, fine to medium, with silt, grayish brown, loose, wet. (poor recovery, lithology inferred from cuttings on the ground)

Sandy silt, with clay, soft, wet. (no recovery, lithology inferred from cuttings on the ground)

**EXPLANATION OF ABBREVIATIONS**

**DRILLING METHODS:**
- HSA: Hollow Stem Auger
- SSA: Solid Stem Auger
- HA: Hand Auger
- AR: Air Rotary
- MR: Mud Rotary
- DP: Direct Push

**SAMPLING TYPES:**
- SH: Slide Hammer
- GP: Geoprobe
- SS: Split Spoon

**OTHER:**
- BGS: Below Ground Surface
- BZ: Breathing Zone
- SH: Sample Head Space
- NR: No Reading/Not Recorded
- NA: Not Applicable
- PID: photo-ionization detector
- ppm: parts per million

**REMARKS**

Collected composite soil sample 11GP26 (10-22') at 1340 for APCL analyses: 6010B/3050B (As, Fe, Mn, V) and SW960. Additional soil samples were submitted to CDM in Bellevue, WA and the USGS for analyses to be chosen.

Collected groundwater sample 11GP26 at 1553 for APCL analyses: 8260B/5030B, 6010B/3010A, CA 8015 modified, 300.0, 160.1, 376.1, RSK-175, and 415.1.

Reviewed by: Date:
GS
Approximately 4-inches of asphalt at surface. Linear riges in some locations may be indicative of former building foundation. Ridges not present at this location. No soil cuttings were derived or observed for this interval. Description is based on historic land use and building information.

Sandy gravel, poorly-graded, fine, subrounded to subangular gravel, fine to medium sand, with clay, brown, wet.

Sand, well-graded, fine to medium, trace gravel, fine, subrounded to subangular, grayish brown, loose, wet.

Sand, poorly-graded, fine to medium, gray, loose, wet.

Silty sand, fine to medium, micaceous, gray, loose, wet.

SP
SM
END OF BORING 25' BGS

REMARKS
Collected composite soil sample 11GP27 (14-22') and 11GP27 (14-22') at 1500 for APCL analyses: 6010B/3050B (As, Fe, Mn, V) and SW960. Additional soil samples were submitted to CDM in Bellevue, WA and the USGS for analyses to be chosen.

Collected groundwater sample 11GP27 at 1825 for APCL analyses: 8260B/5030B, 6010B/3010A, CA 8015 modified, 300.0, 160.1, 376.1, RSK-175, and 415.1.

Reviewed by: Date:
Appendix E
Analytical Data
# APCL Analytical Report

Service ID #: 801-052580  
Received: 05/04/05
Collected by:  
Extracted: N/A
Collected on: 05/03/05  
Tested: 05/05-12/05
Reported: 05/20/05

Sample Description: Soil and Water  
Project Description: 6215.001  
SPAWAR ESTCP.

## Analysis of Water and Soil Samples

### I. Analysis of Water Samples

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<th>11GP25 05-02580-2</th>
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<td>TETRACHLOROETHENE (PCE)</td>
<td>8260B</td>
<td>µg/L</td>
<td>0.5</td>
<td>&lt;13  &lt;25</td>
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</tr>
<tr>
<td>TRICHLOROETHENE (TCE)</td>
<td>8260B</td>
<td>µg/L</td>
<td>0.5</td>
<td>&lt;13  &lt;25</td>
<td></td>
<td></td>
</tr>
<tr>
<td>VINYL CHLORIDE</td>
<td>8260B</td>
<td>µg/L</td>
<td>0.5</td>
<td>2,700 (&lt;d) 1,670 (&lt;b)</td>
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<td>Dilution Factor</td>
<td></td>
<td></td>
<td></td>
<td>5    5</td>
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<tr>
<td>METHANE</td>
<td>RSK175</td>
<td>µg/L</td>
<td>3</td>
<td>200  140</td>
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<td>ETHANE</td>
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<td>µg/L</td>
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<td>&lt;5   &lt;5</td>
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<tr>
<td>ETHENE</td>
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<td>µg/L</td>
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<td>89   27</td>
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<td>11MW07 TRIP BLANK</td>
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<td></td>
<td>05-02580-11 05-02580-12</td>
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<td>BROMIDE</td>
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<td>HARDNESS</td>
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<td>NITRATE AS N</td>
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<tr>
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<td>SOLIDS, TOTAL DISSOLVED (TDS)</td>
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## APCL Analytical Report

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<thead>
<tr>
<th>Component Analyzed</th>
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<th>PQL</th>
<th>11MW07 05-02580-11</th>
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<tr>
<td>CHLORIDE</td>
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<td>Dilution Factor</td>
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<tr>
<td>ARSENIC</td>
<td>6010B</td>
<td>µg/L</td>
<td>5</td>
<td>&lt;5</td>
<td>-</td>
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<tr>
<td>IRON</td>
<td>6010B</td>
<td>µg/L</td>
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<td>3,970</td>
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<td>MANGANESE</td>
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<td>VANADIUM</td>
<td>6010B</td>
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<td>1.3J</td>
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<tr>
<td>PHC AS GASOLINE</td>
<td>M801SV</td>
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<td>0.05</td>
<td>1.51 (a)</td>
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<td>VOLATILE ORGANICS</td>
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<tr>
<td>CIS-1,2-DICHLOROETHENE</td>
<td>8260B</td>
<td>µg/L</td>
<td>0.5</td>
<td>1,000 (c)</td>
<td>&lt;0.5</td>
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<tr>
<td>TETRACHLOROETHENE (PCE)</td>
<td>8260B</td>
<td>µg/L</td>
<td>0.5</td>
<td>&lt;0.5</td>
<td>&lt;0.5</td>
</tr>
<tr>
<td>TRICHLOROETHENE (TCE)</td>
<td>8260B</td>
<td>µg/L</td>
<td>0.5</td>
<td>&lt;0.5</td>
<td>&lt;0.5</td>
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<tr>
<td>VINYL CHLORIDE</td>
<td>8260B</td>
<td>µg/L</td>
<td>0.5</td>
<td>613 (c)</td>
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<td>Dilution Factor</td>
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<td></td>
</tr>
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<td>METHANE</td>
<td>RSK175</td>
<td>µg/L</td>
<td>3</td>
<td>140</td>
<td>-</td>
</tr>
<tr>
<td>ETHANE</td>
<td>RSK175</td>
<td>µg/L</td>
<td>5</td>
<td>&lt;25</td>
<td>-</td>
</tr>
<tr>
<td>ETHENE</td>
<td>RSK175</td>
<td>µg/L</td>
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<td>130</td>
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### II. Analysis of Soil Samples

<table>
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<tr>
<th>Component Analyzed</th>
<th>Method</th>
<th>Unit</th>
<th>PQL</th>
<th>11GP24(10-20) 05-02580-6</th>
<th>11GP25(10-22) 05-02580-7</th>
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</thead>
<tbody>
<tr>
<td>MOISTURE</td>
<td>ASTM-D2216</td>
<td>%Moisture</td>
<td>0.5</td>
<td>23.2</td>
<td>21.0</td>
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<tr>
<td>CARBON, TOTAL ORGANIC (MAX)</td>
<td>9060</td>
<td>mg/kg</td>
<td>100</td>
<td>550</td>
<td>4,970</td>
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<td>CARBON, TOTAL ORGANIC (MIN)</td>
<td>9060</td>
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<td>250</td>
<td>1,250</td>
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<td>CARBON, TOTAL ORGANIC (AVE)</td>
<td>9060</td>
<td>mg/kg</td>
<td>100</td>
<td>400</td>
<td>2,970</td>
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<td>Dilution Factor</td>
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<td>ARSENIC</td>
<td>6010B</td>
<td>mg/kg</td>
<td>0.3</td>
<td>0.31</td>
<td>0.55</td>
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<tr>
<td>IRON</td>
<td>6010B</td>
<td>mg/kg</td>
<td>3</td>
<td>9,830</td>
<td>11,000</td>
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<td>MANGANESE</td>
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<td>mg/kg</td>
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<td>100</td>
<td>117</td>
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<td>VANADIUM</td>
<td>6010B</td>
<td>mg/kg</td>
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<td>30.6</td>
<td>34.5</td>
</tr>
<tr>
<td>Component Analyzed</td>
<td>Method</td>
<td>Unit</td>
<td>PQL</td>
<td>11GP26(10-22)</td>
<td>11GP27(14-22)</td>
</tr>
<tr>
<td>--------------------</td>
<td>---------</td>
<td>------</td>
<td>-----</td>
<td>---------------</td>
<td>---------------</td>
</tr>
<tr>
<td>MOISTURE</td>
<td>ASTM-D2216</td>
<td>%Moisture</td>
<td>0.5</td>
<td>25.1</td>
<td>21.4</td>
</tr>
<tr>
<td>CARBON, TOTAL ORGANIC (MAX)</td>
<td>9060</td>
<td>mg/kg</td>
<td>100</td>
<td>1,300</td>
<td>930</td>
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<td>CARBON, TOTAL ORGANIC (MIN)</td>
<td>9060</td>
<td>mg/kg</td>
<td>100</td>
<td>360</td>
<td>&lt;100</td>
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<td>CARBON, TOTAL ORGANIC (AVE)</td>
<td>9060</td>
<td>mg/kg</td>
<td>100</td>
<td>660</td>
<td>363</td>
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**Dilution Factor**

<table>
<thead>
<tr>
<th>Component Analyzed</th>
<th>Method</th>
<th>Unit</th>
<th>PQL</th>
<th>11GP26(10-22)</th>
<th>11GP27(14-22)</th>
<th>11GP27(14-22)DUP</th>
</tr>
</thead>
<tbody>
<tr>
<td>ARSENIC</td>
<td>6010B</td>
<td>mg/kg</td>
<td>0.3</td>
<td>0.46</td>
<td>0.48</td>
<td>0.45</td>
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<tr>
<td>IRON</td>
<td>6010B</td>
<td>mg/kg</td>
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<td>6,230</td>
<td>4,300</td>
<td>6,090</td>
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<td>MANGANESE</td>
<td>6010B</td>
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<td>57.9</td>
<td>41.8</td>
<td>63.9</td>
</tr>
<tr>
<td>VANADIUM</td>
<td>6010B</td>
<td>mg/kg</td>
<td>0.5</td>
<td>20.8</td>
<td>13.2</td>
<td>18.6</td>
</tr>
</tbody>
</table>


N.D.: Not Detected or less than the practical quantitation limit.  "..": Analysis is not required.

J: Reported between PQL and MDL.

Listed Dilution Factors (DF) are relative to the method default DF.  All unlisted DF's are 1.0

(a) Not a gasoline pattern, sample chromatogram only contained one peak which might be originated from VOC's.

(b) Analyzed with a dilution factor of 1000.
(c) Analyzed with a dilution factor of 100.
(d) Analyzed with a dilution factor of 250.

Respectfully submitted,

Dominic Lau  
Laboratory Director  
Applied P & CH Laboratories

CADHS ELAP No.: 1431  NELAP No.:02114CA  CL-0787 D022 R 05-2580
### Bioavailable Ferric Iron Assay Report

**Client:** ESTCP/NAVFACCO  
**Site:** SPAWAR  
**Sample Collection Date:** 5/3/2005  
**Analyst:** DAB/PJE  
**Analysis Date:** 6/14/2005

<table>
<thead>
<tr>
<th>Sample</th>
<th>Solids (%)</th>
<th>Ambient Fe II (mg/kg)</th>
<th>Bioavailable Fe III (mg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>11GP24</td>
<td>NA</td>
<td>553</td>
<td>1382</td>
</tr>
<tr>
<td>11GP24 dup</td>
<td>NA</td>
<td>553</td>
<td>910</td>
</tr>
<tr>
<td>11GP25</td>
<td>NA</td>
<td>1290</td>
<td>1694</td>
</tr>
<tr>
<td>11GP25 dup</td>
<td>NA</td>
<td>1281</td>
<td>908</td>
</tr>
<tr>
<td>11GP26</td>
<td>NA</td>
<td>369</td>
<td>1267</td>
</tr>
<tr>
<td>11GP26 dup</td>
<td>NA</td>
<td>369</td>
<td>1521</td>
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<tr>
<td>11GP27</td>
<td>NA</td>
<td>276</td>
<td>634</td>
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<tr>
<td>11GP27 dup</td>
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<td>267</td>
<td>597</td>
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Table E1
University of Massachusetts Electron Shuttling Capacity Data

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<tr>
<th>No.</th>
<th>Supplier</th>
<th>Product</th>
<th>Form</th>
<th>Test Conc.</th>
<th>Conc. Units</th>
<th>Shuttling Capacity Fe Reduced (mM)</th>
<th>Shuttling Capacity % of 0.5 g/L IHSS Soil Humic Acid standard</th>
<th>Shuttling Capacity Fe Reduced (mM) for duplicates</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>TeraVita</td>
<td>SP-85</td>
<td>85% soluble solid</td>
<td>1.0</td>
<td>g/L</td>
<td>0.233</td>
<td>0.053</td>
<td>32% 79</td>
</tr>
<tr>
<td>2</td>
<td>Luscar Ltd</td>
<td>Dry Soluble 80</td>
<td>100% soluble solid</td>
<td>1.0</td>
<td>g/L</td>
<td>0.222</td>
<td>0.008</td>
<td>5% 76</td>
</tr>
<tr>
<td>3</td>
<td>Luscar Ltd</td>
<td>Liquid 12000</td>
<td>12% Liquid</td>
<td>8.3</td>
<td>mL/L</td>
<td>0.174</td>
<td>0.025</td>
<td>21% 59</td>
</tr>
<tr>
<td>4</td>
<td>Triad</td>
<td>Huma K</td>
<td>100% soluble solid</td>
<td>1.0</td>
<td>g/L</td>
<td>0.186</td>
<td>0.030</td>
<td>20% 63</td>
</tr>
<tr>
<td>5</td>
<td>Live Earth Products</td>
<td>12% Liquid Humic Acid</td>
<td>12% Liquid</td>
<td>8.3</td>
<td>mL/L</td>
<td>0.189</td>
<td>0.042</td>
<td>32% 76</td>
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<tr>
<td>6</td>
<td>Monterey AgResources</td>
<td>HA-12</td>
<td>12% Liquid</td>
<td>8.3</td>
<td>mL/L</td>
<td>0.243</td>
<td>0.008</td>
<td>5% 98</td>
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<tr>
<td>7</td>
<td>Humate International</td>
<td>Humate AS</td>
<td>100% soluble solid</td>
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<td>g/L</td>
<td>0.141</td>
<td>0.008</td>
<td>9% 57</td>
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<tr>
<td>8</td>
<td>Humus Products of America</td>
<td>15% Concentrated Liquid Humus</td>
<td>15% Liquid</td>
<td>6.7</td>
<td>mL/L</td>
<td>0.158</td>
<td>0.023</td>
<td>21% 51</td>
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<tr>
<td>9</td>
<td>LignoTech</td>
<td>BorreGro HA-2</td>
<td>100% soluble solid</td>
<td>1.0</td>
<td>g/L</td>
<td>0.171</td>
<td>0.013</td>
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<td>LignoTech</td>
<td>BorreGro HA-1</td>
<td>100% soluble solid</td>
<td>1.0</td>
<td>g/L</td>
<td>0.084</td>
<td>0.055</td>
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<td>11</td>
<td>Horizon Ag Products</td>
<td>Quantum H</td>
<td>7% Liquid</td>
<td>14.3</td>
<td>mL/L</td>
<td>0.210</td>
<td>0.047</td>
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<tr>
<td>12</td>
<td>UAS America</td>
<td>Super Hume</td>
<td>17% Liquid</td>
<td>5.9</td>
<td>mL/L</td>
<td>0.123</td>
<td>0.025</td>
<td>29% 36</td>
</tr>
<tr>
<td>13</td>
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<td>3% Liquid</td>
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<td>0.054</td>
<td>0.000</td>
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<td>Natural Resources Group</td>
<td>F Power 10%</td>
<td>10% Liquid</td>
<td>10.0</td>
<td>mL/L</td>
<td>0.041</td>
<td>0.037</td>
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<tr>
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<td>Aqua F</td>
<td>Liquid</td>
<td>33.3</td>
<td>mL/L</td>
<td>0.060</td>
<td>0.042</td>
<td>100% 17</td>
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<tr>
<td>16</td>
<td>Spectrum</td>
<td>Menadione sodium bisulfite</td>
<td>Solid</td>
<td>0.1</td>
<td>g/L</td>
<td>0.000</td>
<td>0.000</td>
<td>NA 0</td>
</tr>
<tr>
<td>17</td>
<td>Acros Fisher</td>
<td>Anthraquinone-2,6-disulfonic acid (AQDS)</td>
<td>Solid</td>
<td>0.1</td>
<td>g/L</td>
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<td>0.060</td>
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<td>Spectrum</td>
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<td>g/L</td>
<td>0.121</td>
<td>0.064</td>
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<tr>
<td>19</td>
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<td>Indigo disulfonate</td>
<td>Solid</td>
<td>0.1</td>
<td>g/L</td>
<td>0.243</td>
<td>0.015</td>
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<tr>
<td>20</td>
<td>Acros Fisher</td>
<td>Lawsson</td>
<td>Solid</td>
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<td>g/L</td>
<td>0.019</td>
<td>0.042</td>
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<td>Henna</td>
<td>Solid</td>
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<td>g/L</td>
<td>0.087</td>
<td>0.019</td>
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<td>IHSS</td>
<td>Soil Humic Acid Standard (control)</td>
<td>Solid</td>
<td>0.5</td>
<td>g/L</td>
<td>0.288</td>
<td>0.04</td>
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</tbody>
</table>

Shuttling Capacity of control 0.5 g/L IHSS Soil Humic Acid Standard 0.288 +/- 0.040 mM Fe reduced.
Appendix F
Isotherm Data
### #1 Teravita SP-85

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Initial Conc (mg/L)</th>
<th>Water (mL)</th>
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**24 hour sample**

**48 hour sample**

**Linear (24 hours)**

\[ y = 47.697x + 49.907 \]

\[ R^2 = 0.9986 \]
#2 - Luscar Dry Soluble 80

## 24 hour sample

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## 48 hour sample

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**Aqueous (mg/L)**

- **24 hours**
- **48 hours**
- **Linear (24 hours)**

**Graph:**

- **Soil (mg/kg) vs. Aqueous (mg/L)**
- **y = 74.744x + 68.399**
- **R² = 0.994**

- **Graph Notes:**
  - 24 hours
  - 48 hours
  - Linear (24 hours)
### #3 Luscar Liquid 12000

#### 24 hour sample

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#### 48 hour sample

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### Graph

**#3 Luscar Liquid 12000**

- **Equation**: \( y = 115.93x + 53.219 \)
- **R²**: 0.9903

**Graph**: 24 hours - 48 hours - Linear (24 hours)
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<th>1 g/L shuttle (mL)</th>
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<th>Abs (350 nm)</th>
<th>Aq Conc (mg/L)</th>
<th>Calc Soil Conc. (mg/kg)</th>
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**#4 Triad Huma K**

$$y = 61.084x - 18.126$$

$$R^2 = 0.9839$$

- **24 hours**
- **48 hours**
- Linear (24 hours)
### #5 Live Earth Products 12% Liquid Humic Acid

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<th>Dilution</th>
<th>Abs (350 nm) Aq Conc (mg/L)</th>
<th>Calc Soil Conc. (mg/kg)</th>
<th>Dilution</th>
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**Graph:**

- **Equation:** $y = 86.632x + 66.86$
- **$R^2$:** 0.9921

- **Legend:**
  - 24 hours
  - 48 hours
  - Linear (24 hours)
### #6 Monterey Ag Resources HA12

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Note: 6-300-1, 6-300-2 had 10 ml of GW added instead of 7ml

![Graph](attachment:image.png)

- **24 hours**
- **48 hours**

Linear (24 hours)
#8 - Humus Products of America 15% Concentrated Liquid Humus

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<th>Dilution</th>
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<th>Aq Conc (mg/L)</th>
<th>Calc Soil Conc. (mg/kg)</th>
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**Linear Regression (24 hours):**

- y = 65.521x - 11.745
- R² = 0.9968

**Graph:**

- 24 hours
- 48 hours
- Linear (24 hours)
# 9 - Borregro HA2

![Graph](image)

**Linear (24 hours)**

\[ y = 94.708x + 19.836 \]

\[ R^2 = 0.9999 \]

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Initial Conc (mg/L)</th>
<th>Water (mL)</th>
<th>1 g/L shuttle (mL)</th>
<th>Dilution</th>
<th>Diluted Sample</th>
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| 24 hour sample | | | 48 hour sample | | |
|----------------|-----------------|-----------------|-----------------|-----------------|
| Abs (350 nm)   | Calc Soil Conc. (mg/kg) | Aq Conc (mg/L) | Soil Conc. (mg/kg) |
| 0.041          | 0.000           | 4.41           | 0.000           |
| 0.037          | 0.000           | 3.98           | 0.000           |
| 0.048          | 0.046           | 4.95           | 0.7536606       | 92.463         |
| 0.052          | 0.052           | 5.60           | 1.3996555       | 86.003         |
| 0.058          | 0.058           | 6.24           | 2.0458503       | 279.543        |
| 0.067          | 0.067           | 7.21           | 3.0146425       | 269.854        |
| 0.118          | 0.118           | 12.70          | 8.5055986       | 914.944        |
| 0.128          | 0.128           | 13.78          | 9.5822567       | 904.177        |
| 0.32           | 0.32            | 34.45          | 30.254091       | 2697.459       |
| 0.286          | 0.286           | 30.79          | 26.93454        | 2734.065       |
### #10 Borregro HA1

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#### Linear Regression

- **y = 5.7948x + 105.97**
- \( R^2 = 0.9384 \)

![Graph of Soil vs Aqueous Concentration](image)
# 12 - UAS America SuperHume

![Graph showing linear relationship between Soil (mg/kg) and Aqueous (mg/L) with equation y = 75.6x + 118.3 and R² = 0.9862]

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Linear (24 hours)
### #19 Indigo Carmine

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### Aqueous - Linear (24 hours)

\[ y = 3.4995x + 26.703 \]

\[ R^2 = 0.9966 \]

### Soil (mg/kg) - Linear (24 hours)

\[ y = 0.0068x + 0.0058 \]

\[ R^2 = 0.9996 \]