

## **Appendix A**

### **Client Request Forms**

U. S. ENVIRONMENTAL PROTECTION AGENCY REGION 9  
Environmental Services Branch  
75 Hawthorne Street  
San Francisco, CA 94105  
Phone: 415/744-1498

Site Name: *Frontier Fertilizer*  
Case/RAP No.:

REGIONAL ANALYTICAL PROGRAM CLIENT REQUEST FORM

*(plus 1,2-dibromoethane and 1,2-dibromo-3-chloropropane)*

The analysis of low concentration water samples for the target compound list (TCL) volatile organic compounds (VOCs) following protocols outlined in the U.S. EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Organics Analysis (OLM03.1) and the Superfund Analytical Methods for Low Concentration Water for Organics Analysis (SAMLCO, 10/92 or more recent version). Where the procedures in this Client Request Form (CRF) differ from the above referenced methods, the procedures listed here are to take precedence.

A 25-mL purge volume is used for sample analysis to achieve low quantitation limits. The CRQLs listed in item 9, page 11 of this CRF must be met.

1. Definition and number of work units involved (specify whether whole samples or fraction; specify sample matrices; and specify concentration): *27 low level groundwater samples*

2. Estimated date(s) of collection (provide a sampling schedule):

*11/15/97 to 11/30/97*

3. Estimated date(s) and method of shipment:

Overnight courier - samples are to be shipped on the day of collection for next day delivery including Saturday deliveries. Laboratory must be capable of accepting Saturday deliveries.

4. Number of days analysis and data required after laboratory receipt of samples:

- a. The contract required analysis holding time is ten (10) days from the date of sample receipt by the laboratory.
- b. The technical analysis holding time is fourteen (14) days from the date of sample collection for preserved samples and seven (7) days from the date of sample collection for unpreserved samples.
- c. Data packages and all other deliverables are required within 35 days from receipt of last sample in each sample delivery group (SDG). A SDG is defined as the following, whichever is most frequent:
- Each case of field samples received; or
  - Each 20 field samples within a case; or
  - Each 14 calendar day period during which field samples in a case are received.

5. Analytical protocol required (attach copy if other than a protocol currently used in this program):
- Follow the procedures outlined in Exhibit D of the CLP SOW (OLM03.1) for the analysis of VOCs in water by gas chromatography/mass spectrometry (GC/MS) with sample introduction by purge-and-trap instrumentation.
  - A list of the VOC target compounds with corresponding contract required quantitation limits (CRQL) is provided in Section 9 of this Regional Analytical Program (RAP) Client Request Form (CRF).
  - Analyze a 25-mL sample aliquot for all samples which do not exceed the calibration range when run without dilution.
  - Determine the pH of all samples to verify preservation to pH <2 according to Exhibit D, Section 10.2 of OLM03.1.
6. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

a. Calibration Procedure and Criteria:

- Perform a GC/MS tuning according to the procedure outlined in Exhibit D, Section 9.2 of the CLP SOW (OLM03.1). A GC/MS instrument performance check with 4-bromofluorobenzene (BFB) is required at the beginning of each 12 hour period during which standards and samples are analyzed. Instrument performance checks must meet the ion abundance criteria specified in Table 1 of Exhibit D, Section 17.0, of the CLP SOW (OLM03.1). The MS tune criteria must be met before any instrument calibration standards, environmental samples, laboratory method blanks, or quality control (QC) samples are analyzed.
- Perform initial instrument calibration according to the procedure outlined in Exhibit D, Section III, Part C, Item 14 of the Superfund Analytical Methods for Low Concentration Water for Organics Analysis (SAMLCO, 10/92 or more recent version). Prepare initial calibration standards, containing all of the target analytes listed in Table 1 of this RAP CRF and system monitoring compounds, according to the requirements of Table D-1 of Exhibit D, Section II, Part B, Item 7.5 of the SAMLCO (10/92) to the following specifications:

*Include 1,2-dibromoethane and 1,2-dibromo-3-chloropropane in initial*

| Volume of Working Standard ( $\mu$ L added to 25 mL) | Final Concentration of Aqueous Standard for Non-Ketones ( $\mu$ g/L) | Final Concentration of Aqueous Standard for Ketones ( $\mu$ g/L) |
|--|--|--|
| 5  | 1  | 5  |
| 10   | 2  | 10   |
| 25   | 5  | 25   |
| 50   | 10   | 50   |
| 125  | 25   | 125  |

*and continuing calibration standard solution. See No. 8, page 11 for criteria*

The minimum relative response factors (RRFs) for all analytes in the initial calibration must meet the acceptance criteria specified in Table D-3 of Exhibit D, Section V and Section III, Item 14.5.4 of SAMLCO (10/92). The percent relative standard deviation (%RSD) of analyte RRFs across the initial calibration curve must not exceed 30% except as noted in Section III, Item 14.5.4.

3. Analyze a sixth initial calibration standard containing carbon tetrachloride, 1,2-dichloroethane, cis-1,3-dichloropropene, trans-1,3-dichloropropene, and vinyl chloride at a concentration of 0.5 µg/L to verify that the requested CRQLs can be achieved. The minimum RRF requirement for these analytes is 0.05. This low level standard need not be included with the other five initial calibration standards in calculating the %RSD for the standard curve.
4. Perform continuing instrument calibration according to the procedure outlined in Exhibit D, Section III, Part C, Item 15 of the SAMLCO (10/92) once per each 12 hour period of instrument operation. Prepare a continuing calibration standard, containing all of the target analytes listed in Section 9 of this RAP CRF, at concentrations equivalent to the midpoint of the initial calibration curve. The minimum RRFs for all analytes in the continuing calibration must meet the acceptance criteria specified in Table D-3 of Exhibit D, Section V and Section III, Item 15.5.4 of SAMLCO (10/92). The percent difference (%D) between the RRFs from the continuing calibration and average RRFs from the initial calibration must not exceed ±30% except as noted in Section III, Item 15.5.4.

b. Internal Quality Control Checks, Control Limits and Corrective Actions:

1. When calibration standard measurements exceed the QC requirements for the initial calibration or the continuing calibration, take corrective action as specified in Exhibit D, Section III, Part C, Items 14.6 (for initial calibration) and 15.6 (for continuing calibration) of SAMLCO (10/92).

The continuing calibration standard reflects the conditions under which the analysis of all associated samples was performed. Reanalyze all samples associated with an out-of-control continuing calibration standard.

2. Analyze a laboratory method blank in each 12-hour time period in which samples are analyzed according to the procedure described in Exhibit D, Section 12.1 of the CLP SOW (OLM03.1). The concentration of target compounds in the laboratory method blanks must meet the requirements specified in Exhibit D, Section 12.1.4.5 of OLM03.1.

If a method blank exceeds these acceptance criteria, the analytical system should be considered to be out-of-control. Follow the corrective action procedures outlined in Exhibit D, Section 12.1.5 of the CLP SOW (OLM03.1). Investigate the source of contamination and document appropriate corrective measures taken before proceeding with further sample analysis. Reanalyze all samples processed with a method

blank that is out-of-control. Reanalyses must be performed at no additional cost to the Region.

3. Analyze instrument blanks after the analysis of a sample containing target analytes at concentrations exceeding the initial calibration range and storage blanks per Exhibit D, Section V, Item 26 of SAMLCO (10/92). The concentration of target compounds in the instrument and storage blanks must meet the requirements specified in Exhibit D, Section V, Item 26.4 of SAMLCO (10/92).

If an instrument blank exceeds these acceptance criteria, the analytical system should be considered to be out-of-control. Follow the corrective action procedures outlined in Exhibit D, Section V, Item 26.5 of SAMLCO (10/95). Investigate the source of the contamination and document the appropriate corrective measures taken before proceeding with further sample analysis. Reanalyze all samples processed with an instrument blank that is out-of-control. Reanalyses must be performed at no additional cost to the Region. Document storage blank contamination in the SDG narrative and retain all storage blank data at the laboratory.

4. Spike all environmental samples, laboratory method blanks, and QC samples with the system monitoring compounds (SMCs), toluene- $d_6$ , 4-bromofluorobenzene (BFB), and 1,2-dichloroethane- $d_4$ , according to the procedure outlined in Exhibit D, Section 10.1.3.7 of the CLP SOW (OLM03.1). Prepare the system monitoring compound spiking solution according to Exhibit D, Section 7.2.4.1 of the CLP SOW (OLM03.1).

System monitoring compound recoveries within the limits for water specified in Table 7 of Exhibit D, Section 17 are required. If these control limits are exceeded, take appropriate action to identify the problem by reanalyzing the affected sample. If reanalysis solves the problem, then the problem was within the laboratory's control. In this case, submit only data from the analysis with system monitoring compound recoveries within QC limits. If reanalysis of the sample does not solve the problem, then submit data from both the initial analysis and the reanalysis.

5. Add the internal standard compounds bromochloromethane, chlorobenzene- $d_5$ , and 1,4-difluorobenzene to all calibration standards, method blanks, and QC samples prior to analysis according to the procedure outlined in Exhibit D, Sections 9.3.3.3 (for initial calibration standards), 9.4.3.2 (for continuing calibration standards), and 10.1.3.7 (for samples and blanks) of the CLP SOW (OLM03.1). Prepare the internal standard compound spiking solution according to Exhibit D, Section 7.2.4.3 of the CLP SOW (OLM03.1).

Internal standard area counts must not vary by more than a factor of 2 (-50% to +100%) from the area counts of the associated continuing calibration standard. Internal standard retention times (RT) must not vary by more than  $\pm 30$  seconds from the (RT) of the associated continuing calibration standard. Refer to Exhibit D, Sections 11.3.5 and 11.3.6 of the CLP SOW (OLM03.1) for internal standard acceptance criteria. If these control limits are exceeded,

reanalyze the affected sample. If reanalysis solves the problem, then the problem was within the laboratory's control. In this case, submit only data from the analysis with internal standard areas within QC limits. If reanalysis of the sample does not solve the problem, then submit data from both the initial analysis and the reanalysis.

6. Analyze matrix spike and matrix spike duplicate (MS/MSD) samples at the frequency of one per sample delivery group according to the procedure outlined in Exhibit D, Section 12.2 of the CLP SOW (OLM03.1). Prepare the matrix spiking solution according to Exhibit D, Section 7.2.4.2 of the CLP SOW (OLM03.1); prepare MS/MSD samples according to the procedure outlined in Exhibit D, Section 12.2.3 of the CLP SOW (OLM03.1).

MS/MSD recoveries, and relative percent differences (RPDs) between MS/MSD recoveries, within the limits specified for water in Table 8 of Exhibit D, Section 17.0 are required. Flag MS/MSD results that exceed these criteria and note noncompliant MS/MSD results in the SDG narrative. No corrective action measures are required due to noncompliant MS/MSD results.

7. Dilute and reanalyze samples which contain one or more target analytes at concentrations above the initial calibration range. If dilution is necessary, adjust the dilution so that the most highly concentrated analyte is determined at a concentration in the upper half of the calibration range. Report the results and submit documentation for both the diluted and undiluted analyses.
8. The QC requirements listed above are the minimum required. It is impossible to address all analytical situations that might be experienced by a laboratory during the analysis of environmental samples. The laboratory is expected to adhere to good laboratory practices when analyzing samples. If the laboratory has questions concerning the analyses of samples not addressed in this document, the Region should be notified IMMEDIATELY. The Laboratory Manager, or designee, must address any problems and its resolution in the SDG narrative.

7. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.

a. Data Calculations and Reporting Units:

1. Calculate the relative response factor (RRF) and the concentration of individual analytes according to the equations specified in Exhibit D, Sections 9.3.4.1 and 11.2.1 of the CLP SOW (OLM03.1).
2. Report sample results in concentration units of micrograms per liter ( $\mu\text{g/L}$ ). Report results less than 10  $\mu\text{g/L}$  to 1 significant figure and results greater than or equal to 10  $\mu\text{g/L}$  to 2 significant figures.

Include 1,2-dibromoethane  
and 1,2-dibromo-3-chloropropane  
in matrix spiking solutions  
75% to 125% recoveries  
are required for these  
compounds.

3. For rounding results, adhere to the following rules:
  - a) If the digit following those to be retained is less than 5, round down;
  - b) If the digit following those to be retained is greater than 5, round up; or
  - c) If the digit following the last digit to be retained is 5, round down if the digit is even, and round up if the digit is odd.
4. Ensure that all records of analysis, dilutions and calculations are legible and sufficient to recalculate all sample concentrations and QC results. Include an example of an actual sample calculation in the data package.

b. Documentation and Deliverables:

All documentation and deliverables as required in Exhibit B of the CLP SOW (OLM03.1) must be submitted. The required deliverables for each SDG must include the following:

1. All original shipping documents and sample tracking reports including signed RAP chain-of-custody forms, airbills, and traffic reports.
2. A completed and signed document inventory on a modified Organics Complete SDG File (CSF) Inventory Sheet (CLP Form DC-2).
3. All original sample receipt documents including sample log-in information on a modified CLP Form DC-1, an SDG cover sheet, and any other receipt forms such as copies of receipt logbooks.
4. A copy of the RAP CRF, as provided by the Region (so that any additions or revisions authorized by the Region will be known). Only the technical portion of the CRF is required.
5. Any telephone logs referring to the samples.
6. An SDG narrative signed by the laboratory manager, or designee, certifying the accuracy and validity of all data reported. The SDG narrative must contain: laboratory name; laboratory code; contract number; RAP number; SDG number; a list of correct EPA sample numbers and the corresponding laboratory sample identification (ID) numbers, differentiating between the initial analyses and any reanalyses; and the pH of all water samples. The SDG narrative must provide a description of all GC columns used for analysis, including brand name, the internal diameter in millimeters (mm), the length in meters, coating material, and film thickness. The SDG narrative must describe any administrative or technical problems encountered during the processing of the samples and a description of the resolution of these problems. The SDG narrative must include an explanation for any manual integrations or manual edits. The SDG narrative must provide a formula (including definitions) showing how the results were calculated, and an example of an actual calculation for a sample in the SDG.
7. The following header information is required for each data reporting form: laboratory name; contract number; laboratory code; RAP number; and SDG number.

8. Tabulated analysis results for all environmental and QC sample results on a modified CLP Form 1. Include the compound name and Chemical Abstract Service (CAS) registry number. Clearly specify concentration units and laboratory qualifiers. Include the following additional information in the header: laboratory sample ID, laboratory file ID, sample volume, purge volume, dilution factor, the validated time of sample receipt (VTSR), analysis date, and pH. Form 1s for any reanalyses or diluted analyses must be included.
9. Modified CLP Form 1E for all environmental samples which lists tentatively identified compounds. The modified CLP Form 1E must report the CAS number, compound name, RT, the estimated concentration, and the laboratory qualifier for each TIC reported. The header information should be the same as that provided in CLP Form 1, listed above.
10. Raw sample data must include the following items:
  - a) Reconstructed ion chromatograms (RICs) labeled with the following information: EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID. Each internal standard and surrogate compound must be labeled on the RIC by name.
  - b) Copies of raw mass spectra and background-subtracted mass spectra of detected target compounds labeled with the compound name, EPA sample number, laboratory file ID, analysis date and time, and instrument ID. For TICs, copies of mass spectra with associated 3 best-match spectra must be provided and labelled as above.
  - c) GC/MS quantitation report/data system printouts containing the following information: EPA sample number, date and time of analysis, RT or scan numbers of identified target compounds, ion used for quantitation, copy of area table from data system, GC/MS instrument ID, laboratory file ID, and analyst ID.
  - d) Extracted ion current profile (EICPs) displaying any manual integrations
11. Modified CLP Form 2A which lists the percent recovery (%R) values, rounded to the nearest whole number, and the QC limits for each system monitoring compound in all samples and laboratory method blanks. Flag all recoveries outside the QC limits with an asterisk.
12. Modified CLP Form 3A, a matrix spike/matrix spike duplicate (MS/MSD) results summary, which reports percent recoveries and RPD values to the nearest whole number for the spike compounds. List the QC limits for percent recovery and RPD for each spike compound and flag all values outside of the QC limits with an asterisk. Tabulate the concentration of analytes added to the sample, the sample concentration, and the MS and MSD concentrations for each analyte spiked. Include the EPA sample number in the header.
13. Modified CLP Form 4A which summarizes the samples that were analyzed with each laboratory method blank. The EPA sample

number, the laboratory sample ID, and analysis time must be reported for each sample associated with the referenced blank. Include the following additional information in the header: the laboratory method blank EPA sample number, laboratory file ID, laboratory sample ID, method blank analysis date and time, the GC column ID (i.e. liquid phase, length, diameter), and instrument ID.

14. CLP Form 5A which summarizes the data for BFB instrument performance checks. The ion, the abundance criteria, and the percent relative abundance for each ion must be reported. The modified CLP Form 5A must include the EPA sample number, laboratory sample ID, laboratory file ID, and date and time of analysis for each sample associated with the referenced BFB instrument performance check. Include the following additional information in the header: laboratory file ID, BFB injection date and time, instrument ID, and GC column ID.

15. Instrument calibration data which include the following items:

a) Initial Calibration

- 1) Modified CLP Form 6A which includes: RRFs for each analyte and surrogate for each concentration level, and average RRFs and %RSDs for each target and surrogate compound
- 2) Include the following additional information in the form header: date and time of calibration, instrument ID, and GC analytical column ID
- 3) Raw data which include: RICs and quantitation reports/data system printouts. RICs must contain the following information: EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID. Each internal standard and surrogate compound must be labeled on the RIC by name. Quantitation reports/data system printouts must contain the following information: EPA sample number, date and time of analysis, RT or scan numbers of identified target compounds, ion used for quantitation, copy of area table from data system, GC/MS instrument ID, laboratory file ID, and analyst ID.
- 4) Extracted ion current profile (EICPs) displaying any manual edits and integrations.

b) Continuing Calibration

- 1) Modified CLP Form 7A which includes: average RRFs (from the initial calibration), RRFs (for the continuing calibration standard), and %D values for each target and surrogate compound.
- 2) Include the following additional information in the header: laboratory file ID, date and time of the continuing calibration, instrument ID, GC

analytical column ID, and the date and time of the referenced initial calibration

- 3) Raw data which include: RICs must contain the following information: EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID. Each internal standard and surrogate compound must be labeled on the RIC by name. Quantitation reports/data system printouts must contain the following information: EPA sample number, date and time of analysis, RT or scan numbers of identified target compounds, ion used for quantitation, copy of area table from data system, GC/MS instrument ID, laboratory file ID, and analyst ID.
  - 4) Extracted ion current profile (EICPs) displaying any manual edits and integrations.
16. Modified CLP Form 8A which summarizes areas and RTs of the internal standards for all samples, blanks, QC samples associated with the referenced continuing calibration standard. Clearly specify the QC limits for the internal standard response and RT and flag all results outside the QC limits. Include the following information in the header: laboratory file ID for the standard, date and time of analysis, instrument ID, and GC column ID.
17. Raw QC data which include the following items:
- a) Blank data, in chronological order:
    - 1) Tabulated results on a modified CLP Form 1A and 1E
    - 2) RIC, mass spectra, and best-match spectra labeled as specified above
    - 3) GC/MS quantitation report/data system printout labeled as specified above
    - 4) EICPs displaying any manual integrations or edits
  - b) MS/MSD data, in chronological order:
    - 1) Tabulated results on a modified CLP Form 1A
    - 2) RIC labeled as specified above
    - 3) GC/MS quantitation report/data system printout labeled as specified above
    - 4) EICPs displaying any manual integrations or edits
  - c) BFB Instrument Performance Check data, in chronological order:
    - 1) Bar graph spectrum labelled with EPA sample number, date and time of analysis, GC/MS

instrument identifier, laboratory file ID, and analyst ID.

- 2) Mass listing labeled with EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID
- 3) RICs labeled with EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID.

18. Bench sheets for pH determination and dilutions
19. Bench sheets for sample analysis including instrument ID and analysis date and time
20. Bench sheets for sample preparation which include spike solution and SMC solution description (i.e. concentrations and volume added)
21. Standards preparation logs, for all standards used for either calibration or spiking, which include source, traceable lot number, and concentrations of all compounds
22. Any internal laboratory sample or sample extract transfer records and tracking sheets

8. Other (use additional sheets or attach supplementary information, as needed):

If a copy of the "U.S. EPA Region 9 Laboratory QC Summary Report" form is attached, complete the form by following the directions on the first page of the form.

Initial and continuing calibration criteria for 1,2-dibromoethane and 1,2-dibromo-3-chloropropane are as follows:

|                             | <u>min. RRF</u> | <u>max % RSD</u> | <u>max % D (continuing calibration)</u> |
|-----------------------------|-----------------|------------------|---|
| 1,2-dibromoethane           | 0.100           | 30.0             | ± 30.0                                  |
| 1,2-dibromo-3-chloropropane | 0.010           | 30.0             | ± 30.0                                  |

1,2-dibromoethane criteria are from SAMLCO. 1,2-dibromo-3-chloropropane criteria are specific for this project. Four point initial calibration of 1,2-dibromo-3-chloropropane is acceptable if five point calibration fails to meet the above criteria due to poor response at the 1 µg/l level.

9. Data Requirements:

The required target compounds with corresponding contract required quantitation limits (CROs) are provided below.

| <u>Analyte</u>             | <u>CAS Number</u> | <u>(CROL) µg/L</u> |
|----------------------------|-------------------|--------------------|
| Chloromethane              | 74-87-3           | 1                  |
| Bromomethane               | 74-83-9           | 1                  |
| Vinyl chloride             | 75-01-4           | 0.5                |
| Chloroethane               | 75-00-3           | 1                  |
| Methylene chloride         | 75-09-2           | 1                  |
| Acetone                    | 67-64-1           | 10                 |
| Carbon disulfide           | 75-15-0           | 1                  |
| 1,1-Dichloroethene         | 75-35-4           | 1                  |
| 1,1-Dichloroethane         | 75-34-3           | 1                  |
| 1,2-Dichloroethene (total) | 540-59-0          | 1                  |
| Chloroform                 | 67-66-3           | 1                  |
| 1,2-Dichloroethane         | 107-06-2          | 0.5                |
| 2-Butanone                 | 78-87-5           | 10                 |
| 1,1,1-Trichloroethane      | 71-55-6           | 1                  |
| Carbon tetrachloride       | 56-23-5           | 0.5                |
| Bromodichloromethane       | 75-27-4           | 1                  |
| 1,2-Dichloropropane        | 78-87-5           | 1                  |
| cis-1,3-Dichloropropene    | 10061-01-5        | 0.5                |
| Trichloroethene            | 79-01-6           | 1                  |
| Dibromochloromethane       | 124-48-1          | 1                  |
| 1,1,2-Trichloroethane      | 79-00-5           | 1                  |
| Benzene                    | 71-43-2           | 1                  |
| trans-1,3-Dichloropropene  | 10061-02-6        | 0.5                |
| Bromoform                  | 75-25-2           | 1                  |
| 4-Methyl-2-pentanone       | 108-10-1          | 10                 |
| 2-Hexanone                 | 591-78-6          | 10                 |
| Tetrachloroethene          | 127-18-4          | 1                  |
| 1,1,2,2-Tetrachloroethane  | 79-34-5           | 1                  |
| Toluene                    | 108-88-3          | 1                  |
| Chlorobenzene              | 108-90-7          | 1                  |
| Ethylbenzene               | 100-41-4          | 1                  |
| Styrene                    | 100-42-5          | 1                  |
| Xylenes (total)            | 1330-20-7         | 1                  |

U. S. ENVIRONMENTAL PROTECTION AGENCY REGION 9  
Environmental Services Branch  
75 Hawthorne Street  
San Francisco, CA 94105  
Phone: 415/744-1498

SITE NAME: *Frontier Facility*  
CASE/RAP NO.:

REGIONAL ANALYTICAL PROGRAM CLIENT REQUEST FORM

The analysis of water samples for 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) by EPA Method 504, Revision 2.0 (1989).

1. Definition and number of work units involved (specify whether whole samples or fraction; specify sample matrices; and specify concentration level): *27 low level groundwater samples*
  
2. Estimated date(s) of collection (provide a sampling schedule):  
*11/15/97 to 11/30/97*
  
3. Estimated date(s) and method of shipment:  
Overnight courier - samples are to be shipped on the day of collection for next day delivery including Saturday deliveries. Laboratory must be capable of accepting Saturday deliveries.
  
4. Number of days analysis and data required after laboratory receipt of samples:
  - a. The contract required holding time for extraction and analysis is twenty-six (26) days from the date of sample receipt by the laboratory.
  - b. The method holding time for extraction and analysis is 28 days from the date of sample collection.
  - c. Data packages and all other deliverables are required within 35 days from receipt of last sample in each sample delivery group (SDG). A SDG is defined as the following, whichever is most frequent:
    - Each case of field samples received; or
    - Each 20 field samples within a case; or
    - Each 14 calendar day period during which field samples in a case are received.
  
5. Analytical protocol required (attach copy if other than a protocol currently used in this program):
  - a. Requirements for the gas chromatograph (GC) system and GC columns are provided in Section 6.7 of Method 504. Columns other than those recommended in Sections 6.7.3 through 6.7.5 are permissible if the laboratory demonstrates that the analyses meet all the

performance and QA/QC criteria specified in Method 504 and in this contract. The use of a linearized electron capture detector (ECD) is required.

- b. A five point initial calibration is required. Aqueous calibration standards are prepared (see Sections 9.1.1 through 9.1.3) and extracted (see Sections 11.1.3 and 11.2.1 through 11.2.6) in a manner identical to sample extraction. These standards are used for instrument calibration and sample quantitation to account for potential analyte losses during extraction.
  - c. Follow EPA Method 504 (Section 11) for extraction and analysis of samples and QC samples. Section 8 presents the required target analytes and contract required quantitation limits (CROs).
  - d. Confirmation on a second column with a liquid phase different from that of the primary column is required for all positive results. Refer to Section 5.a (above) for acceptable confirmation columns. Confirmation analyses must meet all instrument calibration criteria and blank acceptance criteria specified in Sections 6.a and 6.b.2, respectively, of this Client Request Form (CRF).
6. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

a. Calibration Procedure and Criteria:

Calibrate according to Section 8.0 of Method 504, with the following specifications:

1. Perform a five-point initial calibration containing both target analytes and the surrogate compound with a low standard at the concentrations of the CRO or lower. Single point calibration (Method 504, Section 9.1.4) is not a permissible option.

- a) If the external standard method is used, either peak area or peak height may be used for (instrument) response in the calculations. Calculate the calibration factor (CF) for each analyte in each calibration standard following Section 9.1.3 of EPA Method 504.

$$CF = x \div y$$

where CF = calibration factor [in  $\mu\text{g/L}$ ]  
x = concentration of the standard [in  $\mu\text{g/L}$ ]  
y = response, which may be peak area or height [unitless]

Calculate the mean and standard deviation ( $\sigma_{n-1}$ ) of the CFs in the five calibration standards. Calculate the percent relative standard deviation (%RSD) by dividing the standard deviation by the mean. The %RSD for both analytes and the surrogate compound must not exceed 20.

- b) Alternatively, linear regression that does not force the resulting straight line to pass through the origin may be used for instrument calibration. For each analyte, perform a linear regression on the response versus the concentration of the standards. The linear

regression analysis will produce the slope and y-intercept values for a linear equation in the following form:

$$y = ax + b$$

where:

y = response, which may be peak area or height  
x = concentration of the standard [in  $\mu\text{g/L}$ ]  
a = slope of the straight line  
b = the y-intercept

The correlation coefficient (r) generated by the linear regression must be greater than or equal to 0.99 for each target and surrogate compound. If calibration curves are used, provide sufficient documentation so that all analytical results can be recalculated, as specified in Section 7.a.4 of this CRF. Provide the equation for the line generated by the data system.

- c) The low concentration standard must have a signal-to-noise ratio of 5:1 or greater for both target analytes. If this requirement cannot be met, the laboratory must submit a method detection limit (MDL) study as part of the data package, in order to validate its ability to achieve the CRQLs. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.
2. Determine a retention time (RT) window for each target and surrogate compound. Calculate the mean and standard deviation ( $\sigma_{n-1}$ ) of the 5 retention times for each compound over the 5 concentration levels in the initial calibration. Calculate the window as the mean RT  $\pm$  3 times the standard deviation.
3. Demonstrate that the individual analytes in the mid-point calibration standard are resolved by the chromatographic system. The analytes must be  $\geq 90\%$  resolved.
4. Extract and analyze a continuing calibration at the mid-point concentration for each analyte at the beginning of each day, after every 10 injections, and at the end of the analytical sequence. This standard is to be used to verify instrument performance. Percent differences between the nominal amount and calculated amount for each analyte in the continuing calibrations must be within  $\pm 15\%$  for both EDB and DECP.
5. Calculate the daily RT windows as the continuing calibration RT  $\pm$  3 times the standard deviation as determined in Section 6.a.2 of this CRF. Use these daily RT windows for target analyte identification.

b. Internal Quality Control Checks, Control Limits and Corrective Actions:

1. When calibration standard measurements exceed the quality control (QC) requirements for the initial calibration or the

continuing calibration, terminate analysis, correct the problem, and recalibrate the instrument.

The continuing calibration standard reflects the conditions under which the analysis of samples was performed. Associated samples are considered to be the samples analyzed following the continuing calibration up to the subsequent acceptable continuing calibration standard. Reanalyze all samples associated with an out-of-control continuing calibration standard.

2. Extract and analyze laboratory reagent blanks (LRBs) at a frequency of one per SDG or one for each extraction batch, whichever is greater. The method blanks must contain less than or equal to the CRQL of the target analytes listed in Section 8.

If a LRB exceeds these criteria, the laboratory must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples associated with a LRB that is out of control must be re-extracted and reanalyzed at no additional cost to the Region.

3. Spike all standards, samples, LRBs, and QC samples with an appropriate surrogate (i.e., 4-bromofluorobenzene). The amount of surrogate added must be at least 10 times the concentration of the lowest standard. Recoveries of 65-125% are required, unless documentation (such as control charts) is available to support a different range of recoveries. The laboratory must submit, as part of the data package, all supporting documentation for surrogate recoveries, and historical surrogate recovery data if necessary.

If surrogate control limits are exceeded, take appropriate actions to identify the problem by re-analyzing the affected samples. If reanalysis does not solve the problem, the affected sample must be re-extracted and re-analyzed. If re-extraction and re-analysis solves the problem, then the problem was within the laboratory's control. Therefore, submit only data from the analyses with surrogate recoveries within QC limits. If re-extraction and re-analysis of the sample does not solve the problem, then submit the surrogate recovery data and sample analysis data from the initial analysis of both sample extracts.

4. Dilute and reanalyze sample extracts containing one or more analytes at concentrations above the initial calibration range. If dilution is necessary, the dilution must be selected so that the highest concentration analyte is determined at a concentration in the upper half of the calibration range. Report the results and submit documentation for both the diluted and undiluted analyses.
5. Analyze laboratory fortified matrix and laboratory fortified matrix duplicate (LFM/LFMD) at the frequency of one per SDG. Both EDB and DBCP must be spiked into the LFM/LFMD analyses. The concentration of the matrix spike solution should be such that the final extracts contain each analyte at an amount at the mid-range of the calibration curve.

Recoveries of 75-115% and relative percent differences (RPDs) of ≤15% are required. Re-extraction and reanalysis are not required if the LFM/LFMD criteria are exceeded. However, document LFM/LFMD outliers in the SDG narrative.

6. Extract and analyze a laboratory fortified blank (LFB) for each SDG or each batch of samples prepared, whichever is more frequent. The final extracts should contain EDB and DECP at a concentration of 0.25 µg/L each. Percent recovery limits of 60-140% are required. If the QC limits are exceeded, re-extract and reanalyze the LCS and all samples associated with the noncompliant LCS.
  7. Perform weekly MDL checks according to Section 10.4 of Method 504. Recovery must be 60-140% for each analyte. Document outliers in the SDG narrative.
  8. Analyze a QC sample from an external source quarterly according to Section 10.5 of Method 504. Document outliers in the SDG narrative.
  9. The QC requirements listed above are the minimum required. It is impossible to address all analytical situations that might be experienced by a laboratory during the analysis of environmental samples. The laboratory is expected to adhere to good laboratory practices when analyzing samples. If the laboratory has questions concerning the analyses of samples not addressed in this document, the Region must be notified IMMEDIATELY. The Laboratory Manager, or designee, must address problems and their solutions in the SDG narrative.
7. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.

a. Data Calculations and Reporting Units:

1. Quantitation of target and surrogate compounds in field and QC samples are performed as follows.
  - a) To calculate the concentration of an analyte or surrogate using the external standard method, use the average CF for the compound from the initial calibration (see Section 6.a.1.a) in the equation:

$$\text{Concentration} = (y \times \text{average CF} \times \text{DF} \times 35 \text{ mL}) \div V_s$$

[µg/L]

where y = response, which may be peak area or height  
CF = calibration factor [µg/L]  
DF = dilution factor  
V<sub>s</sub> = volume of the sample in mL

- b) If using linear regression for data calculations, then use the following equation:

$$x = (y - b) \div a$$

x = concentration of the detected analyte [µg/L]  
y = response, which may be peak area or height  
b = the y-intercept

a = slope of the line

2. The sample results are to be reported in the concentration units of micrograms per liter ( $\mu\text{g/L}$ ) for water samples. Report all results to two significant figures.
3. For rounding results, adhere to the following rules:
  - a) If the digit following those to be retained is less than 5, round down;
  - b) If the digit following those to be retained is greater than 5, round up; or
  - c) If the digit following the last digit to be retained is equal to 5, round down if the digit is even, and round up if the digit is odd.
4. All records of analysis, dilutions and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example of the calculations in the data package.

b. Documentation and Deliverables:

Deliverables (in the form of a purge file, i.e., original documents) for each SDG shall include the following items:

1. All original shipping documents and sample tracking reports, including signed chain-of-custody forms, airbills, and traffic reports.
2. A completed and signed document inventory on a modified Organics Complete SDG File (CSF) Inventory Sheet (CLP Form DC-2).
3. All original sample receipt documents, including sample log-in information on a modified CLP Form DC-1, an SDG cover sheet, and any other sample receipt records.
4. A copy of the CRF, as provided by the Region (so that any additions or revisions authorized by the Region will be known). Only the technical portion of the CRF is required.
5. Any telephone logs referring to the samples.
6. An SDG Narrative, signed by the laboratory manager or designee, certifying the accuracy and validity of all data reported. The SDG Narrative must contain: laboratory name; case/RAP number; SDG number; EPA sample numbers in the SDG, differentiating between initial analyses and reanalyses; the corresponding laboratory sample identification (ID) numbers; and contract number. The SDG narrative must provide a description of all GC columns used for analysis, including brand name, the internal diameter in millimeters (mm), the length in meters, coating material, and film thickness. The SDG narrative must describe any administrative or technical problems encountered such as QC, sample shipment, or analytical problems and the resolution of these problems. The SDG narrative must include an explanation for any manual integrations or manual edits. The SDG narrative must include a formula (including definitions) showing how the results were calculated, and an example of an actual calculation for a sample in the SDG.

7. Include the following information in the header for each data reporting form: laboratory name, contract number, laboratory code, case/RAP number, and SDG number.
8. Tabulated analysis results for all field and QC samples on a modified CLP Form 1. Include both compound name and Chemical Abstracts Service (CAS) registry number for both analytes. Clearly specify concentration units and laboratory data qualifiers. Include the following additional information in the header: EPA sample number, laboratory sample ID, matrix, laboratory file ID, instrument ID, validated time of sample receipt (VTSR), extraction method, sample volume in milliliters (mLs), concentrated extract volume and injection volume in microliters ( $\mu$ Ls), and dilution factors.
9. Raw sample data, including:
  - a) All data system printouts, including: RT and corresponding peak area or height for each peak detected. The printouts must be labeled with the EPA sample number. When manual integrations or edits are made, analyst must sign and date each edit and include the integration time range.
  - b) All chromatograms, including: EPA sample number; volume injected ( $\mu$ L); date and time of injection; GC instrument ID; GC column identifier including stationary phase and internal diameter; and scaling factor.
  - c) Any manual worksheets
10. Surrogate result summaries on a modified CLP Form 2 with QC acceptance criteria and calculated percent recovery (%R) values rounded to the nearest whole number. If dual columns are used, surrogate recoveries should be reported from each column. Identify on the form header the GC column ID. Flag all recoveries outside the QC limits with an asterisk.
11. LFM/LFMD and LFB result summaries on a modified CLP Form 3 with calculated percent recovery (%R) and RPD values. Include in the form the recovery and RPD QC limits for each spike compound. Tabulate also the concentration of the spike in the sample, the sample concentration, and the LFM and LFMD or the LFB concentrations on the form. If dual columns are used, report the lower result from the two columns for each target compound. Flag all percent recoveries and RPDs exceeding the QC limits. Include the EPA sample number in the header. Also provide on a modified Form 3, the results from the weekly MDL analyses and the quarterly QC sample. It is not necessary to provide the raw data for analyses of MDLs and the quarterly QC sample.
12. Method blank summary on a modified CLP Form 4 specifying which samples are associated with each blank. Include on the form header the blank EPA sample number, the laboratory sample ID, the laboratory file ID, matrix, method blank extraction date, method blank analysis date and time, the instrument ID, and the GC column ID. The EPA sample number, the laboratory sample ID, and date analyzed must be included for each sample associated with the referenced blank. If

dual columns are used, report the items listed above for each column.

13. Instrument calibration data for each GC instrument and GC column used for analysis.

a) Initial Calibration

- 1) Standards summaries for initial calibration on modified CLP Form 6 tabulating: CFs, mean CFs, %RSD values, RTs, mean RTs, and RT windows for each compound. Include the following additional information in the header: GC instrument ID, column ID, concentration levels for each standard, and analysis date.
- 2) Resolution of the mid-point standards for each set analyzed in the initial calibration on a modified Form 6I with analyte names, retention time, and percent resolution. Include the following additional information in the header: GC instrument ID, column ID, standard EPA sample number, laboratory sample ID, and date and time analyzed.
- 3) All data system printouts, including: RT and corresponding peak area or height for each peak detected. Label printouts with standard EPA sample number and the total nanograms injected for each analyte. When manual integrations or edits are made, analyst must initial and date each edit and include the integration time range. Chromatograms must be labeled with the following: standard EPA sample number, volume injected ( $\mu\text{L}$ ), date and time of injection, GC instrument ID, GC column ID, and scaling factor.
- 4) If linear regression is used for quantitation, include for each analyte the equation of the straight line, correlation coefficient, and graph of the straight line on axes labeled to identify what is plotted.

b) Continuing Calibration

- 1) Standard summaries for continuing calibration on modified CLP Form 7 tabulating: the nominal amount, calculated amount, the %D values, RTs, and daily RT windows for each compound. Include the following additional information in the header: dates of associated initial calibration, GC column ID, EPA sample number and laboratory sample ID for the continuing calibration standard(s), and date and time of continuing calibration standard(s).
- 2) All data system printouts, including: RT and corresponding peak area or height for each peak detected. Label printouts with the standard EPA sample number and the total nanograms injected for each analyte. When manual integrations or edits are made, analyst must initial and date each edit and include the integration time range. Chromatograms must be labeled with the

following: standard EPA sample number, volume injected ( $\mu\text{L}$ ), date and time of injection, GC instrument ID, GC column ID, and scaling factor.

14. Analytical sequence information on a modified CLP Form 8 for each GC instrument and GC column used for analysis. Tabulate the EPA sample number, the laboratory sample ID, the date and time of analysis, and surrogate RTs for all standards and field and QC samples associated with the initial calibration reported in the header. Include the following additional information in the header: instrument ID, GC column ID with internal diameter, and date of initial calibration.
15. Target analyte results on a modified CLP Form 10 for each field and QC sample that has one or more detected result(s). Include the names of the detected analytes, the RTs, the RT windows, the concentrations, and %D between the concentrations on the two columns. Include the following additional information in the header: laboratory sample ID, analysis date, instrument ID, and GC column ID.
16. Raw QC data must include the following items for both primary and confirmation analyses:
  - a) Blank data, in chronological order:
    - 1) Tabulated results on a modified CLP Form 1
    - 2) All data system printouts, including: RT and corresponding peak area or height for each peak detected. The printout must be labeled with the EPA sample number. When manual integrations or edits are made, analyst must initial and date each edit and include the integration time range. Chromatograms must be labeled with the following: EPA sample number, volume injected ( $\mu\text{L}$ ), date and time of injection, GC instrument ID, GC column ID, and scaling factor.
    - 3) Any manual worksheets
  - b) MS/MSD and LCS data, in chronological order:
    - 1) Tabulated results on a modified CLP Form 1
    - 2) All data system printouts, including: RT and corresponding peak area or height for each peak detected. The printout must be labeled with the EPA sample number. When manual integrations or edits are made, analyst must initial and date each edit and include the integration time range. Chromatograms must be labeled with the following: EPA sample number, volume injected ( $\mu\text{L}$ ), date and time of injection, GC instrument ID, GC column ID, and scaling factor.
    - 3) Any manual worksheets
17. All computer printouts resulting from sample screening, with integrated areas, peak heights, and calibration factors.

18. Bench sheets for sample preparation and analysis, including the following:
  - a) Extraction dates and times
  - b) Sample extraction or preparation method
  - c) Spiking solution identification with volumes and concentrations added
  - d) Instrument run logs listing instrument ID and time and date of analyses
19. Standards preparation logs, for all standard solutions used for either calibration or spiking, which include source, traceable lot number, and concentrations of all compounds.
20. Any internal laboratory sample or sample extract transfer records and tracking sheets.

8. **Data Requirements**

The required target compounds with corresponding contract required quantitation limits (CROs) are provided below.

| <u>Compound</u>             | <u>CAS Number</u> | <u>Water (<math>\mu\text{g/L}</math>)</u> |
|-----------------------------|-------------------|---|
| 1,2-Dibromoethane           | 106-93-4          | 0.05                                      |
| 1,2-Dibromo-3-chloropropane | 96-12-8           | 0.05                                      |

9. **Other (use additional sheets or attach supplementary information, as needed):**

If a copy of the "U.S. EPA Region 9 Laboratory QC Summary Report" form is attached, complete the form by following the directions on the first page of the form.

**Appendix B**  
**DMLS Sampling Depths**

ORIGINAL  
with DWR

STATE OF CALIFORNIA  
THE RESOURCES AGENCY  
DEPARTMENT OF WATER RESOURCES  
WATER WELL DRILLERS REPORT

(MW7)  
Do not fill in

No. 18136T

of Interest No. \_\_\_\_\_  
Permit No. or 186-001

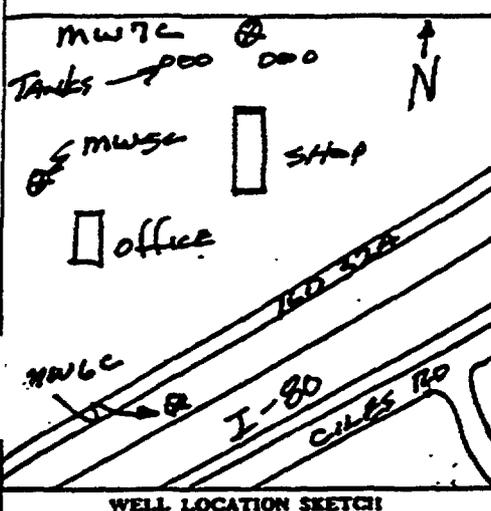
State Well No. \_\_\_\_\_  
Other Well No. \_\_\_\_\_

1) OWNER: Name FRONTIER FERTILIZER  
Address P.O. BOX 1048  
City DAVIS Zip 95617

2) LOCATION OF WELL (See instructions):  
County YOLO (Owner's Well Number MW7C)  
Well address if different from above \_\_\_\_\_  
Township 8N Range 2E Section 12  
Distance from cities, roads, railroads, fences, etc. \_\_\_\_\_

(12) WELL LOG: Total depth 98 ft. Depth of completed well 96 ft.

| from ft. | to ft. | Formation (Describe by color, character, size or material) |
|----------|--------|--|
| 0        | 24.5   | silty/clay   |
| 24.5     | 28.8   | silty/sand   |
| 28.8     | 39.7   | silty clay   |
| 39.7     | 43.8   | silty clay   |
| 43.8     | 46.5   | silty clay   |
| 46.5     | 48     | silty sand   |
| 48       | 64.5   | silty clay   |
| 64.5     | 65.8   | sand & clay  |
| 65.8     | 69.5   | silty sand   |
| 69.5     | 71.6   | sand & clay  |
| 71.6     | 87.5   | silty sand   |
| 87.5     | 95.5   | silty clay   |



(3) TYPE OF WORK:  
New Well  Deepening   
Reconstruction   
Reconditioning   
Horizontal Well   
Destruction  (Describe destruction materials and procedures in Item 12)

(4) PROPOSED USE:  
Domestic   
Irrigation   
Industrial   
Test Well   
Stock   
Municipal   
WATER MONITOR

(5) EQUIPMENT:  
Battery  Reverse   
Cable  Air   
Other  Auger Packet

(6) GRAVEL PACK:  
Yes  No  Size 12 x 20  
Diameter of base 10  
Packed from 70 to 90 ft.

(7) CASING INSTALLED:  
Steel  Plastic  Concrete

(8) PERFORATIONS:  
Type of perforation or size of screen \_\_\_\_\_

| From ft. | To ft. | Dia. in. | Gauge or Wall | From ft. | To ft. | Slot size |
|----------|--------|----------|---------------|----------|--------|-----------|
| 0        | 76     | 4        | sch40         | 76       | 86     | .040      |
| 86       | 96     | 4        | sch40         |          |        |           |

(9) WELL SEAL:  
Was surface sanitary seal provided? Yes  No  If yes, to depth 65 ft.  
Were struts sealed against pollution? Yes  No  Interval \_\_\_\_\_ ft.  
Method of sealing GROUT

(10) WATER LEVELS:  
Depth of first water, if known \_\_\_\_\_ ft.  
Standing level after well completion 34.9 ft.

(11) WELL TESTS:  
Was well test made? Yes  No  If yes, by whom? \_\_\_\_\_  
Type of test Pump  Boiler  Air lift   
Depth to water at start of test \_\_\_\_\_ ft. At end of test \_\_\_\_\_ ft.  
Recharge \_\_\_\_\_ gal/min after \_\_\_\_\_ hours Water temperature \_\_\_\_\_  
Chemical analysis made? Yes  No  If yes, by whom? \_\_\_\_\_  
Was electric log made? Yes  No  If yes, attach copy to this report

TEST HOLE 7-617-87  
Work started 7-27 19 87 Completed 8-3 19 87

WELL DRILLER'S STATEMENT:  
This well was drilled under my jurisdiction and this report is true to the best of my knowledge and belief.  
SIGNED Layne Western (Well Driller)  
NAME LAYNE WESTERN COMPANY, INC.  
(Person, firm, or corporation) (Typed or printed)  
Address P.O. BOX 1326  
City WOODLAND Zip 95695  
License No. 510011 Date of this report 11-24-87

100-2-7-87

# Lithologic Log: Frontier Fertilizer

elevation: (ground level \_\_\_\_\_ ft.)

drilling contractor, method: WDC, auger

completion date: 4/22/95

logger: Tim Colen

location: northing \_\_\_\_\_, easting \_\_\_\_\_

diameter: (borehole 12" (8" pilot))

| GRAPHIC LOG | DEPTH BELOW SURFACE (feet) | SAMPLE INTERVAL      | BLOW COUNT | SOIL DESCRIPTION<br>(name, color, particle size distribution, consistency (soft, hard, etc.), moisture content, structure...)                         | USCS | Pilot Boring Time | SAMPLE NUMBERS and COMMENTS  |
|-------------|----------------------------|----------------------|------------|---|------|-------------------|--|
|             | 0                          |                      |            | Dark brown (5YR 3/4). loamy silt. root hairs. damp.   | OL   | 1245              | spud on 4/21/95  |
|             | 5                          |                      |            | Dark red brown to medium brown (5YR 4/4). clayey silt. very moist. soft. plastic.   | ML   | 1246              |  |
|             | 10                         |                      |            | Same as above. but also dusky brown (10YR 2/2).   | ML   | 1247              | Grout: 9 bags of Portland (at 0930 on 4/22/95)                                 |
|             | 15                         |                      |            | Same as above.  | ML   | 1249              |  |
|             | 20                         |                      |            |   | ML   | 1252              |  |
|             | 25                         | 6/13/19<br>23.5'-25' |            | Mottled dark yellow orange to dark yellow brown (10YR 6/6 to 4/2). clayey sandy silt. crumbly to silty. very fine-grained sand. damp. stiff. plastic. | ML   | 1254              | Bentonite seal: one 5-gallon pail of 1/4-inch bentonite pellets                |
|             | 30                         | 6/10/13<br>28.5'-30' |            | Mottled yellow brown to medium brown (10YR 5/4 to 5YR 4/4). very fine-grained. silty. sand. local dark brown splotches. moist. slightly plastic.      | ML   | 1306              | Filter pack is #2/12 sand. 13 to 17 bags from 28.9' BGS to bottom of hole (TD) |
|             | 35                         | 7/12/13<br>33.5'-35' |            | Same as above in color. grades finer in grain size to clayey silt. stiff. slightly plastic. damp. trace of very fine-grained sand.                    | ML   | 1312              |  |
|             | 40                         | 4/5/7<br>38.5'-40'   |            | Light to medium brown (5YR 5/6 - 4/4 - 3/4). clayey silt to silty clay. soft. very plastic. moist. trace of very fine-grained sand.                   | ML   | 1325              |  |

# Lithologic Log: Frontier Fertilizer

elevation: (ground level \_\_\_\_\_ ft.)

drilling contractor, method: WDC/HCA, auger

completion date: 4/22/95

logger: Tim Colen

location: northing \_\_\_\_\_, easting \_\_\_\_\_

diameter: (borehole 12" (8" pilot))

| GRAPHIC LOG | DEPTH BELOW SURFACE (feet) | SAMPLE INTERVAL | BLOW COUNT            | SOIL DESCRIPTION<br>(name, color, particle size distribution, consistency (soft, hard, etc.), moisture content, structure ...)                           | USCS | Pilot Boring Time | SAMPLE NUMBERS and COMMENTS |
|-------------|----------------------------|-----------------|-----------------------|--|------|-------------------|-----------------------------|
|             | 40                         |                 |                       |  |      |                   |                             |
|             | 45                         |                 | 7/10/10<br>43.5'-45'  | Silty sand to sand, very fine- to medium-grained, medium brown (5YR 3/4), moist to wet, very slightly plastic, trace of mica flakes (very fine-grained). | SM   |                   | 1336                        |
|             | 50                         |                 | 18/17/22<br>48.5'-50' | Sandy, clayey silt, medium yellow brown to medium brown (10YR 5/4 to 5YR 4/4), wet, soft, plastic.   | ML   |                   | 1350                        |
|             | 55                         |                 |                       |  |      |                   | 1407                        |
|             | 60                         |                 |                       |  |      |                   |                             |
|             | 65                         |                 |                       |  |      |                   |                             |
|             | 70                         |                 |                       |  |      |                   |                             |
|             | 75                         |                 |                       |  |      |                   |                             |
|             | 80                         |                 |                       |  |      |                   |                             |

TD = 53.5'

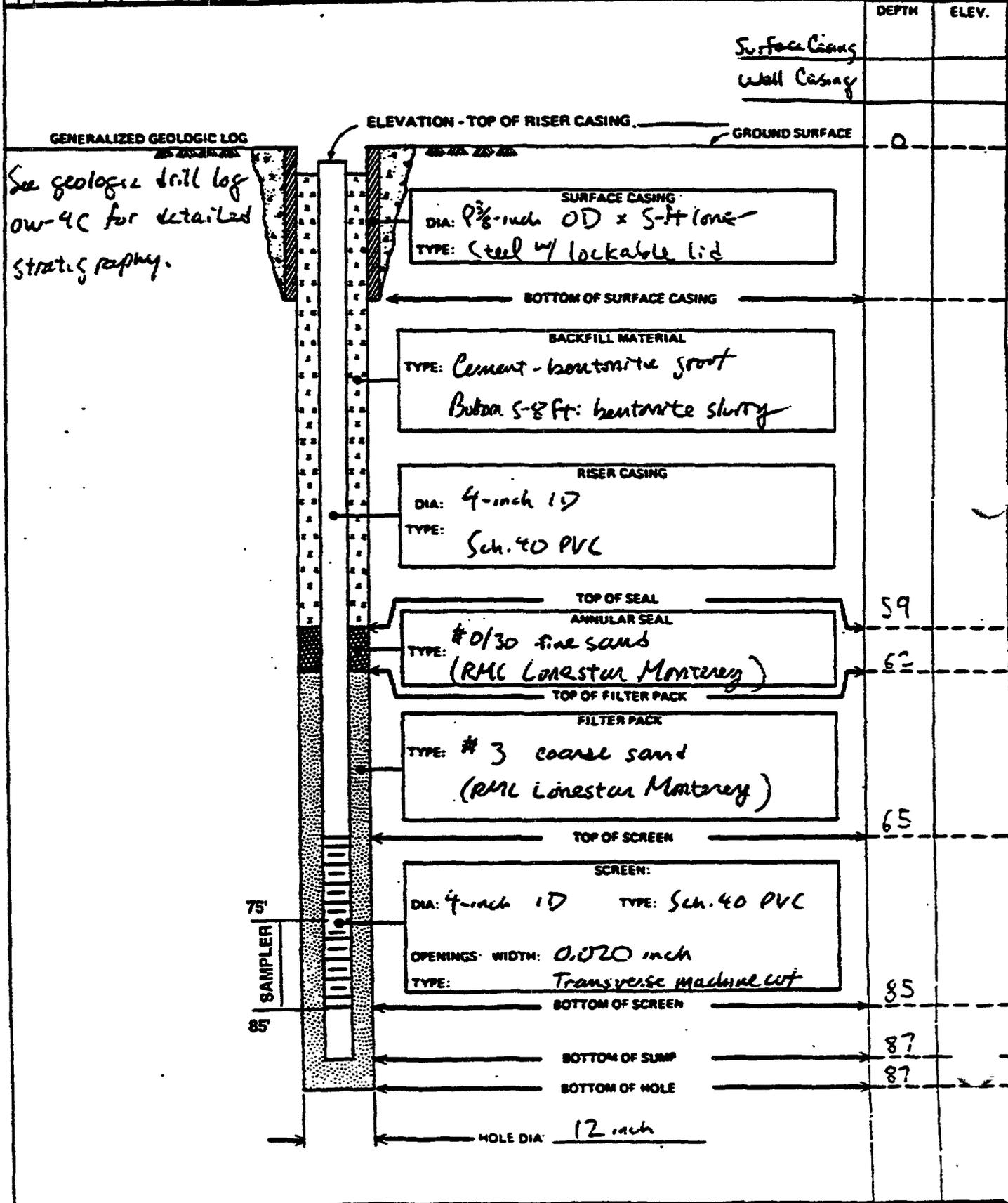
SAMPLER



|                        |                                |                   |
|------------------------|--------------------------------|-------------------|
| <b>MONITORING WELL</b> | PROJECT<br>Frontier Fertilizer | WELL NO.<br>OW-4B |
|------------------------|--------------------------------|-------------------|

|                          |                             |             |
|--------------------------|-----------------------------|-------------|
| JOB NO.<br>20376<br>-028 | SITE<br>Frontier Fertilizer | COORDINATES |
|--------------------------|-----------------------------|-------------|

|                 |                     |                         |                                  |
|-----------------|---------------------|-------------------------|----------------------------------|
| BEGUN<br>8/3/95 | COMPLETED<br>8/3/95 | PREPARED BY<br>C.M. Ovi | REFERENCE POINT FOR MEASUREMENTS |
|-----------------|---------------------|-------------------------|----------------------------------|



028 001521

U.S. Environmental Protection Agency Region IX  
Toxic Waste Management Division  
Field Operations Branch

Amendment 1  
Field Sampling Plan  
Groundwater Investigation

Frontier Fertilizer  
4309 Second Street  
Davis, California 95617

Site EPA ID Number CAD 071530380

Anticipated Sampling Dates: June 17 to June 30, 1998

Prepared by:  
Bechtel Environmental, Inc.  
P.O. Box 193965  
50 Beale Street  
San Francisco, CA 94119

Revision 1  
June 11, 1998

EPA Remedial Project Manager: Janet Rosati

Phone: (415) 744-2403

EPA Contract Number : 68-W9-0060

EPA Work Assignment Number: 60-28-9L4R

For EPA use:

Received by Superfund Project Manager:  
Reviewed by: Janet Rosati  
Status:  Approved

Date: \_\_\_\_\_  
Date: 6/14/98  
 Not Approved

Expedited Review?  Yes  
Received by QA Management Section:  
Reviewed by: Quetta C. Clark  
Status:  Approved  
Concurrence: Vanessa King

No  
Date: \_\_\_\_\_  
Date: 6/15/98  
 Not Approved  
Date: 6/15/98

Chief, Quality Assurance Office  
Management Section  
Environmental Services Branch, OPM D

FILE COPY

U.S. Environmental Protection Agency Region IX  
Toxic Waste Management Division  
Field Operations Branch

**Amendment 1**  
**Field Sampling Plan**  
Groundwater Investigation

Frontier Fertilizer  
4309 Second Street  
Davis, California 95617

Site EPA ID Number CAD 071530380

Anticipated Sampling Dates: June 10 to June 26, 1998

Prepared by:  
**Bechtel Environmental, Inc.**  
P.O. Box 193965  
50 Beale Street  
San Francisco, CA 94119

Revision 1  
June 4, 1998

**EPA Remedial Project Manager: Janet Rosati**

Phone: (415) 744-2403

EPA Contract Number : 68-W9-0060

EPA Work Assignment Number: 60-28-9L4R

---

**For EPA use:**

---

|   |                                       |
|---|---------------------------------------|
| Received by Superfund Project Manager:    | Date: _____                           |
| Reviewed by: _____                        | Date: _____                           |
| Status: <input type="checkbox"/> Approved | <input type="checkbox"/> Not Approved |

---

|  |                                       |
|--|---------------------------------------|
| Expedited Review? <input type="checkbox"/> Yes | <input type="checkbox"/> No           |
| Received by QA Management Section:             | Date: _____                           |
| Reviewed by: _____                             | Date: _____                           |
| Status: <input type="checkbox"/> Approved      | <input type="checkbox"/> Not Approved |
| Concurrence: _____                             | Date: _____                           |

Chief, Quality Assurance  
Management Section  
Environmental Services Branch, OPM

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## 1.0 Objective of Sampling Effort

Bechtel Environmental, Inc. (Bechtel) will conduct this groundwater field sampling effort to gather data as part of a Remedial Investigation under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) at the Frontier Fertilizer site. This field sampling plan amendment has been prepared under contract with the Environmental Protection Agency (EPA), Contract Number 68-W9-0060 and specific authorization of EPA Region IX, Work Assignment Number 60-28-9L4R.

Field sampling will be conducted under a protocol accepted by the EPA as specified in the *Preparation of a U.S. EPA Region IX Sample Plan for EPA-Lead Superfund Projects* guidance document (Quality Assurance Management Section, U.S. EPA, Region IX, August, 1993), the Field Sampling Plan (FSP) Groundwater Investigation, Rev. 0, approved by EPA on May 5, 1995, and the Quality Assurance Project Plan, Rev. 1, approved by EPA on October 2, 1997. Laboratory services will be obtained and coordinated through the EPA Quality Assurance Program.

It is the Frontier Fertilizer site (CAD 071530380) in Davis, Yolo County, California that is being investigated. From 1972 to 1983, the site was used as a fertilizer and pesticide distribution facility. Pesticides and fertilizers were stored and mixed on site and sold to farmers in 500 to 1,000-gallon tank trailers. When the trailers or other containers were returned, residual material was rinsed out and deposited into an unlined basin near the northwest corner of the site. Analytical results of soil and groundwater samples collected on or adjacent to the site indicated the presence of several pesticides compounds in the shallow groundwater beneath the downgradient of the site and in onsite soils.

The objectives of this third phase of groundwater investigation are to determine the extent of site-related contaminant migration in groundwater, install observation wells beyond the contaminant groundwater plumes, and characterize the site's geology and hydrogeology. This third phase of study will be restricted to characterizing the extent of groundwater contamination north of observation well cluster OW-6 and northeast of observation well cluster OW-7. These areas are the only remaining gaps data on the extent of groundwater contamination.

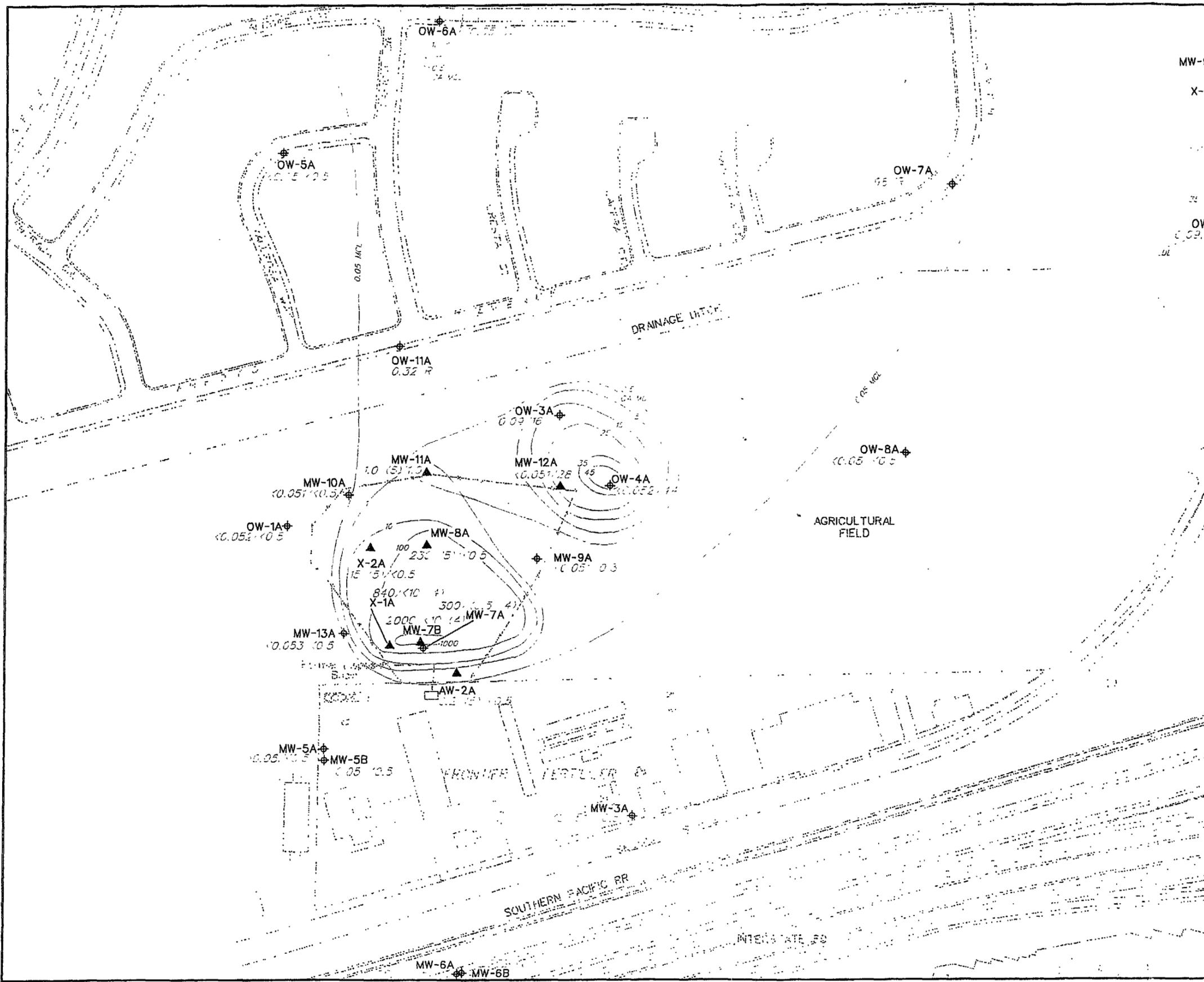
## 2.0 Background for Identification of Data Gap

Figures 1, 2, and 3 present the extent of 1,2-dibromoethane (EDB) and carbon tetrachloride ( $\text{CCl}_4$ ) in the three water bearing zones underlying Frontier Fertilizer and its environs. These zones are designated S-1, encountered at depths ranging from 35 to 40 feet below ground surface (bgs); S-2, encountered at depths ranging from 60 to 70 feet bgs; and A-1, found 105 to 130 feet bgs. As apparent from these figures, the S-1 and S-2 zones are most highly contaminated with contaminant concentrations decreasing rapidly with distance north of the former disposal basin. The extent of groundwater contaminated with other chemicals of concern is similar to the extent of EDB contamination.

While contaminant concentrations decrease rapidly with distance, the concentration of EDB is above its extremely low MCL, 0.05 parts per billion (ppb), in the S-1 and S-2 wells associated with observation well clusters OW-6 and OW-7. To define the extent of groundwater contamination it is necessary to locate groundwater north of OW-6 and northeast of OW-7 that is not contaminated with EDB and  $\text{CCl}_4$  at concentrations above their respective MCLs.

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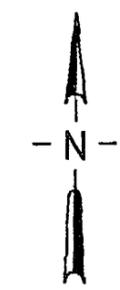


**EXPLANATION**

- MW-9A  $\oplus$  Monitoring Well
- X-2A  $\blacktriangle$  Active Extraction Well
- Interpreted 1,2 Dibromethane (EDB) Concentration Contour, October 1997
- Interpreted Carbon Tetrachloride (CCl<sub>4</sub>) Concentration Contour, October 1997
- OW-3A  $\oplus$  Concentrations at each location are represented for EDB/CCl<sub>4</sub>
- CCl<sub>4</sub> Data Rejected

**NOTES:**

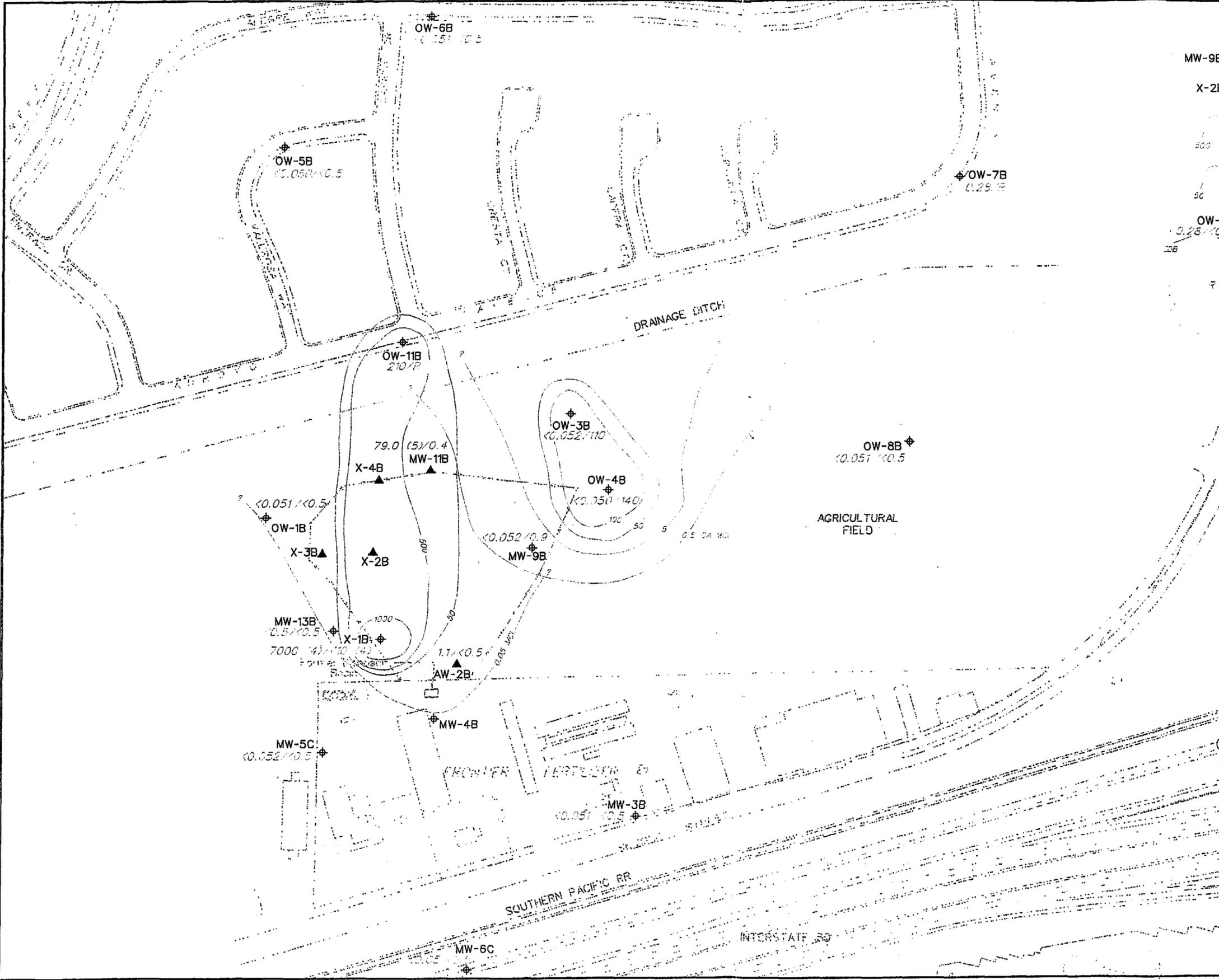
- 1) EDB concentrations in ug/L determined using EPA Method 504 unless otherwise noted.
- 2) CCl<sub>4</sub> by 25 ml purge unless otherwise noted.
- 3) Units: ug/L
- 4) Concentration determined by 5 ml purge method.
- 5) Concentration determined by 25 ml purge method.



Approximate Scale in Feet

|  |            |             |     |
|--|------------|-------------|-----|
| <b>Bechtel</b>   |            |             |     |
| SAN FRANCISCO  |            |             |     |
| FRONTIER FERTILIZER<br>DAVIS, CALIFORNIA   |            |             |     |
| <b>1, 2-DIBROMOETHANE (EDB) AND CCl<sub>4</sub><br/>IN THE S1 ZONE, OCTOBER 1997</b> |            |             |     |
|  | Job Number | Drawing No. | Rev |
|  | 20376      | FIGURE 1    | 0   |

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 5/27/98dg  
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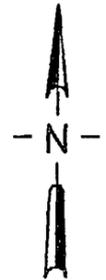


**EXPLANATION**

- MW-9B ◊ Monitoring Well
- X-2B ▲ Active Extraction Well
- Interpreted 1,2 Dibromomethane (EDB) Concentration Contour, October 1997
- Interpreted Carbon Tetrachloride (CCl<sub>4</sub>) Concentration Contour, October 1997
- Concentrations at each location are represented for EDB/CCl<sub>4</sub>
- CCl<sub>4</sub> Data Rejected

**NOTES:**

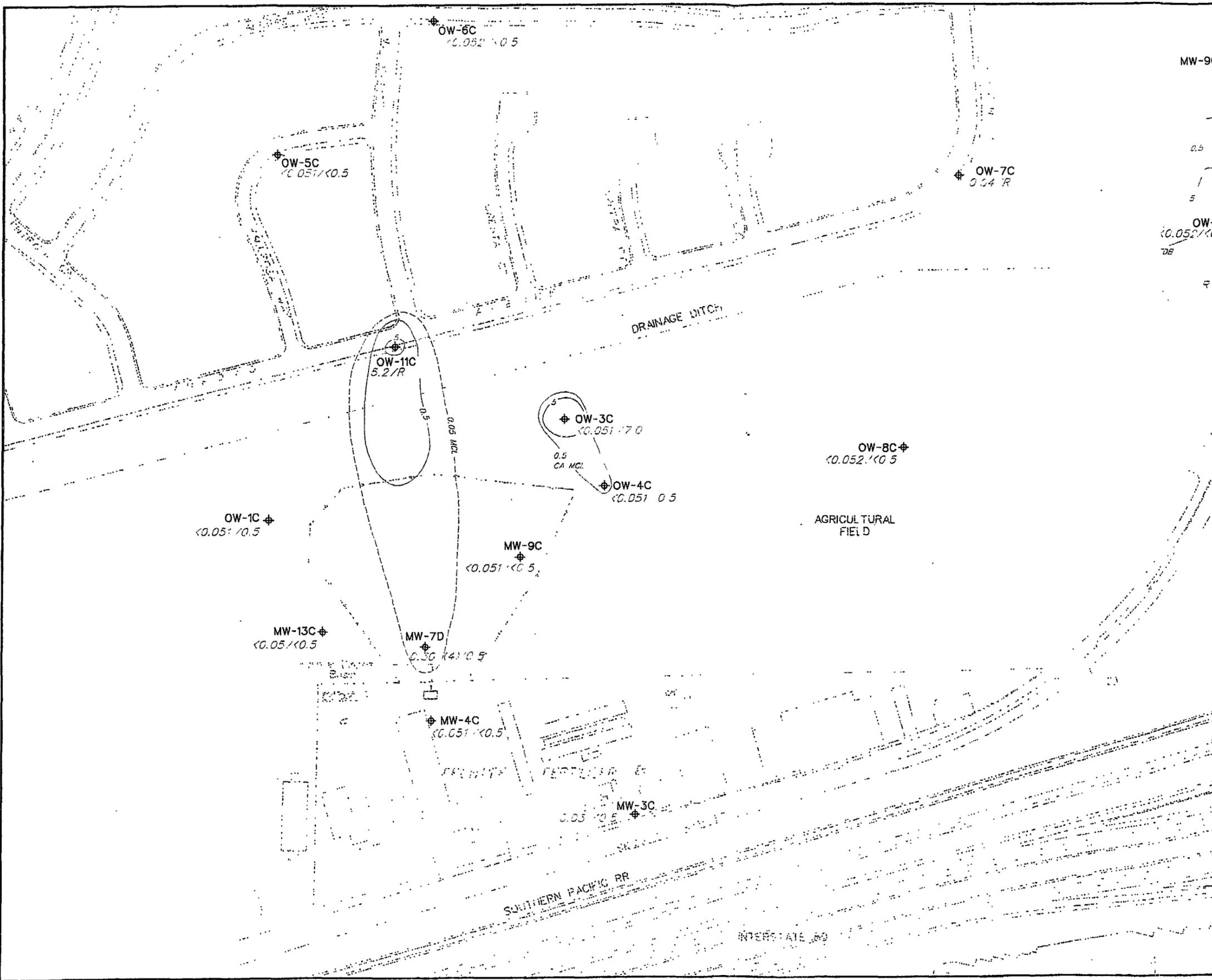
- 1) EDB concentrations in ug/L determined using EPA Method 504 unless otherwise noted.
- 2) CCl<sub>4</sub> by 25 ml purge unless otherwise noted.
- 3) Units: ug/L
- 4) Concentration determined by 5 ml purge method.
- 5) Concentration determined by 25 ml purge method.



Approximate Scale in Feet

|  |            |             |     |
|--|------------|-------------|-----|
| <b>Bechtel</b>   |            |             |     |
| SAN FRANCISCO  |            |             |     |
| FRONTIER FERTILIZER<br>DAVIS, CALIFORNIA   |            |             |     |
| <b>1, 2-DIBROMOETHANE (EDB) AND CCl<sub>4</sub><br/>IN THE S2 ZONE, OCTOBER 1997</b> |            |             |     |
|  | Job Number | Drawing No. | Rev |
|  | 20376      | FIGURE 2    | 0   |

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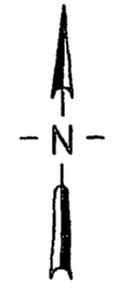


**EXPLANATION**

- MW-9C ⊕ Monitoring Well
- Interpreted 1,2-Dibromoethane (EDB) Concentration Contour, October 1997
- Interpreted Carbon Tetrachloride (CCl<sub>4</sub>) Concentration Contour, October 1997
- Concentrations at each location are represented for EDB/CCl<sub>4</sub>
- CCl<sub>4</sub> Data Rejected

**NOTES:**

- 1) EDB concentrations in ug/L determined using EPA Method 504 unless otherwise noted.
- 2) CCl<sub>4</sub> by 25 ml purge
- 3) Units: ug/L
- 4) Concentration determined by 25 ml purge method.



Approximate Scale in Feet

|   |             |     |
|---|-------------|-----|
| <b>Bechtel</b>  |             |     |
| SAN FRANCISCO   |             |     |
| FRONTIER FERTILIZER<br>DAVIS, CALIFORNIA                                      |             |     |
| 1, 2-DIBROMOETHANE (EDB) AND CCl <sub>4</sub><br>IN THE A1 ZONE, OCTOBER 1997 |             |     |
| Job Number  | Drawing No. | Rev |
| 20376   | FIGURE 3    | 0   |

### **3.0 Sampling and Analysis Program and Rationale**

The proposed Phase III groundwater investigation will include exploratory drilling, groundwater sampling and analysis, and monitoring well installation. The exploratory drilling will consist of a SimulProbe™ survey program involving drilling up to eight borings to depths up to 80 feet bgs (B-11 through B-18). The SimulProbe™ sampler is similar to a HydroPunch™, but has the advantage of forming a more effective seal with the surrounding formation, not leaking drilling fluid or groundwater through joints into the sample canister, and the sample canister can be back pressurized with nitrogen to assure a more representative sample is recovered.

Groundwater samples will be collected from each of the two upper most water bearing zones, S-1 and S-2. These samples will be submitted for quick turn around time analysis via Method 504 for EDB and 1,2-dibromo-3-chloropropane (DBCP) and via the 25 ml purge method for low level volatiles, including CCl<sub>4</sub>. Monitoring wells will be installed where EDB and CCl<sub>4</sub> concentrations are below the lowest available contract required quantitation limit, 0.05 ppb and 0.5 ppb, respectively, in the S-1 and S-2 water bearing zones. All four borings for monitoring wells will be logged and the monitoring well locations surveyed (OW-9A, OW-9B, OW-10A, and OW-10B). Groundwater samples will be collected from the newly installed wells after development and submitted for laboratory analysis.

#### **3.1 Sampling Recommendations**

The proposed locations of the first two exploratory borings are as follows: B-11 will be approximately 300 feet north of the OW-6 well cluster; and B-12 will be approximately 300 feet north east of the OW-7 well cluster. These locations were chosen based on the previous sampling results presented in Figures 1 and 2.

Each boring will be drilled to the S-1 and S-2 groundwater zones and groundwater samples will be extracted for chemical analysis from each groundwater zone using a SimulProbe™ sampler. The stratigraphic and chemical data from each preceding boring will be used to adjust the depth and location of the next boring. The placement of additional borings will be determined based on analytical results. If no contaminants of concern are detected in samples collected from both the S-1 and S-2 groundwater zones, then the next boring locations will be half way between the nearest existing well and the previous location. If a contaminant of concern is detected in either sample collect from the S-1 or S-2 groundwater zones, then the next boring location will be 150 feet beyond the previous location. If contaminants are detected in samples collected from the S-1 and S-2 groundwater zones the borings will be properly abandoned as described in Section 5 of the FSP.

Each groundwater sample collected during the SimulProbe™ survey will have a unique sample number. The sample number will be the boring location number (B-11 through B-18) appended with an A for a sample collected from the S-1 groundwater zone and a B for a sample collected from the S-2 groundwater zone. Therefore, groundwater sample B-10A will represent a sample collected from the S-1 groundwater zone from boring B-10 and groundwater sample B-10B will represent a sample collected from the S-2 groundwater zone from boring B-10.

#### **3.2 Monitoring Well Installation Recommendations**

Based on the information collected during the SimulProbe™ survey, two specific areas for groundwater monitoring will be selected. Monitoring well clusters will be constructed beyond the northern and northeastern leading edges of the groundwater pesticide plume. The locations of the monitoring wells

will be approximately 150 feet beyond the leading edges of the plumes. This is based on the 75 ft/yr rate of groundwater contaminant migration, and an observation period of 2 years. Each well cluster will be comprised of two wells, one each screened in the S-1 and S-2 units. Since these monitoring wells will be located beyond the groundwater plume, they will serve as observation wells.

Following installation of the wells, each well will be developed as described in Section 5. Groundwater samples will be collected from the new monitoring wells after development and submitted for the laboratory analyses described in Section 3. The analytical data will be compared to the initial water quality data for groundwater samples collected during exploratory drilling.

Each monitoring well in the clusters will have a unique well identification number. The well identification will be appended with either the letter A or B to coincide with the groundwater zone the well is screened in (A = S-1 zone, B = S-2 zone). The wells will be numbered as follows: the two wells drilled at the northern leading edge of the plume will be labeled OW-9A, and OW-9B. The two wells drilled at the north eastern leading edge of the plume will be labeled OW-10A and OW-10B.

### 3.3 Analysis Recommendations

#### *SimulProbe™ Samples*

Each groundwater sample collected during exploratory drilling using a SimulProbe™ sampling device will be analyzed for EDB, DBCP, and carbon tetrachloride using EPA Method 504 and the 25 ml purge method for volatile organic compounds to achieve detection limits of 0.05 µg/l, 0.05 µg/l, and 0.5 µg/l, respectively. The EPA Region IX laboratory will be utilized for groundwater sample analyses via both methods. The analyses will be used to characterize the vertical distribution and concentration of pesticides and carbon tetrachloride at each borehole location and to select locations for monitoring well installation. Specific conductance, pH, and temperature will also be measured for every groundwater sample collected during drilling. Water levels will be measured in each permeable water-bearing zone encountered during drilling.

#### *Monitoring Well Samples*

Groundwater samples will be collected from each monitoring well after development as described in Section 5. Groundwater samples will be analyzed by the EPA Region IX laboratory using the same methods for the same analytes as determined in the SimulProbe samples. Analytical results will be compared to the initial sampling and analyses results performed during the SimulProbe™ survey for a quality and consistency check. Field measurements such as pH, temperature, and conductivity will be collected from each monitoring well during development.

### 4.0 Request for Analyses

The Frontier Fertilizer site was identified as a potential hazardous waste site and entered into the CERCLIS database on August 1, 1985 (CAD 071530380). Bechtel will conduct this field sampling effort to gather data as part of an RI under CERCLA. The anticipated sampling dates for this sampling effort are June 10 through June 26, 1998. Because of the nature of this Phase III RI, it is impossible to accurately predict the exact number of samples to be collected and analyzed. The following numbers of samples represent the range (minimum and maximum) of samples (including duplicate, QA/QC, and equipment rinsate samples) expected to be collected and analyzed as part of this effort. In addition, a reasonable estimate of the number of samples expected is given.

- 20 to 30 low concentration water samples for EDB, DBCP, and carbon tetrachloride using EPA Method 504 and the 25 ml low level volatiles method to achieve detection limits of 0.05 µg/l, 0.05 µg/l, and 0.5 µg/l, respectively. The EPA Region IX laboratory will be utilized for the analyses. It is estimated that 25 water samples will be collected and analyzed by the EPA Region FASP laboratory.

Table 4-1 shows the preservative requirements, analytical and contract required holding times, and sample container requirements for each analyses by matrix. Client Request Forms are included as Appendix A.

#### 4.1 Groundwater Sample Analyses

Since the exact number of samples collected and analyzed as part of this Phase III RI is unknown, the following frequencies of quality control samples (including duplicates and laboratory QA/QC samples), instead of the numbers and locations, are given.

- Duplicate samples will be collected from areas of known or suspected contamination. Each analytical group for which a standard sample is analyzed will also be tested for in one or more duplicate samples.
- Laboratory QC samples will be collected from areas of known or suspected contamination. At a minimum, 1 laboratory QC sample is required per week or one per 20 samples (including blanks and duplicates), whichever is greater. If the sample event lasts longer than 1 week or involves collection of more than 20 samples per matrix, additional QC samples will be designated.

#### *EPA Region IX Laboratory*

As described in Section 3, groundwater samples will be collected using a SimulProbe™ sampling device from up to 8 exploratory bore holes. Groundwater samples will be collected from the S-1 and S-2 groundwater zones in each exploratory bore hole to define the leading edge of the pesticide and carbon tetrachloride/pesticide contaminated groundwater plumes.

As described in Section 3, groundwater samples will also be collected from the newly installed monitoring wells. All of these samples will be analyzed on a quick turn around basis for EDB and DBCP via Method 504 and for carbon tetrachloride by the 25 ml purge low level volatiles method.

#### 4.2 Equipment Rinsate Sample Analyses

Equipment rinsate samples will be collected each day that equipment is decontaminated in the field as described in Section 5. Equipment rinsate samples will be analyzed for EDB and DBCP by EPA Method 504 and for CCl<sub>4</sub> via the EPA 25 ml purge low level volatiles method. The EPA Region IX laboratory will be utilized for all equipment rinsate analyses.

### 5.0 Field Methods and Procedures

SimulProbe™ sampling and monitoring well installation will be conducted in accordance with the applicable procedures presented in Section 5 of the FSP and the technical specification provided as Appendix B.

**Table 4-1  
Request for Analyses**

| <b>Laboratory Matrix</b>              | <b>EPA Region IX Laboratory<br/>Water</b> | <b>EPA Region IX Laboratory<br/>Water</b>                      |
|---------------------------------------|---|--|
| <b>Analysis</b>                       | EDB, DBCP by EPA Method 504               | Carbon tetrachloride by 25 ml purge low level volatiles method |
| <b>Preservatives</b>                  | Cool to 4°C                               | Add 1:1 HCl to pH<2, cool to 4°C                               |
| <b>Analytical Holding Time(s)</b>     | Hold <14 days                             | Hold <14 days  |
| <b>Contract Holding Time(s)</b>       | Hold <10 days                             | Hold <14 days  |
| <b>Sample Containers per Analysis</b> | Three 40-ml VOA vials                     | Three 40-ml VOA vials  |

## **6.0 Disposal of Investigation-Derived Wastes**

Disposal of investigation-derived wastes will be conducted in accordance with the requirements specified in Section 6.0 of the FSP.

## **7.0 Sample Documentation and Shipment**

All sample documentation and shipment will conform to the requirement specified in Section 7.0 of the FSP.

## **8.0 Quality Control**

All quality control measures specified in Section 8.0 of the FSP and applicable section of the QAPjP will be followed during the conduct of the work described in this FSP amendment.

**Appendix A**  
**Client Request Forms**

**Attachment A**

# **STANDARD OPERATING PROCEDURES FOR IN-SITU SAMPLING WITH THE MAXI SIMULPROBE**

## **1.0 INTRODUCTION**

The SimulProbe is an in-situ sampling device which allows the simultaneous collection of either in-situ soil gas with soil core or in-situ liquid with soil core. The purpose of the tool is to collect soil samples simultaneously with liquid or gas which are directly correlative and can be economically collected in one sampling event. The directly correlative nature of the fluids and solids provides more accurate interpretations about:

- (1) The relationship between contaminant distribution and stratigraphic characteristics.
- (2) The opportunity to compare contaminant concentration in gas and liquid with that found in directly correlative soil core.

## **2.0 DESCRIPTION**

The SimulProbe (see schematic) is shaped much like a split spoon sampler, but has some distinct differences. A listing of the tool's important dimensions are presented below:

- (1) 3.38 inches outside diameter (OD) along the length of the entire tool; excluding the cutting edge of the Drive Shoe.
- (2) The inside diameter (ID) of the core barrel is 2.5 inches.
- (3) The core barrel length is 18 inches.
- (4) The Drive Shoe length is 3 inches.
- (5) The 2 liter stainless steel water canister is a 19 inch long modular unit to the base unit.

**3.0 OPERATION: Assembly and Operating Instructions for Soil/Soil Gas Mode Using Wire-Line Down-Hole Hammer (or Up-Hole Hammer) and Up-Hole Vacuum Pump**

- 3.1 Place clean Teflon Tubes along the slots in the edges of the Core Barrel Primary Half and push Teflon Tubes into the fluid pathway holes at the top of the slots. The lower end of the Teflon Tubes should end about 1/4 inch short of the lower end of the slots.
- 3.2 Insert a Reed Valve into the Core Barrel Primary Half fluid displacement port at top of the core barrel.
- 3.3 Place a Blue O-Ring on the bottom of a Vent Cover and push the Nipple of the Vent Cover into the bottom of the Reed Valve.
- 3.4 Place Encapsulated Core Sleeves into the Core Barrel Primary Half.

**Note:**

- (1) **Do not** use standard 6"-long core sleeves as they will not allow the SimulProbe Drive Shoe to fully close. (STI MaxiSimulProbe core sleeves are 5 and 13/16 inches long.)
  - (2) **Do Not** remove the core sleeve encapsulation. SimulProbe core sleeves come pre-encapsulated in a polyolifin shrink-wrap. The encapsulation helps to minimize cross contamination of the sleeves prior to and after use and prevents sand locking between the bottom core sleeve and the shoe sleeve.
- 3.5 Place the Core Barrel Cover Half over Core Barrel Primary Half, making sure that the Teflon Tubes are in the grooves between the core barrel halves.
  - 3.6 Clamp the core barrel halves together using the chain vise on the driller's breakdown rack. This allows the Coupling to be easily screwed into place.
  - 3.7 Place the Circular Screen over circumferential channel and fluid entry ports at the bottom of core barrel. The circumferential channel is the channel on the outside of the core barrel halves just above the threads to which the Coupling is later attached.
  - 3.8 Screw the Coupling onto the bottom of the core barrel, being careful to keep you fingers back from the sharp edges of the screen.

- 3.9 Place the Rust-colored O-Ring into the bottom of the Cover Sleeve and insert the Shoe Sleeve, male threads first, into the bottom (threaded end) of the Cover Sleeve. Attach this assembly to the bottom of the core barrel by screwing it into the Coupling. (See schematic)
- 3.10 Screw the Drive Shoe onto the bottom of the Cover Sleeve.
- 3.11 Screw the Sampler Head into the top of the Core Barrel Primary Half; making sure to place a Black O-Ring above the threads of the Sampler Head.
- 3.12 Wrap Teflon tape around the pipe threads of a 3/8" NPT x 1/4" Swagelok Connector and screw it into one of the ports on top of the Sampler Head. A 3/8" NPT Plug with Teflon tape should be placed in the second port.
- 3.13 Close the Drive Shoe and pull a Gooch Tube over the junction between the core barrel and the Drive Shoe. A Gooch Tube is a 2 inch piece of rubber tubing which is included in your soil gas (or groundwater) kit. The purpose of the Gooch Tube is to hold the Shoe in the closed position while the tool is lowered into the borehole.
- 3.14 Attach the down-hole end of a vacuum line to the Swagelok fitting on the top of Sampler Head; using a 3/16 inch ID, 1/4 inch OD Teflon line.
- 3.15 Attach the SimulProbe to a downhole hammer using a one-foot long AW rod and lower the SimulProbe to the bottom of the borehole.
- 3.16 Attach the up-hole end of the vacuum line to the vacuum pump intake.
- 3.17 For the drive and sniff mode, turn on the vacuum pump after driving the SimulProbe about 6 inches and use an OVM (or OVA) to monitor the vacuum pump exhaust. Hammer in the SimulProbe a total of 21 inches to collect the soil core sample, pausing where appropriate to "sniff" the vacuum pump exhaust with an OVM.
- 3.18 Pull SimulProbe back 1 to 2 inches to retract the sliding Drive Shoe and expose the Circular Screen.
- 3.19 Use vacuum pump to purge vacuum line and collect soil gas sample. The purging process is complete when the OVM readings stabilize, and the flow meter indicates that the specified purge volumes have been removed. The MaxiSimulProbe in the soil gas mode has a 200 ml purge volume. The 3/16" id Teflon line has a purge volume of 5.43 cc/ft.
- 3.20 Pull SimulProbe to surface for core retrieval, disassembly, and decontamination.

3.21 Preferably use SimulProbe wrenches to disassemble SimulProbe, except as noted below. (The wrenches are designed to maximize mechanical advantage and prevent damage.) The chain vise on the driller's breakdown table may be used to hold the Cover Sleeve while removing the Drive Shoe and to hold the core barrel while removing the Coupling.

**4.0 OPERATION:            Assembly and Operating Instructions for Soil/Soil Gas Mode Using the Drive and Sniff Technique for Continuous and Discrete Soil Gas and Pressure Response Profiling of Soil Core**

4.1 Follow assembly instructions as described in Section 3 above.

4.2 Drive the SimulProbe 4 to 6 inches below the bottom of the borehole so that the length of the drive shoe and the bottom edge of the core barrel are fully buried in the new geologic material.

4.3 Turn on the vacuum pump and purge the vacuum line until the OVM (or OVA) and vacuum pressure response readings stabilize and the flow meter indicates that sufficient volume has been purged. Then record OVM (or OVA), vacuum gauge pressure and flow meter response.

4.4 Repeat Step 3 as many time as necessary for the desired detail along the core length.

4.5 Remove the SimulProbe after it has penetrated 21 inches. Breakdown core barrel and compare geologic characteristics of the core to the OVM and pressure response logs.

## **5.0 OPERATION/Soil/Groundwater Mode Modular Groundwater Canister**

### **Assembly and Operating Instructions for Soil/Groundwater Mode Using Wire-Line Down-Hole Hammer, Modular Water Canister, and Ambient Hydrostatic Pressure**

- 5.1 Follow Section 3.0 instructions (1) through (10) above (excluding Step 9).
- 5.2 Screw a Reed Valve Support, with o-ring, into the bottom of the Water Canister Base and attach silicone Reed Valve.
- 5.3 Place a black o-ring above the threads of the water canister base.
- 5.4 Screw the Water Canister Base into the top of the Core Barrel Primary Half.
- 5.5 Place a Black O-Ring above the threads at the bottom of the Water Canister and screw the Water Canister into the Water Canister Base.
- 5.6 Wrap Teflon tape around the threads of the Upper Reed Valve Support and screw it into the base of the Sampler Head.
- 5.7 Wrap Teflon tape around the threads of the Hex plug and screw it into the center of the Upper Reed Valve Support
- 5.8 Place a Black O-Ring above the Sampler Head's threads and screw the Sampler Head into the top of the Water Canister.
- 5.9 Close the Drive Shoe and pull a Rubber Gooch Tube over the junction between the core barrel and the Drive Shoe.
- 5.10 Push and snap Yellow Diaphragm (hydrostatic heads of 50 feet or less) into cutting edge of Drive Shoe. Use stainless steel Diaphragm for pressures greater than 50 feet of hydrostatic head. Then, place inside of drive shoe. If used, the stainless steel diaphragm is pushed onto the edge of a standard 2.5 inch sand catcher and placed behind the drive shoe.
- 5.11 Roll on latex Condom from shoe along length of probe. Be careful not to rest the weight of the probe on the Swagelok connector during this procedure. Place the second Gooch tube over the Condom; along the bottom of the drive shoe. This serves as a bumper to protect the Condom as the tool is lowered into the borehole.
- 5.12 Attach MaxiSimulProbe to one-foot AW rod. Be careful not to rest weight of probe on Condom covered shoe while attaching the AW rod and to the drive hammer.

- 5.13 Attach Teflon tube (pneumatic line) to Swagelok connector at top of probe.
- 5.14 Driller raises probe with winch line and lowers a few feet into casing.
- 5.15 Attach the up-hole end of spool to the inert gas tank regulator. The three way valve handle should be pointed towards regulator line.
- 5.16 With the probe suspended a few feet down inside the casing, pressurize the water canister. The rule of thumb for pressurizing is to assume 60 PSI/100 feet of hydrostatic head (pure water is approximately 43 PSI/100 feet; the additional pressure assumes suspended and dissolved solids and thus increased specific gravity). See attached tables from Groundwater and Wells, Driscoll, 1987. **Note: for safety, the SimulProbe should always be pressurized and depressurized inside the casing.**
- 5.17 Observe line pressure gauge on regulator. When line pressure is equal to 60 PSI/100 feet of hydrostatic head (0.6 PSI/ft), close 3-way valve on hose spool to trap pressure. Closed position is when the valve handle is at 90 degrees to 3-way valve's body. The 3-way valve is attached to the side of the hose reel and is the uphole terminus for the Teflon line. The line is attached to the center port on the "T" shaped valve. One of the two side ports is connected to the Nitrogen gas supply and the second is attached to a short (2.5") length of Teflon line. This short length is used for the inverted soda bottle method described in section 5.22. If the valve is positioned halfway between these two ports, which is 90 degrees to the 3-way valve's body, all three ports are closed to each other. Pointing the valve handle at either of the two side ports connects the central port to that side port.
- 5.18 Disconnect the Teflon line from the regulator and hook it onto the side of the spool.
- 5.19 Slowly lower the SimulProbe to the bottom of the borehole and hammer it 21 inches into subsurface to collect the soil core.
- 5.20 Pull the SimulProbe back 2 to 3 inches to retract the sliding Drive Shoe and expose the Circular Screen.
- 5.21 Open valve to allow pressure bleed off through the short length of Teflon tube attached to the valve.
- 5.22 Allow water to enter tool under ambient hydrostatic pressure. After the initial pressure bleed of, the fill rate can be observed by placing the open

end of the Teflon tube into a bucket of water. For extremely slow fill rates, the end of the Teflon tube can be placed inside an inverted water filled bottle inside the bucket (i.e. one liter plastic soda bottle). When the bottle is full, it can be emptied and reused for additional volume measurements.

- 5.23 After sufficient water sample has been collected, repressurize the SimulProbe by following steps 15 through 18.
- 5.24 Pull the SimulProbe to a few feet below the top of the casing and depressurize. **Note: for safety, the SimulProbe should always be pressurized and depressurized inside the casing.**
- 5.25 Remove the SimulProbe from the casing and stand it vertically on ground.
- 5.26 To remove water canister, unscrew the Water Canister Base from the Core Barrel Primary Half (using spanner wrenches). Keep the Water Canister upright to minimize sample agitation, and insert a short length of 5/16-inch OD Teflon tube through the bottom of the Reed Valve to drain and collect a water sample from the bottom of the Water Canister.
- 5.27 Disassemble the core barrel using SimulProbe wrenches and remove the soil core.

**6.0 OPERATION: Assembly and Operating Instructions for Soil/Groundwater Mode Using Wire-Line Down-Hole Hammer, Modular Water Canister and Vacuum Assist**

Instructions are the same as above with the additional step of using a peristaltic or vacuum pump to lift water into the water canister. Vacuum assist is not recommended when sampling for volatile compounds.

**7.0 OPERATION: No Modular Water Canister Required**

**Soil/Groundwater Mode Using Peristaltic Pump to Pump Groundwater to Surface**

This is a shallow (less than 25 feet) groundwater sampling technique for non-VOC sample collection. There is no Water Canister required when it is thought that the formation permeability is sufficient to allow water to be drawn through a peristaltic pump.

The peristaltic pump technique will only work if the piezometric water level

of the formation being sampled is 25 feet or less below the ground surface. Note: Suspended and dissolved solids will increase the fluid specific gravity and thus reduce the effective depth range of a peristaltic pump.

**8.0 OPERATION: Assembly and Operating Instructions for Using Soil/Groundwater Mode Using Up-Hole Hammer, Hollow NW Rods and Sample Bailer**

- 8.1 Assemble the SimulProbe as in Section 7.0.
- 8.2 Attach a hollow NW rod to the top of the SimulProbe using a hollow NW/AW pin converter. This will allow fluids to pass through the Upper Reed Valve port at the bottom of the Sampler Head and into the hollow NW rods for collection by bailer.
- 8.3 Lower the SimulProbe to the bottom of the borehole, adding more NW rods as needed. **Use O-rings or Teflon paste between NW rod connections to prevent cross contamination from borehole fluids.**
- 8.4 Drive the SimulProbe 21 inches using an up-hole hammer on the top of the NW rod string.
- 8.5 Retract the SimulProbe 2 to 3 inches to expose the Circular Screen. The water sample will flow into the hollow NW rods.
- 8.6 Lower a decontaminated water level sensor inside the NW rods to determine when there is enough water to collect a sample.
- 8.7 Collect the water sample using a Teflon bailer.
- 8.8 Withdraw the SimulProbe and collect the soil core sample.

**9.0 OPERATION: Assemble and Operating Instructions for Using Soil/Groundwater Mode Using Up-Hole Hammer, NW Rods/Bailer and Peristaltic Pump**

The NW Rods/Bailer and Peristaltic Pump water sampling methods can be combined when sampling protocols include the collection of samples for volatile compounds and the sampled formations have shallow (<25' BGS) piezometric water levels.

- 9.1 Follow the instructions for sampling using hollow NW rods and a bailer, except add a peristaltic vacuum line to the Swagelok fitting on top of the Sampler Head.
- 9.2 Once the SimulProbe has been hammered into place and opened, the first sample should be collected by bailer for volatile compounds.
- 9.3 Subsequent samples can then be collected by peristaltic pump.

**10. DECONTAMINATION**

Follow procedures specified in site specific work plan and/or quality assurance project plan for standard operating procedures for sampling device decontamination. Always use a new consumable kit for each sample. Reuse of consumables may result in cross contamination of samples through incomplete decontamination or from leakage through damaged O-Rings and Reed Valves.

# MAXI-SIMULPROBE®

SOIL / GROUNDWATER

TO RIG

TO RIG

Teflon lines to surface used for soil gas sampling and nitrogen back-pressure for groundwater sampling.

Water canister capacity 2 liters

Tool length 45.5 inches (116mm)

CONDOM

Condom covers core barrel section, sealing out bore hole fluids.

Teflon Tube Pathway

Groundwater or gas flow path.

GROUNDWATER

TEFLON LINE TO SURFACE

NPT PLUG

SWAGLOK CONNECTOR

Modular Water Canister (2 liters-stackable)

One-way reed valve

Core Barrel Section

50-Mesh screen 1/2 in length

4" Long Extension Screen Available.

CLOSED

OPEN

## **Appendix B**

# **Technical Specification for Drilling, Sampling, and Monitoring Well Installation**

# FRONTIER FERTILIZER

## TECHNICAL SPECIFICATION

### FOR

### DRILLING, SAMPLING, AND MONITORING WELL INSTALLATION

Prepared for

United States Environmental Protection Agency

Region IX

By

Bechtel Environmental, Inc.

San Francisco, California

|   |         |   |                   |               |               |
|---|---------|---|-------------------|---------------|---------------|
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| A   | 4/13/98 | Issued for Review   | WAM               | RBD           | PMA           |
| REV   | DATE    | REASON FOR REVISION   | ORIG.             | PROJ.<br>MGR. | ENG.<br>SUP.  |
|  |         | FRONTIER FERTILIZER<br>DRILLING, SAMPLING, AND<br>MONITORING WELL<br>INSTALLATION | JOB NO. 20376-028 |               |               |
|   |         |   | SPECIFICATION NO. |               | REV.          |
|   |         |   | 20376-028-012     |               | 0             |
|   |         |   |                   |               | Sheet 1 of 20 |

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COMPLETION

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COMPLETION

# General

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## 1.1 OBJECTIVE

The work shall consist of drilling approximately eight exploratory borings to depths up to 80 feet. Groundwater samples shall be collected from each of the two permeable water-bearing zone during the exploratory drilling of six of these borings using a Maxi-Simulprobe sampling tool. Based on the results of this exploration, four groundwater monitoring wells will be installed; two wells with a total depth of about 80 feet each, and two wells with a total depth of about 40 feet each.

This specification defines the minimum standards of quality, safety, procedure, and performance required to successfully meet the objective of this program.

The work to be performed by the Subcontractor shall consist of furnishing all supervision, labor, equipment, tools, supplies, and materials; and the performance of all operations in connection with the designated subsurface investigation and in accordance with the provisions of this specification.

## 1.2 LOCATION OF WORK

The drilling operations shall be conducted at the site of the former Frontier Fertilizer facilities located at 4309 Second Street in Davis, Yolo County, California. Figure 1 shows the site location; Figure 2 shows the approximate locations of the existing groundwater monitoring wells.

## 1.3 STANDARDS AND DEFINITIONS

The following is an alphabetical reference list of the standards and definitions contained in this technical specification:

|          |   |
|----------|---|
| ASTM     | American Society of Testing Materials   |
| DOT      | U.S. Department of Transportation   |
| Driller  | Person designated by the Subcontractor as the field representative  |
| DWR      | California Department of Water Resources  |
| Engineer | Bechtel Environmental, Inc., and any of its authorized geologists, engineers, or other representatives assigned to this job |
| EPA      | U.S. Environmental Protection Agency, Region IX   |
| I.D.     | Inner Diameter  |
| O.D.     | Outer Diameter  |

|             |   |
|-------------|---|
| OSHA        | Occupational Safety and Health Administration                                     |
| PPE         | Personal Protective Equipment   |
| Well Volume | Volume of water calculated to be contained within the well casing and filter pack |

Unless otherwise specified, materials and field operations shall conform to the latest issue of the following codes and standards and shall apply to the extent indicated herein, including:

|                    |   |
|--------------------|---|
| ASTM C150:         | Standard Specifications for Portland Cement   |
| ASTM D1586:        | Standard Method for Penetration Test and Split-Barrel Sampling of Soils                     |
| ASTM D 1785:       | Standard Specification for Polyvinyl Chloride (PVC) Plastic Pipe, Schedules 40, 80, and 120 |
| DWR Bulletin 74-81 | Water Well Standards: State of California, Dec. 1981  |
| DWR Bulletin 74-90 | California Well Standards (Supplement to Bulletin 74-81): State of California, June 1991    |

#### 1.4 ACCESS TO WORK

The Subcontractor shall provide his own means of moving equipment on site and between drilling locations. The Subcontractor may use available existing public or private roads and shall be responsible for the maintenance and repair of such roads to keep them in the condition that existed immediately prior to start of the use by the Subcontractor.

Physical access to the drill sites may be restricted because of the field conditions. The Subcontractor shall attend a walkthrough at the site to ascertain what provisions, if any, need to be made to set up on each of the preferred drilling locations.

## **Safety and Health Requirements**

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The Subcontractor shall certify in writing to Bechtel that all Subcontractor personnel who will perform work at the site have completed the necessary Occupational Safety and Health Administration (OSHA) training and have had the proper medical surveillance for work at hazardous waste sites in accordance with the Code of Federal Regulations, Title 29, Part 1910.120, entitled "Hazardous Waste Operations and Emergency Response."

Upon award of the subcontract, the Subcontractor shall be responsible for developing a Subcontractor Site Safety and Health Plan for onsite implementation. The plan shall be consistent with, and at least as stringent as, the Bechtel-prepared Frontier Fertilizer Safety and Health Plan. As an option, the Subcontractor may fulfill this requirement by adopting, in writing, the Bechtel-prepared Frontier Fertilizer Safety and Health Plan.

The Subcontractor shall designate one individual as the Safety and Health contact who will be responsible for serving as the Subcontractor's site Safety and Health representative. The Subcontractor's Safety and Health representative shall be responsible for ensuring that all the Subcontractor's employees working at the site comply with the established Bechtel-prepared Frontier Fertilizer Safety and Health Plan. At a minimum, the Subcontractor shall provide all personal protective equipment (PPE) and other safety and health equipment, as specified in the Bechtel-prepared Frontier Fertilizer Safety and Health Plan for each Subcontractor employee working at the site. The Subcontractor shall ensure that all PPE is clean and in proper working order prior to the start of any work.

The Subcontractor shall conduct the work described in this technical specification with due regard to safety, and shall take measures to ensure the safety of the public, Subcontractor employees, and other onsite workers. The Subcontractor shall implement all measures required to comply with the applicable OSHA laws and regulations. If the Subcontractor has not taken the necessary steps for compliance, Bechtel shall direct the Subcontractor to suspend all of his work. Work shall not resume until the Subcontractor has implemented the required measures to the satisfaction of Bechtel.

## **Equipment and Materials**

---

### **3.1 EQUIPMENT FURNISHED BY SUBCONTRACTOR**

#### **3.1.1 Drilling Equipment**

The Subcontractor shall provide a truck-mounted, hydraulic-rotary drill rig and the drill tools, supplies, and accessories necessary to achieve the required boring diameters and depths, conduct the sampling required, and install/develop the monitoring wells. All equipment brought to the site shall be clean and in good working condition, as approved by the engineer. The equipment shall be capable of using hollow-stem flight augers to drill holes with nominal diameters ranging from 7 to 11 inches and to depths up to 80 feet.

The Subcontractor shall also provide equipment and materials to capture and contain designated drill cuttings in and all fluids ejected from the borings in containers such as temporary storage tanks and/or tank trucks. The soils shall be transported to a central onsite area and dumped on the ground in an area directed by the engineer. Upon completion of all soil borings, the Subcontractor shall grade the soils disposal area. All contained fluids shall be transported to a central onsite liquid holding tank.

#### **3.1.2 Maxi-Simulprobe Groundwater Sampler**

Groundwater samples will be collected during drilling from 2 permeable water-bearing zones located at depths ranging from 6 to 80 feet. A Maxi-Simulprobe groundwater sampler shall be used for sample collection. An illustration of a standard Maxi-Simulprobe sampling scheme is shown in Figure 3.

#### **3.1.3 Decontamination Equipment**

The Subcontractor shall provide a steam cleaner with appropriate accessories and personnel required to clean all drill rigs, equipment, tools, and supplies that shall come in contact with the subsurface materials, as further discussed in Section 4.3.

#### **3.1.4 Water-Level Meter**

The Subcontractor shall furnish a functioning water-level meter with electrically sensed probe and depth-graduated tape, in feet, to measure water levels during drilling, sampling, and well development.

### 3.1.5 Equipment for Well Development

The Subcontractor shall supply a development rig, bailer, surge block, swab, submersible pump and generator or airlift pipe and compressor, and other materials and accessories needed for well development.

The submersible pump and generator or airlift pipe and compressor shall be capable of delivering pumping rates from less than 1 to 30 gallons per minute (gpm) at depths ranging from 10 to 80 feet.

## 3.2 MATERIALS FURNISHED BY THE SUBCONTRACTOR

### 3.2.1 Cement

Cement used for the grout mixture shall conform to ASTM Standard C150 and shall be Type II Portland Cement.

### 3.2.2 Grout

Grout shall consist of a mixture of 7.5 gallons of water and 2.5 pounds of powdered bentonite per sack of Type II Portland cement.

### 3.2.3 Bentonite

Bentonite shall be a high-swelling, sodium-based Wyoming-type bentonite supplied in free-flowing powdered, and pellet form.

### 3.2.4 Riser Casing

Riser casing shall be threaded, flush-jointed, Schedule 40 polyvinyl chloride (PVC) pipe for all wells. Casing for all permanent monitoring wells shall be 4-inch nominal diameter and the top of the riser casing shall contain a vented PVC cap. The casing shall be clean, straight, and free of obstructions. All PVC casings shall conform to ASTM Standard D1785. New casings shall be used for all permanent monitoring wells.

### 3.2.5 Screens

Well screens shall consist of threaded, flush-jointed, wire-wrapped, Schedule 40 PVC. The screens shall have a nominal 4-inch diameter for all permanent monitoring wells. The width of the slots shall be 0.020 inches. Screens shall be provided in 2-, 5- and 10-foot lengths. The bottom of the screen shall have a 2-foot sump.

### 3.2.6 Filter Pack

Filter pack materials shall be clean, rounded #2/12 medium silica sand with less than 5 percent nonsiliceous material. U.S. standard sieve size gradation shall be 16 x 30 for all 4-inch monitoring well screens.

### 3.2.7 Protective Casing and Concrete Pad

Protective surface casing with a lockable steel cap and a reinforced concrete pad shall be installed for all monitoring wells. The surface casing shall be a minimum 8-inch-diameter steel casing extending at least 3 feet below and 2 feet above the ground surface, unless otherwise directed by the engineer. Alternatively, the engineer may require a flush mounted surface completion. The protective casing shall be centered over the PVC well casing and inserted into the grouted annulus prior to grout hardening. A reinforced concrete well pad of nominal dimensions 3 feet by 3 feet by 4 inches square shape shall be constructed at the ground surface around the protective casing. Well pads shall be sloped slightly away from the casing to prevent ponding of water around the well. The reinforcement used in the well pad shall be standard chicken wire or equivalent, as approved by the engineer.

### 3.2.8 Plastic Sheeting

The Subcontractor shall provide plastic sheeting to keep clean samplers, tools, and materials from contacting the ground surface; to temporarily store clean drilling equipment; and to lay out boring soil cuttings for inspection.

The Subcontractor shall also supply absorbent padding for cleanup of accidental spills of gasoline or other nonaqueous fluids, and all other such items necessary to maintain the work area and equipment in a clean, safe, and orderly condition.

### 3.2.9 Waste Containers

The Subcontractor shall provide clean storage containers with lids to contain drill cuttings. Hazardous waste labels, to be affixed to the side of the container, shall also be furnished by the Subcontractor.

The Subcontractor shall provide a tank truck or storage tank (1,500 gallon minimum capacity) to temporarily store wastewater collected during drilling and sampling and pumped water collected during well development.

**3.2.10 Christie Box and Concrete**

The Subcontractor shall provide one 8-inch inside diameter traffic rated Christie box that is complete with bolts, cover, and locking mechanism. The Subcontractor shall provide Type 1 Portland cement as defined by ASTM C150. The Subcontractor shall also provide a concrete backfill mix that achieves a minimum 28-day compressive strength of 3,000 pounds per square inch.

**3.2.11 Portable Toilet Facility**

The Subcontractor shall provide portable toilet facilities adequate for the subcontractors crew and the Engineer for the duration of the project.

**3.3 EQUIPMENT AND MATERIALS FURNISHED BY OTHERS**

Central onsite storage tank to store all water collected from drilling, sampling, decontamination operations, and well development.

## **General Conduct of Work**

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### **4.1 WATER SUPPLY**

The Subcontractor shall locate and use an appropriate clean water source. The Subcontractor shall provide all equipment necessary to transport the water to the jobsite. The equipment required may include pumps, water trucks or trailers, hoses, storage tanks, and all other items necessary for an adequate water supply. All discharge of supply water shall be handled in an environmentally acceptable manner and controlled to prevent erosion or any other damage.

### **4.2 CONTROL OF DRILL CUTTINGS AND FLUIDS**

The Subcontractor shall supply equipment and materials to capture, contain, transport, and transfer all drill cuttings and fluids ejected from the borings to containers. Soil cutting and fluid disposal shall be under the direction of the engineer (see Sections 3.1.1 and 3.2.9).

Petroleum-based grease or pipe dope shall not be used. Environmentally acceptable grease, such as vegetable oils, "King's Stuff" (Teflon™-based), or an equivalent, shall be allowed.

### **4.3 DECONTAMINATION OF EQUIPMENT AND TOOLS**

The Subcontractor shall decontaminate all equipment that comes into contact with potentially contaminated soil and groundwater prior to and after each use. The Subcontractor shall provide the necessary equipment and materials to construct a bermed and lined decontamination pad on site that can accommodate the decontamination of all soil sampling equipment and tools and all groundwater purging and sampling equipment. The location of the decontamination pad shall be designated by the engineer at the start of the field work. All decontamination activities shall be performed by the Subcontractor within the decontamination pad. Clean equipment shall be stored on clean plastic sheeting in clean areas. Materials to be stored more than a few hours shall also be covered. All soils and fluids generated from equipment decontamination, with the exception of hexane, shall be collected and contained in temporary storage tanks or a water truck (Section 3.2.9). It shall then be transported to the onsite treatment plant, to the maximum extent possible. Hexane shall be allowed to evaporate from a decontamination bucket.

#### **4.3.1 Soil and Groundwater Sampling Equipment**

Prior to the start of each day of sampling, the Subcontractor shall set up a decontamination pad. Five 5-gallon plastic tubs, a metal Hudson sprayer, a metal bucket, a roll of plastic sheeting, and two brushes shall be needed to decontaminate equipment. A 10-foot by 10-foot section of plastic sheeting large enough to contain any spilled or splashed liquid shall be placed on the ground by the Subcontractor. The Subcontractor shall fill the five 5-gallon plastic tubs with the following liquids: Tub 1 with tap water and a small amount of non-phosphate detergent; Tub 2 with tap

water; Tubs 3 and 4 with deionized/distilled water; and Tub 5 with organic-free water (HPLC grade). Tubs 1 and 2 shall have tub-dedicated brushes. The Subcontractor shall place hexane in a metal Hubson sprayer.

The following, to be carried out by the Subcontractor in sequence, is an EPA Region IX recommended procedure for the decontamination of soil and groundwater sampling equipment such as brass tubes, Maxi-Simulprobe samplers and Teflon bailers:

1. Equipment shall be submersed in Tub 1 and scrubbed with a brush. The piece of equipment shall then be moved to Tub 2. If the contents of the tub becomes dirty, the tub shall be emptied and fresh tap water and detergent shall be added.
2. Equipment that has been washed with detergent shall be submersed in Tub 2 and scrubbed with a brush. The piece of equipment shall then be moved to Tub 3. If the contents of the tub becomes dirty, the tub shall be emptied and fresh tap water shall be added.
3. Equipment shall be submersed in Tub 3. The piece of equipment shall then be placed over a metal bucket. If the contents of the tub becomes dirty, the tub shall be emptied and fresh deionized/distilled water shall be added.
4. Equipment shall be sprayed with a small amount of hexane over the metal bucket. Plastic, rubber, or Teflon equipment does not need to be sprayed with hexane. The hexane on the equipment shall be allowed to volatilize. The piece of equipment shall then be moved to Tub 4.
5. Equipment shall be submersed twice in Tub 4. The piece of equipment shall then be moved to Tub 5.
6. Equipment shall be submersed in Tub 5. The water in Tubs 4 and 5 shall be changed daily.

#### 4.3.2 Drill Rig and Boring Equipment

All boring equipment (i.e., augers,, etc.) shall be steam cleaned or decontaminated according to the following procedure, to be carried out in sequence:

1. Non-phosphate detergent wash, including scrubbing the equipment with a tub-dedicated brush
2. Tap water rinse, twice

The exterior surfaces of drill rigs and any large equipment shall be thoroughly steam-cleaned with potable water. The equipment shall be clean of all debris and contaminated fluids (such as obvious leaks from hydraulic lines, couplings, and fittings) to avoid contamination of onsite soils and soil borings. At the end of each work day and/or after the completion of the work, the Subcontractor shall completely decontaminate his drill rig and soil sampling equipment to the satisfaction of the engineer before leaving the site. Accessible interior portions of augers, pipes, hoist rods, cables, bits, and miscellaneous boring equipment shall be decontaminated at the start of the job and between borings.

#### 4.3.3 Groundwater Development and Purging Equipment

The following decontamination procedure must be carried out in sequence for all groundwater development and purging equipment:

1. Non-phosphate detergent wash, including scrubbing the outside of the hose and running soapy water through the lines for a minimum of 5 minutes. The soapy water may be recirculated in the tub.
2. Tap water rinse, minimum 3 minute recirculating. Rinse outside of hose.
3. Tap water rinse, non-recirculating, 3 minutes. Fresh tap water should be pumped through hose for 3 minutes.

The exterior surfaces and accessible interior portions of submersible and hand pumps, pipes, hoist rods, cables, Teflon bailers, tubing, and cord shall be cleaned at the start of the job and between wells.

Inaccessible interior portions of the pumps shall also be cleaned prior to each use as described above.

#### 4.4 WORK AREA PROTECTION AND CLEANING

The work areas shall be kept in a neat and orderly condition at all times. During demobilization, the Subcontractor shall remove his equipment and all surplus and unused material, and shall restore the area to its original condition.

#### 4.5 RECORDS AND REPORTS

The Subcontractor shall prepare a daily report of pay item expenditures covered under the Schedule of Quantities and Prices, including dates, times of beginning and completing work, location and number of borings as applicable, and other pertinent data, as requested by the engineer. At the end of each work day, the Subcontractor shall deliver a daily report to the

engineer for approval. If the engineer approves the report, he shall sign it. If the engineer does not approve the report, he shall substantiate the nonapproval in writing. Invoiced items not shown on a daily report shall be disallowed.

During the drilling operations, the Subcontractor shall keep an accurate driller's log of the top and bottom depth of, and description of, each stratum penetrated and each sample obtained during drilling. The depth to groundwater shall be measured at the start and end of each shift, and any unusual groundwater conditions during drilling or sampling shall be noted. The Subcontractor shall provide copies of the driller's log to the engineer at the completion of the boring.

#### 4.6 PERSONNEL AND SUPERVISION

The Subcontractor shall provide an adequate number of qualified personnel to carry out the required tasks in a safe, efficient, and expeditious manner. A designated competent supervisor or foreman shall supervise the work process and serve as liaison between the engineer and the Subcontractor personnel. Equipment operators shall be competent, experienced, and fully capable of performing to specifications.

## Exploratory Drilling

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### 5.1 DRILLING METHOD

The drilling method shall advance the boring using hollow-stem flight augers. The requirement for drilling equipment is given in Section 3.1.1.

The Subcontractor shall exercise careful control of the drilling. Drill cuttings shall be collected from the boring by the Subcontractor at every 1-foot increment of drilling advance and shall be placed sequentially on a plastic sheet for inspection. The Subcontractor shall provide plastic sheeting as described in Section 3.2.8.

All drilling cuttings shall be placed in containers and purged groundwater shall be contained in temporary storage tanks or a water truck. (Section 3.2.9). All drilling water and groundwater shall be contained and transported to an onsite storage tank identified by the engineer. Each boring shall be securely covered when left unattended, and no borings will be left open overnight.

### 5.2 GROUNDWATER OBSERVATION

The Subcontractor shall report the groundwater conditions observed during drilling and assist the engineer by measuring water levels in each permeable water-bearing zone using a water-level meter (see Section 3.1.4).

Water levels shall be measured in borings during drilling immediately before and following each groundwater sampling. The water level recovery after sampling shall be measured until a near steady-state water level is obtained. The water level shall be assumed to have reached a near steady-state condition if no significant water level changes occur in 10 minutes. The length of observation shall be limited to a maximum of 20 minutes if the water level cannot stabilize within this time, or as directed by the engineer.

### 5.3 GROUNDWATER AND SOIL SAMPLING USING A MAXI-SIMULPROBE

Approximately two groundwater samples shall be collected during drilling of each of the first six borings. The water-bearing zones to be sampled shall range in depth from 6 to 80 feet. The sample locations shall be specified by the engineer based when drill cuttings reveal coarse-grained soils (sand, gravel, or a mix of sand and gravel)

The driller shall indicate to the engineer when this condition is observed. Drilling advance shall be stopped whenever a permeable water-bearing zone is identified and the depth to groundwater measured before insertion of the Maxi-Simulprobe sampler.

The wire-line down-hole hammer, modular water canister, and ambient hydrostatic pressure soil/groundwater sampling mode of the Maxi-Simulprobe, as shown in Figure 3, shall be used to collect the water and soil samples through the hollow-stem auger. A Maxi-Simulprobe sampler shall be driven into the undisturbed soil several feet below the bottom of the boring, as directed by the engineer, using a down-hole hammer consistent with the manufacturer's Standard Operating Procedures (Attachment A). The driller shall closely monitor and report to the engineer any change in resistance during the driving process. When the desired depth is reached, the body of the tool shall then be pulled back 4 to 6 inches so groundwater can flow into the open screen of the Maxi-Simulprobe, past the lower intake valve, and into the sample chamber. Discharge of nitrogen through the Teflon line shall be monitored to insure an adequate amount of water has been collected.

If significant reduction in resistance is observed during driving, or for other causes, the engineer may determine that a nonwater-bearing zone has been encountered and abort the sampling attempt. In some cases, the engineer may direct that Maxi-Simulprobe be driven 1 to 2 feet deeper than normal to determine whether a sand or gravel water-bearing zone exists below the low-resistance nonwater-bearing layer.

The Subcontractor shall provide all parts and accessories required for groundwater and soil sampling using a Maxi-Simulprobe.

Immediately prior to sampling and at the completion of sampling, the groundwater level in the boring shall be measured following the procedure specified in Section 5.2.

#### 5.4 BACKFILLING OF EXPLORATORY BORINGS

Upon completion of auger drilling, each exploratory boring that is not to be completed as a groundwater monitoring well shall be filled with a cement/bentonite grout to the top of the boring, unless otherwise required by the engineer. The grout shall be mixed in the following proportions: 7.5 gallons of water and 2.5 pounds of powdered bentonite per sack of Type II Portland cement. The bentonite shall be thoroughly mixed with the water prior to adding the cement. The grout shall be pumped through the hollow-stem augers to the bottom of the boring. The augers shall be concurrently extracted as the grout is placed to ensure a good seal. Should loss or shrinkage of grout occur, the borings shall be refilled until they remain full.

## **Groundwater Monitoring Wells**

---

The monitoring well designs for this job are shown in Figures 4 and 5. All lengths and dimensions are approximate and may vary in accordance with site conditions as specified by the engineer.

Depths of the monitoring wells are expected to range from approximately 30 to 80 feet below the ground surface. Monitoring wells may be added, eliminated, or relocated, as directed by the engineer.

### **6.1 INSTALLING SCREEN AND RISER CASING**

The final depth of the boring shall be measured by the Subcontractor with a weighted tape to  $\pm$  0.1 foot. The sump and end cap shall be placed on the bottom of the screen. The screen and riser casing shall then be lowered into the boring through the hollow-stem auger and suspended at the depth specified by the engineer. Any portion of the riser casing extending above ground surface, including the cutoff, shall be measured and reported to the engineer.

### **6.2 INSTALLING FILTER PACK**

As soon as the riser casing and screen are in place, clean water shall be pumped into the riser casing so that the return flow shall rise to the ground surface through the annular space between hollow-stem auger and the casing.

The sand for the filter pack shall be tremied into the annular space between the riser casing and hollow-stem auger as the velocity of the return flow is adjusted to allow the sand to sink. At the same time, the augers shall be slowly withdrawn from the boring. The removal of the augers from the borehole shall continue until all the filter pack is in place. The filter pack shall extend from the bottom of the hole to at least 2 feet, but not more than 3 feet, above the top of the screen. The depth to the top of the filter pack in the boring below the augers shall be periodically measured by the Subcontractor during placement, and the final depth after installation shall be reported to the engineer.

### **6.3 INSTALLING SEAL AND BACKFILL**

After the filter pack is placed, 3/8-inch bentonite pellets shall be introduced between the riser casing and the walls of the hollow-stem auger as the auger is withdrawn. After about 10 minutes has elapsed, which will give sufficient time for the bentonite to reach the top of the filter pack, water shall be added to the boring to hydrate the bentonite pellets, if the seal is above the water table. After the seal has had about 1 hour to hydrate, the depth to the seal shall be measured. The minimum thickness of this bentonite seal shall be 2 feet. If problems occur with the bentonite, fine sand may be used in its place, as approved by the engineer. Following

emplacement of the bentonite seal, the annular space between the riser casing and the walls of the hole shall be filled with a cement/bentonite grout to the top of the hole. The flight augers shall be slowly removed from the boring in tandem with placement of the grout backfill through the hollow-stem augers.

#### 6.4 SURFACE COMPLETION

The monitoring well surface completion to be constructed by the Subcontractor shall consist of approved protective casing extending from at least 3 feet below grade to 2 feet above the ground surface as shown in Figure 4 or a flush mounted surface completion as shown in Figure 5 (see Section 3.2.7). The 8-inch-diameter protective steel casing shall be centered over the 4-inch diameter PVC well casing and inserted into the grouted annulus prior to grout hardening. No sooner than 24 hours after grouting, a reinforced concrete surface well pad of nominal dimensions 3 ft. x 3 feet x 4 inch squared shall be constructed around the protective casing of each well. The well pad shall slope slightly away from the boring to prevent ponding of water around the well. A seep hole shall be drilled in the protective casing, 1 or 2 inches above the well pad. A steel lockable cap shall be provided by the Subcontractor for the protective casing or the riser pipe. The protective casing shall be painted with a rust-preventive, yellow-colored paint.

#### 6.5 WELL DEVELOPMENT

After monitoring well installation, each well shall be developed by the Subcontractor. Well development shall not be initiated sooner than 24 hours and not later than 48 hours following the completion of grouting, unless otherwise directed by the engineer. Development shall proceed by bailing, surging, pumping, and/or air-lifting. The Subcontractor shall provide all supervision, personnel, and equipment (including a development rig, bailers, swabs, air-lifting pipe and compressor and/or pumps and generators, and other accessories) necessary for well development.

Development shall continue until the well produces relatively clear water, as judged by the engineer and the water temperature, pH, and conductivity have stabilized, as indicated by three consecutive measurements within 10 percent of each other, or a minimum of 10 well volumes have been removed. Water characteristics shall be monitored by the engineer. If wells are bailed dry, they shall be allowed to recharge to 80 percent of the static water level and then bailed again. The wells shall be developed until they are bailed dry 10 times to be considered complete.

Water removed from the wells during the initial stage of development shall be contained in a water truck or a temporary storage tank provided by the Subcontractor as described in Section 3.2.9. The water shall be transported to the central, onsite treatment system provided by the engineer as described in Section 5.1.

## 6.6 ACCEPTANCE CRITERIA

All monitoring wells shall be inspected by the engineer at the completion of the well installation. The engineer shall not accept the completed well if any one of the following conditions is observed: (1) broken sump/end cap, screen or riser casing, (2) the presence of grout, filter pack, or foreign material in the well casing, (3) failure to pass the alignment test, and (4) any other evidence that indicates unacceptable well construction.

The alignment of the well shall be unacceptable if a straight, 10-foot length of minimum 2-inch O.D. (for 4-inch-diameter wells) PVC pipe cannot be passed freely down the length of riser casing to the top of the screen. This test shall be performed by the Subcontractor after completion of well development and shall be monitored by the engineer.

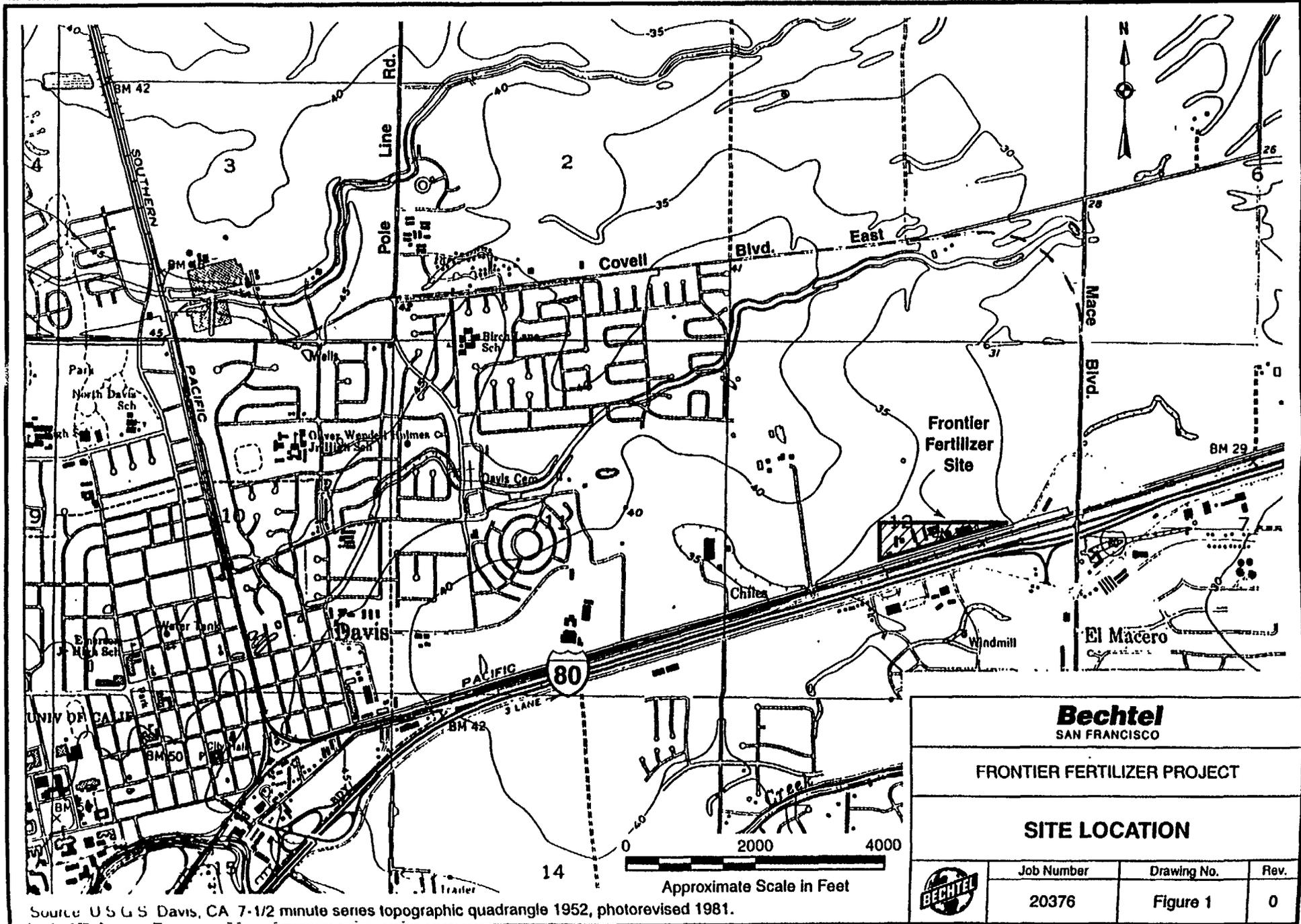
## 6.7 GROUNDWATER SAMPLING DURING WELL DEVELOPMENT

Groundwater samples shall be obtained from each new monitoring well immediately after completion of well development, as directed by the engineer. The Subcontractor shall provide labor and equipment to assist the engineer in sampling. Samples shall be collected by the Subcontractor using a Teflon bailer.

## 6.8 WELL ABANDONMENT

The Subcontractor, at his cost, shall provide all labor, equipment and materials for the abandonment of monitoring wells that fail to meet the acceptance criteria and cannot be readily repaired as determined by the engineer (see Section 6.6). Abandonment shall be conducted according to the standards presented in California Department of Water Resources (DWR) Bulletins 74-81 and 74-90.

The monitoring well construction materials, including the protective casing, riser casing, screen, filter pack, sump, and grout, shall be removed. The well boring shall then be tremie-grouted with the grout mixed in the following proportions: 7.5 gallons of water and 2.5 pounds of bentonite per sack of Type II cement. The use of a Tremie pipe shall be required to ensure proper abandonment.



**Bechtel**  
SAN FRANCISCO

FRONTIER FERTILIZER PROJECT

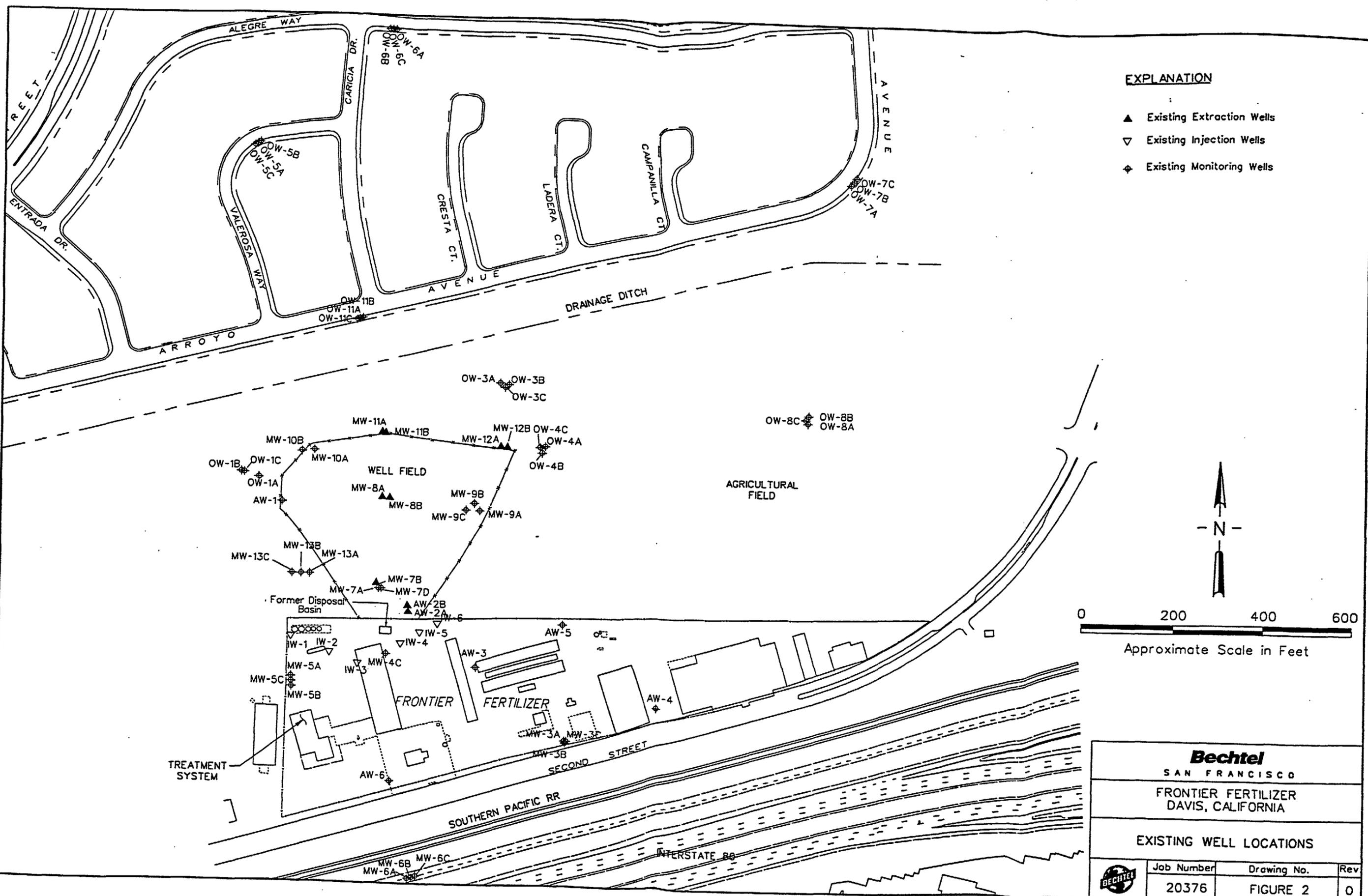
**SITE LOCATION**

| Job Number | Drawing No. | Rev. |
|------------|-------------|------|
| 20376      | Figure 1    | 0    |

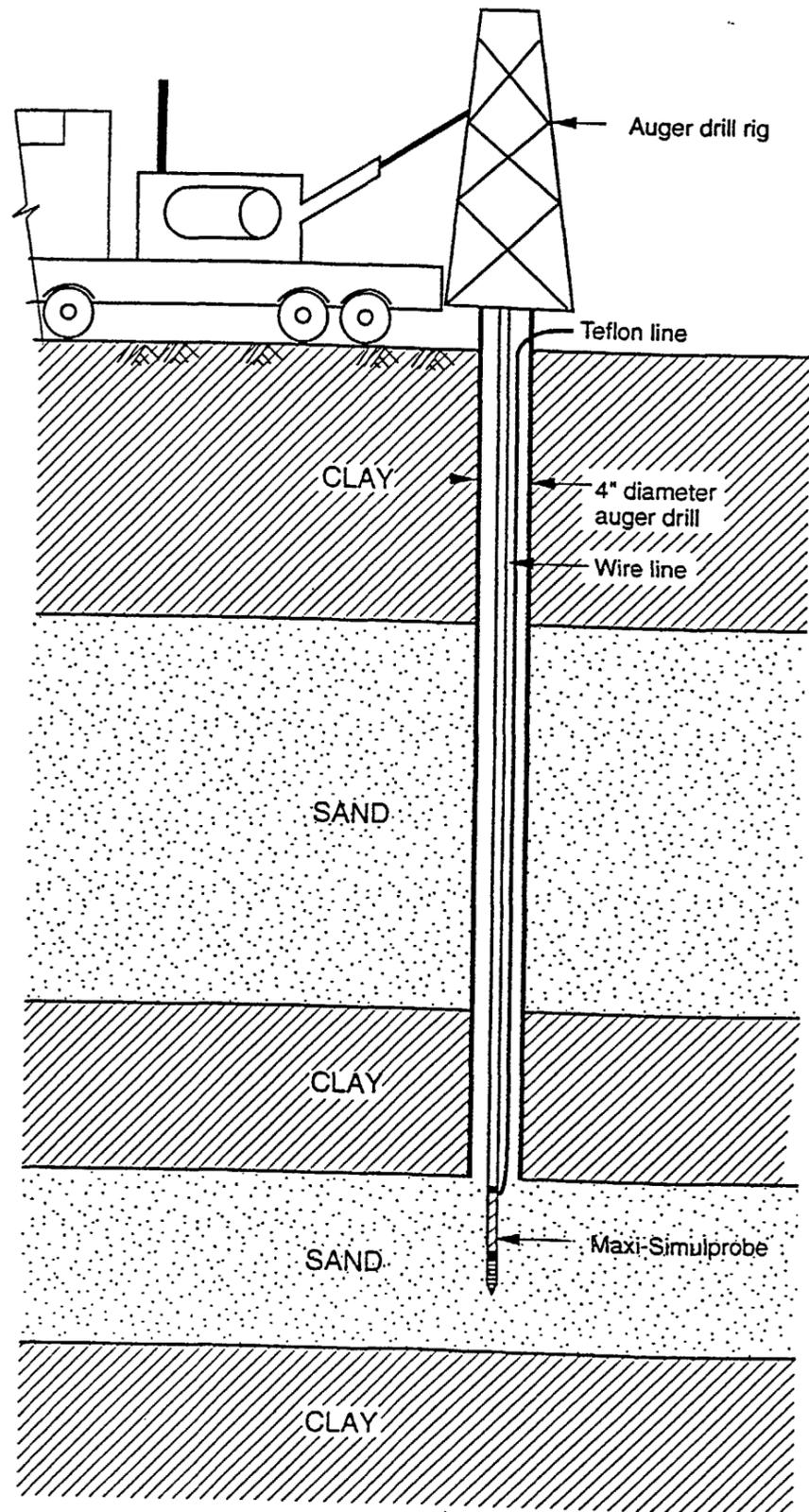
Source: U.S.G.S. Davis, CA 7-1/2 minute series topographic quadrangle 1952, photorevised 1981.

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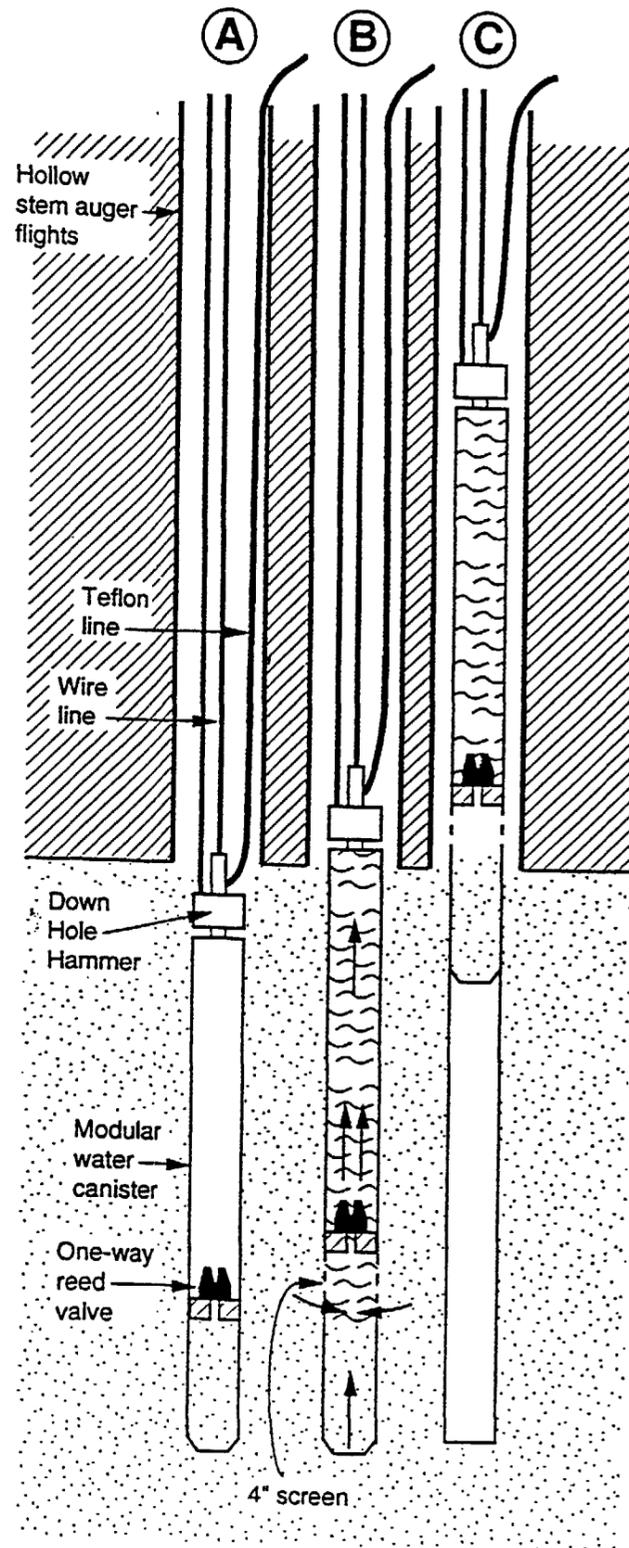
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|--|------------|-------------|-----|
| <b>Bechtel</b>                           |            |             |     |
| SAN FRANCISCO                            |            |             |     |
| FRONTIER FERTILIZER<br>DAVIS, CALIFORNIA |            |             |     |
| EXISTING WELL LOCATIONS                  |            |             |     |
|  | Job Number | Drawing No. | Rev |
|  | 20376      | FIGURE 2    | 0   |



a) Typical Drilling/Sampling Arrangement



b) Water Sample Collection

**EXPLANATION**

- (A) Maxi-Simulprobe closed while being driven into position.
- (B) Maxi-Simulprobe tool open to collect sample.
- (C) Tool withdrawn from hole.

**NOTE:**

Drawing is not to scale.

|   |                     |                         |
|---|---------------------|-------------------------|
| <b>Bechtel</b><br>SAN FRANCISCO   |                     |                         |
| FRONTIER FERTILIZER PROJECT   |                     |                         |
| <b>MAXI-SIMULPROBE GROUNDWATER SAMPLING ILLUSTRATION</b>                              |                     |                         |
|  | Job Number<br>20376 | Drawing No.<br>FIGURE 3 |
|   |                     | Rev.<br>0               |

U. S. ENVIRONMENTAL PROTECTION AGENCY REGION 9  
Environmental Services Branch  
75 Hawthorne Street  
San Francisco, CA 94105  
Phone: 415/744-1498

SITE NAME: *Frontier Fertilizer*  
CASE/RAP NO.:

REGIONAL ANALYTICAL PROGRAM CLIENT REQUEST FORM

The analysis of water samples for 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) by EPA Method 504, Revision 2.0 (1989).

1. Definition and number of work units involved (specify whether whole samples or fraction; specify sample matrices; and specify concentration level):

*30 low level groundwater samples on a quick turn around time basis*

2. Estimated date(s) of collection (provide a sampling schedule):

*June 10 to June 26, 1998*

3. Estimated date(s) and method of shipment:

Overnight courier - samples are to be shipped on the day of collection for next day delivery including Saturday deliveries. Laboratory must be capable of accepting Saturday deliveries.

4. Number of days analysis and data required after laboratory receipt of samples:

a. The contract required holding time for extraction and analysis is twenty-six (26) days from the date of sample receipt by the laboratory.

b. The method holding time for extraction and analysis is 28 days from the date of sample collection.

c. Data packages and all other deliverables are required within 35 days from receipt of last sample in each sample delivery group (SDG). A SDG is defined as the following, whichever is most frequent:

- Each case of field samples received; or
- Each 20 field samples within a case; or
- Each 14 calendar day period during which field samples in a case are received.

5. Analytical protocol required (attach copy if other than a protocol currently used in this program):

a. Requirements for the gas chromatograph (GC) system and GC columns are provided in Section 6.7 of Method 504. Columns other than those recommended in Sections 6.7.3 through 6.7.5 are permissible if the laboratory demonstrates that the analyses meet all the

performance and QA/QC criteria specified in Method 504 and in this contract. The use of a linearized electron capture detector (ECD) is required.

- b. A five point initial calibration is required. Aqueous calibration standards are prepared (see Sections 9.1.1 through 9.1.3) and extracted (see Sections 11.1.3 and 11.2.1 through 11.2.6) in a manner identical to sample extraction. These standards are used for instrument calibration and sample quantitation to account for potential analyte losses during extraction.
  - c. Follow EPA Method 504 (Section 11) for extraction and analysis of samples and QC samples. Section 8 presents the required target analytes and contract required quantitation limits (CRQLs).
  - d. Confirmation on a second column with a liquid phase different from that of the primary column is required for all positive results. Refer to Section 5.a (above) for acceptable confirmation columns. Confirmation analyses must meet all instrument calibration criteria and blank acceptance criteria specified in Sections 6.a and 6.b.2, respectively, of this Client Request Form (CRF).
6. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

a. Calibration Procedure and Criteria:

Calibrate according to Section 8.0 of Method 504, with the following specifications:

- 1. Perform a five-point initial calibration containing both target analytes and the surrogate compound with a low standard at the concentrations of the CRQL or lower. Single point calibration (Method 504, Section 9.1.4) is not a permissible option.

- a) If the external standard method is used, either peak area or peak height may be used for (instrument) response in the calculations. Calculate the calibration factor (CF) for each analyte in each calibration standard following Section 9.1.3 of EPA Method 504.

$$CF = x \div y$$

where CF = calibration factor [in  $\mu\text{g/L}$ ]  
x = concentration of the standard [in  $\mu\text{g/L}$ ]  
y = response, which may be peak area or height [unitless]

Calculate the mean and standard deviation ( $\sigma_{n-1}$ ) of the CFs in the five calibration standards. Calculate the percent relative standard deviation (%RSD) by dividing the standard deviation by the mean. The %RSD for both analytes and the surrogate compound must not exceed 20.

- b) Alternatively, linear regression that does not force the resulting straight line to pass through the origin may be used for instrument calibration. For each analyte, perform a linear regression on the response versus the concentration of the standards. The linear

regression analysis will produce the slope and y-intercept values for a linear equation in the following form:

$$y = ax + b$$

where:

y = response, which may be peak area or height  
x = concentration of the standard [in  $\mu\text{g/L}$ ]  
a = slope of the straight line  
b = the y-intercept

The correlation coefficient (r) generated by the linear regression must be greater than or equal to 0.99 for each target and surrogate compound. If calibration curves are used, provide sufficient documentation so that all analytical results can be recalculated, as specified in Section 7.a.4 of this CRF. Provide the equation for the line generated by the data system.

- c) The low concentration standard must have a signal-to-noise ratio of 5:1 or greater for both target analytes. If this requirement cannot be met, the laboratory must submit a method detection limit (MDL) study as part of the data package, in order to validate its ability to achieve the CRQLs. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.
2. Determine a retention time (RT) window for each target and surrogate compound. Calculate the mean and standard deviation ( $\sigma_{n-1}$ ) of the 5 retention times for each compound over the 5 concentration levels in the initial calibration. Calculate the window as the mean RT  $\pm$  3 times the standard deviation.
  3. Demonstrate that the individual analytes in the mid-point calibration standard are resolved by the chromatographic system. The analytes must be  $\geq 90\%$  resolved.
  4. Extract and analyze a continuing calibration at the mid-point concentration for each analyte at the beginning of each day, after every 10 injections, and at the end of the analytical sequence. This standard is to be used to verify instrument performance. Percent differences between the nominal amount and calculated amount for each analyte in the continuing calibrations must be within  $\pm 15\%$  for both EDB and DBCP.
  5. Calculate the daily RT windows as the continuing calibration RT  $\pm$  3 times the standard deviation as determined in Section 6.a.2 of this CRF. Use these daily RT windows for target analyte identification.
- b. Internal Quality Control Checks, Control Limits and Corrective Actions:
1. When calibration standard measurements exceed the quality control (QC) requirements for the initial calibration or the

continuing calibration, terminate analysis, correct the problem, and recalibrate the instrument.

The continuing calibration standard reflects the conditions under which the analysis of samples was performed. Associated samples are considered to be the samples analyzed following the continuing calibration up to the subsequent acceptable continuing calibration standard. Reanalyze all samples associated with an out-of-control continuing calibration standard.

2. Extract and analyze laboratory reagent blanks (LRBs) at a frequency of one per SDG or one for each extraction batch, whichever is greater. The method blanks must contain less than or equal to the CRQL of the target analytes listed in Section 8.

If a LRB exceeds these criteria, the laboratory must consider the analytical system to be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. All samples associated with a LRB that is out of control must be re-extracted and reanalyzed at no additional cost to the Region.

3. Spike all standards, samples, LRBs, and QC samples with an appropriate surrogate (i.e., 4-bromofluorobenzene). The amount of surrogate added must be at least 10 times the concentration of the lowest standard. Recoveries of 65-125% are required, unless documentation (such as control charts) is available to support a different range of recoveries. The laboratory must submit, as part of the data package, all supporting documentation for surrogate recoveries, and historical surrogate recovery data if necessary.

If surrogate control limits are exceeded, take appropriate actions to identify the problem by re-analyzing the affected samples. If reanalysis does not solve the problem, the affected sample must be re-extracted and re-analyzed. If re-extraction and re-analysis solves the problem, then the problem was within the laboratory's control. Therefore, submit only data from the analyses with surrogate recoveries within QC limits. If re-extraction and re-analysis of the sample does not solve the problem, then submit the surrogate recovery data and sample analysis data from the initial analysis of both sample extracts.

4. Dilute and reanalyze sample extracts containing one or more analytes at concentrations above the initial calibration range. If dilution is necessary, the dilution must be selected so that the highest concentration analyte is determined at a concentration in the upper half of the calibration range. Report the results and submit documentation for both the diluted and undiluted analyses.
5. Analyze laboratory fortified matrix and laboratory fortified matrix duplicate (LFM/LFMD) at the frequency of one per SDG. Both EDB and DBCP must be spiked into the LFM/LFMD analyses. The concentration of the matrix spike solution should be such that the final extracts contain each analyte at an amount at the mid-range of the calibration curve.

Recoveries of 75-115% and relative percent differences (RPDs) of  $\leq 15\%$  are required. Re-extraction and reanalysis are not required if the LFM/LFMD criteria are exceeded. However, document LFM/LFMD outliers in the SDG narrative.

6. Extract and analyze a laboratory fortified blank (LFB) for each SDG or each batch of samples prepared, whichever is more frequent. The final extracts should contain EDB and DBCP at a concentration of  $0.25 \mu\text{g/L}$  each. Percent recovery limits of 60-140% are required. If the QC limits are exceeded, re-extract and reanalyze the LCS and all samples associated with the noncompliant LCS.
  7. Perform weekly MDL checks according to Section 10.4 of Method 504. Recovery must be 60-140% for each analyte. Document outliers in the SDG narrative.
  8. Analyze a QC sample from an external source quarterly according to Section 10.5 of Method 504. Document outliers in the SDG narrative.
  9. The QC requirements listed above are the minimum required. It is impossible to address all analytical situations that might be experienced by a laboratory during the analysis of environmental samples. The laboratory is expected to adhere to good laboratory practices when analyzing samples. If the laboratory has questions concerning the analyses of samples not addressed in this document, the Region must be notified IMMEDIATELY. The Laboratory Manager, or designee, must address problems and their solutions in the SDG narrative.
7. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.

a. Data Calculations and Reporting Units:

1. Quantitation of target and surrogate compounds in field and QC samples are performed as follows.
  - a) To calculate the concentration of an analyte or surrogate using the external standard method, use the average CF for the compound from the initial calibration (see Section 6.a.1.a) in the equation:

$$\text{Concentration} = (y \times \text{average CF} \times \text{DF} \times 35 \text{ mL}) \div V_s$$

[ $\mu\text{g/L}$ ]

where  $y$  = response, which may be peak area or height  
CF = calibration factor [ $\mu\text{g/L}$ ]  
DF = dilution factor  
 $V_s$  = volume of the sample in mL

- b) If using linear regression for data calculations, then use the following equation:

$$x = (y - b) \div a$$

$x$  = concentration of the detected analyte [ $\mu\text{g/L}$ ]  
 $y$  = response, which may be peak area or height  
 $b$  = the  $y$ -intercept

a = slope of the line

2. The sample results are to be reported in the concentration units of micrograms per liter ( $\mu\text{g/L}$ ) for water samples. Report all results to two significant figures.
3. For rounding results, adhere to the following rules:
  - a) If the digit following those to be retained is less than 5, round down;
  - b) If the digit following those to be retained is greater than 5, round up; or
  - c) If the digit following the last digit to be retained is equal to 5, round down if the digit is even, and round up if the digit is odd.
4. All records of analysis, dilutions and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example of the calculations in the data package.

b. Documentation and Deliverables:

Deliverables (in the form of a purge file, i.e., original documents) for each SDG shall include the following items:

1. All original shipping documents and sample tracking reports, including signed chain-of-custody forms, airbills, and traffic reports.
2. A completed and signed document inventory on a modified Organics Complete SDG File (CSF) Inventory Sheet (CLP Form DC-2).
3. All original sample receipt documents, including sample log-in information on a modified CLP Form DC-1, an SDG cover sheet, and any other sample receipt records.
4. A copy of the CRF, as provided by the Region (so that any additions or revisions authorized by the Region will be known). Only the technical portion of the CRF is required.
5. Any telephone logs referring to the samples.
6. An SDG Narrative, signed by the laboratory manager or designee, certifying the accuracy and validity of all data reported. The SDG Narrative must contain: laboratory name; case/RAP number; SDG number; EPA sample numbers in the SDG, differentiating between initial analyses and reanalyses; the corresponding laboratory sample identification (ID) numbers; and contract number. The SDG narrative must provide a description of all GC columns used for analysis, including brand name, the internal diameter in millimeters (mm), the length in meters, coating material, and film thickness. The SDG narrative must describe any administrative or technical problems encountered such as QC, sample shipment, or analytical problems and the resolution of these problems. The SDG narrative must include an explanation for any manual integrations or manual edits. The SDG narrative must include a formula (including definitions) showing how the results were calculated, and an example of an actual calculation for a sample in the SDG.

7. Include the following information in the header for each data reporting form: laboratory name, contract number, laboratory code, case/RAP number, and SDG number.
8. Tabulated analysis results for all field and QC samples on a modified CLP Form 1. Include both compound name and Chemical Abstracts Service (CAS) registry number for both analytes. Clearly specify concentration units and laboratory data qualifiers. Include the following additional information in the header: EPA sample number, laboratory sample ID, matrix, laboratory file ID, instrument ID, validated time of sample receipt (VTSR), extraction method, sample volume in milliliters (mLs), concentrated extract volume and injection volume in microliters ( $\mu$ Ls), and dilution factors.
9. Raw sample data, including:
  - a) All data system printouts, including: RT and corresponding peak area or height for each peak detected. The printouts must be labeled with the EPA sample number. When manual integrations or edits are made, analyst must sign and date each edit and include the integration time range.
  - b) All chromatograms, including: EPA sample number; volume injected ( $\mu$ L); date and time of injection; GC instrument ID; GC column identifier including stationary phase and internal diameter; and scaling factor.
  - c) Any manual worksheets
10. Surrogate result summaries on a modified CLP Form 2 with QC acceptance criteria and calculated percent recovery (%R) values rounded to the nearest whole number. If dual columns are used, surrogate recoveries should be reported from each column. Identify on the form header the GC column ID. Flag all recoveries outside the QC limits with an asterisk.
11. LFM/LFMD and LFB result summaries on a modified CLP Form 3 with calculated percent recovery (%R) and RPD values. Include in the form the recovery and RPD QC limits for each spike compound. Tabulate also the concentration of the spike in the sample, the sample concentration, and the LFM and LFMD or the LFB concentrations on the form. If dual columns are used, report the lower result from the two columns for each target compound. Flag all percent recoveries and RPDs exceeding the QC limits. Include the EPA sample number in the header. Also provide on a modified Form 3, the results from the weekly MDL analyses and the quarterly QC sample. It is not necessary to provide the raw data for analyses of MDLs and the quarterly QC sample.
12. Method blank summary on a modified CLP Form 4 specifying which samples are associated with each blank. Include on the form header the blank EPA sample number, the laboratory sample ID, the laboratory file ID, matrix, method blank extraction date, method blank analysis date and time, the instrument ID, and the GC column ID. The EPA sample number, the laboratory sample ID, and date analyzed must be included for each sample associated with the referenced blank. If

dual columns are used, report the items listed above for each column.

13. Instrument calibration data for each GC instrument and GC column used for analysis.

a) Initial Calibration

- 1) Standards summaries for initial calibration on modified CLP Form 6 tabulating: CFs, mean CFs, %RSD values, RTs, mean RTs, and RT windows for each compound. Include the following additional information in the header: GC instrument ID, column ID, concentration levels for each standard, and analysis date.
- 2) Resolution of the mid-point standards for each set analyzed in the initial calibration on a modified Form 6I with analyte names, retention time, and percent resolution. Include the following additional information in the header: GC instrument ID, column ID, standard EPA sample number, laboratory sample ID, and date and time analyzed.
- 3) All data system printouts, including: RT and corresponding peak area or height for each peak detected. Label printouts with standard EPA sample number and the total nanograms injected for each analyte. When manual integrations or edits are made, analyst must initial and date each edit and include the integration time range. Chromatograms must be labeled with the following: standard EPA sample number, volume injected ( $\mu\text{L}$ ), date and time of injection, GC instrument ID, GC column ID, and scaling factor.
- 4) If linear regression is used for quantitation, include for each analyte the equation of the straight line, correlation coefficient, and graph of the straight line on axes labeled to identify what is plotted.

b) Continuing Calibration

- 1) Standard summaries for continuing calibration on modified CLP Form 7 tabulating: the nominal amount, calculated amount, the %D values, RTs, and daily RT windows for each compound. Include the following additional information in the header: dates of associated initial calibration, GC column ID, EPA sample number and laboratory sample ID for the continuing calibration standard(s), and date and time of continuing calibration standard(s).
- 2) All data system printouts, including: RT and corresponding peak area or height for each peak detected. Label printouts with the standard EPA sample number and the total nanograms injected for each analyte. When manual integrations or edits are made, analyst must initial and date each edit and include the integration time range. Chromatograms must be labeled with the

following: standard EPA sample number, volume injected ( $\mu\text{L}$ ), date and time of injection, GC instrument ID, GC column ID, and scaling factor.

14. Analytical sequence information on a modified CLP Form 8 for each GC instrument and GC column used for analysis. Tabulate the EPA sample number, the laboratory sample ID, the date and time of analysis, and surrogate RTs for all standards and field and QC samples associated with the initial calibration reported in the header. Include the following additional information in the header: instrument ID, GC column ID with internal diameter, and date of initial calibration.
15. Target analyte results on a modified CLP Form 10 for each field and QC sample that has one or more detected result(s). Include the names of the detected analytes, the RTs, the RT windows, the concentrations, and %D between the concentrations on the two columns. Include the following additional information in the header: laboratory sample ID, analysis date, instrument ID, and GC column ID.
16. Raw QC data must include the following items for both primary and confirmation analyses:
  - a) Blank data, in chronological order:
    - 1) Tabulated results on a modified CLP Form 1
    - 2) All data system printouts, including: RT and corresponding peak area or height for each peak detected. The printout must be labeled with the EPA sample number. When manual integrations or edits are made, analyst must initial and date each edit and include the integration time range. Chromatograms must be labeled with the following: EPA sample number, volume injected ( $\mu\text{L}$ ), date and time of injection, GC instrument ID, GC column ID, and scaling factor.
    - 3) Any manual worksheets
  - b) MS/MSD and LCS data, in chronological order:
    - 1) Tabulated results on a modified CLP Form 1
    - 2) All data system printouts, including: RT and corresponding peak area or height for each peak detected. The printout must be labeled with the EPA sample number. When manual integrations or edits are made, analyst must initial and date each edit and include the integration time range. Chromatograms must be labeled with the following: EPA sample number, volume injected ( $\mu\text{L}$ ), date and time of injection, GC instrument ID, GC column ID, and scaling factor.
    - 3) Any manual worksheets
17. All computer printouts resulting from sample screening, with integrated areas, peak heights, and calibration factors.

18. Bench sheets for sample preparation and analysis, including the following:
  - a) Extraction dates and times
  - b) Sample extraction or preparation method
  - c) Spiking solution identification with volumes and concentrations added
  - d) Instrument run logs listing instrument ID and time and date of analyses
19. Standards preparation logs, for all standard solutions used for either calibration or spiking, which include source, traceable lot number, and concentrations of all compounds.
20. Any internal laboratory sample or sample extract transfer records and tracking sheets.

8. Data Requirements

The required target compounds with corresponding contract required quantitation limits (CRQLs) are provided below.

| <u>Compound</u>             | <u>CAS Number</u> | <u>Water (<math>\mu\text{g/L}</math>)</u> |
|-----------------------------|-------------------|---|
| 1,2-Dibromoethane           | 106-93-4          | 0.05                                      |
| 1,2-Dibromo-3-chloropropane | 96-12-8           | 0.05                                      |

9. Other (use additional sheets or attach supplementary information, as needed):

If a copy of the "U.S. EPA Region 9 Laboratory QC Summary Report" form is attached, complete the form by following the directions on the first page of the form.

U. S. EPA REGION 9  
LABORATORY QC SUMMARY REPORT

LABORATORY: \_\_\_\_\_

SAS #:

SUBMITTED BY: \_\_\_\_\_

# SAMPLES:

TITLE: \_\_\_\_\_

MATRIX:

ANALYSIS: EDB and DBCP by EPA METHOD 504

DATE:

QC SUMMARY TABLE

| QC PARAMETER                | QC LIMITS  | FREQUENCY                                     |
|-----------------------------|--|---|
| Laboratory Blank            | <CRQL for all analytes                           | 1 per SDG (1/20)                              |
| Initial Calibration (%RSD)  | 5-pt Calibration Curve<br><20% for all analytes  | as needed                                     |
| Continuing Calibration (%D) | Midpoint Concentration<br>< 15% for all analytes | Daily   |
| MS/MSD (%R)                 | 75-115%  | 1 per SDG (1/20)                              |
| MS/MSD (RPD)                | 15%  | 1 per SDG (1/20)                              |
| Surrogates (%R)             | 65-125%  | all standards, blanks, samples and QC samples |

1. Were all samples analyzed within the contract required holding time of 28 days from sample receipt? YES          NO
- a. If no, list the samples that were analyzed outside of the holding time
- b. How many days outside of the holding time were these samples analyzed?
- 
2. Were all samples received intact and in good condition? YES          NO
- 
3. Was the data package sent within 35 days from the receipt of the last samples in the SDG? YES          NO
- a. If no, how many days late was the data package sent?

4. Was EPA Method 504 used to analyze these samples? YES NO
- a. If no, specify which method was used.
- b. If no, why was this method used and who authorized its use?
- c. Was the approved method followed without modifications or deviations? YES NO
- d. If no, specify what the modification or deviations were and who approved them.
5. Was a 5-point initial calibration curve run? YES NO
- a. If yes, when?
- b. If no, why not?
6. Did the initial calibration curve meet the QC requirement of <20% RSD for all analytes? YES NO
- a. If no, specify the analyte(s) and RSD(s) that were outside of the QC limits.
7. Was a continuing calibration run on each day samples were analyzed? YES NO
- a. If no, why not?
8. Did the continuing calibration standards meet the QC requirement of <15% RSD for all analytes? YES NO
- a. If no, specify the analyte(s) and RSD(s) that were outside of the QC limits.

9. Were laboratory blanks analyzed at a minimum frequency of 1 per SDG? YES NO
- a. If no, at what frequency were blanks performed.
10. Was blank contamination, if any, <CRQL for all analytes? YES NO
- a. If no, specify contaminant(s) and at what level(s) it was (they were) present.
11. Specify the surrogate(s) used.
12. Were surrogate recoveries within the QC requirement of 65-125%? YES NO
- a. If no, list the recoveries that were above this range.
- b. If no, list the recoveries that were below this range.
- c. How many non-compliant recoveries were caused by dilutions?
- d. How many non-compliant recoveries were caused by matrix interferences?
- e. Where samples re-analyzed to confirm matrix interference? YES NO
- If no, why not?
13. Were MS/MSD analyses performed at a minimum frequency of 1 per SDG? YES NO
- a. If no, why not?

14. Did the MS/MSD meet the percent recovery (%R) QC requirements of 75-115%? YES NO
- a. If no, specify the analyte(s) and %R(s) that were outside of the QC limits.
15. Did the MS/MSD meet the relative percent difference (RPD) QC requirement of <\_15%? YES NO
- a. If no, specify the analyte(s) and RPD(s) that were outside of the QC limits.
16. Was it possible to analyze all analytes within the initial calibration range? YES NO
- a. If no, were these samples diluted according to the instructions in Section 8.b.5 of the SAS CRF? YES NO
- b. List these samples and the associated analytes.
- c. If no, explain why not and list those samples.
17. Was second column confirmation performed on all samples with positive results? YES NO
- a. If no, why not?
18. Were the CRQLs met for all analytes? YES NO
- a. If no, why not?

U. S. ENVIRONMENTAL PROTECTION AGENCY REGION 9  
Environmental Services Branch  
75 Hawthorne Street  
San Francisco, CA 94105  
Phone: 415/744-1498

Site Name: *Frontier Fertilizer*  
Case/RAP No.:

REGIONAL ANALYTICAL PROGRAM CLIENT REQUEST FORM

*plus 1,2-dibromoethane and 1,2-dibromo-3-chloropropane*  
The analysis of low concentration water samples for the target compound list (TCL) volatile organic compounds (VOCs) following protocols outlined in the U.S. EPA Contract Laboratory Program (CLP) Statement of Work (SOW) for Organics Analysis (OLM03.1) and the Superfund Analytical Methods for Low Concentration Water for Organics Analysis (SAMLCO, 10/92 or more recent version). Where the procedures in this Client Request Form (CRF) differ from the above referenced methods, the procedures listed here are to take precedence.

A 25-mL purge volume is used for sample analysis to achieve low quantitation limits. The CRQLs listed in item 9, page 11 of this CRF must be met.

1. Definition and number of work units involved (specify whether whole samples or fraction; specify sample matrices; and specify concentration):

*30 low level groundwater samples on a quick turnaround time basis*

2. Estimated date(s) of collection (provide a sampling schedule):

*June 10 to June 26, 1998*

3. Estimated date(s) and method of shipment:

Overnight courier - samples are to be shipped on the day of collection for next day delivery including Saturday deliveries. Laboratory must be capable of accepting Saturday deliveries.

4. Number of days analysis and data required after laboratory receipt of samples:

- a. The contract required analysis holding time is ten (10) days from the date of sample receipt by the laboratory.
- b. The technical analysis holding time is fourteen (14) days from the date of sample collection for preserved samples and seven (7) days from the date of sample collection for unpreserved samples.
- c. Data packages and all other deliverables are required within 35 days from receipt of last sample in each sample delivery group (SDG). A SDG is defined as the following, whichever is most frequent:
- Each case of field samples received; or
  - Each 20 field samples within a case; or
  - Each 14 calendar day period during which field samples in a case are received.

5. Analytical protocol required (attach copy if other than a protocol currently used in this program):
- Follow the procedures outlined in Exhibit D of the CLP SOW (OLM03.1) for the analysis of VOCs in water by gas chromatography/mass spectrometry (GC/MS) with sample introduction by purge-and-trap instrumentation.
  - A list of the VOC target compounds with corresponding contract required quantitation limits (CRQL) is provided in Section 9 of this Regional Analytical Program (RAP) Client Request Form (CRF).
  - Analyze a 25-mL sample aliquot for all samples which do not exceed the calibration range when run without dilution.
  - Determine the pH of all samples to verify preservation to pH <2 according to Exhibit D, Section 10.2 of OLM03.1.
6. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

a. Calibration Procedure and Criteria:

- Perform a GC/MS tuning according to the procedure outlined in Exhibit D, Section 9.2 of the CLP SOW (OLM03.1). A GC/MS instrument performance check with 4-bromofluorobenzene (BFB) is required at the beginning of each 12 hour period during which standards and samples are analyzed. Instrument performance checks must meet the ion abundance criteria specified in Table 1 of Exhibit D, Section 17.0, of the CLP SOW (OLM03.1). The MS tune criteria must be met before any instrument calibration standards, environmental samples, laboratory method blanks, or quality control (QC) samples are analyzed.
- Perform initial instrument calibration according to the procedure outlined in Exhibit D, Section III, Part C, Item 14 of the Superfund Analytical Methods for Low Concentration Water for Organics Analysis (SAMLCO, 10/92 or more recent version). Prepare initial calibration standards, containing all of the target analytes listed in Table 1 of this RAP CRF and system monitoring compounds, according to the requirements of Table D-1 of Exhibit D, Section II, Part B, Item 7.5 of the SAMLCO (10/92) to the following specifications:

*Include 1,2-dibromoethane and 1,2-dibromo-3-chloropropane in initial*

| Volume of Working Standard ( $\mu$ L added to 25 mL) | Final Concentration of Aqueous Standard for Non-Ketones ( $\mu$ g/L) | Final Concentration of Aqueous Standard for Ketones ( $\mu$ g/L) |
|--|--|--|
| 5  | 1  | 5  |
| 10   | 2  | 10   |
| 25   | 5  | 25   |
| 50   | 10   | 50   |
| 125  | 25   | 125  |

*and continuing calibration standard solution See No. 8, page for criteria*

The minimum relative response factors (RRFs) for all analytes in the initial calibration must meet the acceptance criteria specified in Table D-3 of Exhibit D, Section V and Section III, Item 14.5.4 of SAMLCO (10/92). The percent relative standard deviation (%RSD) of analyte RRFs across the initial calibration curve must not exceed 30% except as noted in Section III, Item 14.5.4.

3. Analyze a sixth initial calibration standard containing carbon tetrachloride, 1,2-dichloroethane, cis-1,3-dichloropropene, trans-1,3-dichloropropene, and vinyl chloride at a concentration of 0.5 µg/L to verify that the requested CRQLs can be achieved. The minimum RRF requirement for these analytes is 0.05. This low level standard need not be included with the other five initial calibration standards in calculating the %RSD for the standard curve.
4. Perform continuing instrument calibration according to the procedure outlined in Exhibit D, Section III, Part C, Item 15 of the SAMLCO (10/92) once per each 12 hour period of instrument operation. Prepare a continuing calibration standard, containing all of the target analytes listed in Section 9 of this RAP CRF, at concentrations equivalent to the midpoint of the initial calibration curve. The minimum RRFs for all analytes in the continuing calibration must meet the acceptance criteria specified in Table D-3 of Exhibit D, Section V and Section III, Item 15.5.4 of SAMLCO (10/92). The percent difference (%D) between the RRFs from the continuing calibration and average RRFs from the initial calibration must not exceed ±30% except as noted in Section III, Item 15.5.4.

b. Internal Quality Control Checks, Control Limits and Corrective Actions:

1. When calibration standard measurements exceed the QC requirements for the initial calibration or the continuing calibration, take corrective action as specified in Exhibit D, Section III, Part C, Items 14.6 (for initial calibration) and 15.6 (for continuing calibration) of SAMLCO (10/92).

The continuing calibration standard reflects the conditions under which the analysis of all associated samples was performed. Reanalyze all samples associated with an out-of-control continuing calibration standard.

2. Analyze a laboratory method blank in each 12-hour time period in which samples are analyzed according to the procedure described in Exhibit D, Section 12.1 of the CLP SOW (OLM03.1). The concentration of target compounds in the laboratory method blanks must meet the requirements specified in Exhibit D, Section 12.1.4.5 of OLM03.1.

If a method blank exceeds these acceptance criteria, the analytical system should be considered to be out-of-control. Follow the corrective action procedures outlined in Exhibit D, Section 12.1.5 of the CLP SOW (OLM03.1). Investigate the source of contamination and document appropriate corrective measures taken before proceeding with further sample analysis. Reanalyze all samples processed with a method

blank that is out-of-control. Reanalyses must be performed at no additional cost to the Region.

3. Analyze instrument blanks after the analysis of a sample containing target analytes at concentrations exceeding the initial calibration range and storage blanks per Exhibit D, Section V, Item 26 of SAMLCO (10/92). The concentration of target compounds in the instrument and storage blanks must meet the requirements specified in Exhibit D, Section V, Item 26.4 of SAMLCO (10/92).

If an instrument blank exceeds these acceptance criteria, the analytical system should be considered to be out-of-control. Follow the corrective action procedures outlined in Exhibit D, Section V, Item 26.5 of SAMLCO (10/95). Investigate the source of the contamination and document the appropriate corrective measures taken before proceeding with further sample analysis. Reanalyze all samples processed with an instrument blank that is out-of-control. Reanalyses must be performed at no additional cost to the Region. Document storage blank contamination in the SDG narrative and retain all storage blank data at the laboratory.

4. Spike all environmental samples, laboratory method blanks, and QC samples with the system monitoring compounds (SMCs), toluene-d<sub>8</sub>, 4-bromofluorobenzene (BFB), and 1,2-dichloroethane-d<sub>4</sub>, according to the procedure outlined in Exhibit D, Section 10.1.3.7 of the CLP SOW (OLM03.1). Prepare the system monitoring compound spiking solution according to Exhibit D, Section 7.2.4.1 of the CLP SOW (OLM03.1).

System monitoring compound recoveries within the limits for water specified in Table 7 of Exhibit D, Section 17 are required. If these control limits are exceeded, take appropriate action to identify the problem by reanalyzing the affected sample. If reanalysis solves the problem, then the problem was within the laboratory's control. In this case, submit only data from the analysis with system monitoring compound recoveries within QC limits. If reanalysis of the sample does not solve the problem, then submit data from both the initial analysis and the reanalysis.

5. Add the internal standard compounds bromochloromethane, chlorobenzene-d<sub>4</sub>, and 1,4-difluorobenzene to all calibration standards, method blanks, and QC samples prior to analysis according to the procedure outlined in Exhibit D, Sections 9.3.3.3 (for initial calibration standards), 9.4.3.2 (for continuing calibration standards), and 10.1.3.7 (for samples and blanks) of the CLP SOW (OLM03.1). Prepare the internal standard compound spiking solution according to Exhibit D, Section 7.2.4.3 of the CLP SOW (OLM03.1).

Internal standard area counts must not vary by more than a factor of 2 (-50% to +100%) from the area counts of the associated continuing calibration standard. Internal standard retention times (RT) must not vary by more than ±30 seconds from the (RT) of the associated continuing calibration standard. Refer to Exhibit D, Sections 11.3.5 and 11.3.6 of the CLP SOW (OLM03.1) for internal standard acceptance criteria. If these control limits are exceeded,

reanalyze the affected sample. If reanalysis solves the problem, then the problem was within the laboratory's control. In this case, submit only data from the analysis with internal standard areas within QC limits. If reanalysis of the sample does not solve the problem, then submit data from both the initial analysis and the reanalysis.

6. Analyze matrix spike and matrix spike duplicate (MS/MSD) samples at the frequency of one per sample delivery group according to the procedure outlined in Exhibit D, Section 12.2 of the CLP SOW (OLM03.1). Prepare the matrix spiking solution according to Exhibit D, Section 7.2.4.2 of the CLP SOW (OLM03.1); prepare MS/MSD samples according to the procedure outlined in Exhibit D, Section 12.2.3 of the CLP SOW (OLM03.1).

*Include 1,2-dibromoethane  
and 1,2-dibromo-3-chloropropane  
in matrix spiking solution  
75% to 125% recoveries  
are required for these  
compounds.*

MS/MSD recoveries, and relative percent differences (RPDs) between MS/MSD recoveries, within the limits specified for water in Table 8 of Exhibit D, Section 17.0 are required. Flag MS/MSD results that exceed these criteria and note noncompliant MS/MSD results in the SDG narrative. No corrective action measures are required due to noncompliant MS/MSD results.

7. Dilute and reanalyze samples which contain one or more target analytes at concentrations above the initial calibration range. If dilution is necessary, adjust the dilution so that the most highly concentrated analyte is determined at a concentration in the upper half of the calibration range. Report the results and submit documentation for both the diluted and undiluted analyses.
8. The QC requirements listed above are the minimum required. It is impossible to address all analytical situations that might be experienced by a laboratory during the analysis of environmental samples. The laboratory is expected to adhere to good laboratory practices when analyzing samples. If the laboratory has questions concerning the analyses of samples not addressed in this document, the Region should be notified IMMEDIATELY. The Laboratory Manager, or designee, must address any problems and its resolution in the SDG narrative.

7. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion.

a. Data Calculations and Reporting Units:

1. Calculate the relative response factor (RRF) and the concentration of individual analytes according to the equations specified in Exhibit D, Sections 9.3.4.1 and 11.2.1 of the CLP SOW (OLM03.1).
2. Report sample results in concentration units of micrograms per liter ( $\mu\text{g/L}$ ). Report results less than 10  $\mu\text{g/L}$  to 1 significant figure and results greater than or equal to 10  $\mu\text{g/L}$  to 2 significant figures.

3. For rounding results, adhere to the following rules:
  - a) If the digit following those to be retained is less than 5, round down;
  - b) If the digit following those to be retained is greater than 5, round up; or
  - c) If the digit following the last digit to be retained is 5, round down if the digit is even, and round up if the digit is odd.
4. Ensure that all records of analysis, dilutions and calculations are legible and sufficient to recalculate all sample concentrations and QC results. Include an example of an actual sample calculation in the data package.

b. Documentation and Deliverables:

All documentation and deliverables as required in Exhibit B of the CLP SOW (OLM03.1) must be submitted. The required deliverables for each SDG must include the following:

1. All original shipping documents and sample tracking reports including signed RAP chain-of-custody forms, airbills, and traffic reports.
2. A completed and signed document inventory on a modified Organics Complete SDG File (CSF) Inventory Sheet (CLP Form DC-2).
3. All original sample receipt documents including sample log-in information on a modified CLP Form DC-1, an SDG cover sheet, and any other receipt forms such as copies of receipt logbooks.
4. A copy of the RAP CRF, as provided by the Region (so that any additions or revisions authorized by the Region will be known). Only the technical portion of the CRF is required.
5. Any telephone logs referring to the samples.
6. An SDG narrative signed by the laboratory manager, or designee, certifying the accuracy and validity of all data reported. The SDG narrative must contain: laboratory name; laboratory code; contract number; RAP number; SDG number; a list of correct EPA sample numbers and the corresponding laboratory sample identification (ID) numbers, differentiating between the initial analyses and any reanalyses; and the pH of all water samples. The SDG narrative must provide a description of all GC columns used for analysis, including brand name, the internal diameter in millimeters (mm), the length in meters, coating material, and film thickness. The SDG narrative must describe any administrative or technical problems encountered during the processing of the samples and a description of the resolution of these problems. The SDG narrative must include an explanation for any manual integrations or manual edits. The SDG narrative must provide a formula (including definitions) showing how the results were calculated, and an example of an actual calculation for a sample in the SDG.
7. The following header information is required for each data reporting form: laboratory name; contract number; laboratory code; RAP number; and SDG number.

8. Tabulated analysis results for all environmental and QC sample results on a modified CLP Form 1. Include the compound name and Chemical Abstract Service (CAS) registry number. Clearly specify concentration units and laboratory qualifiers. Include the following additional information in the header: laboratory sample ID, laboratory file ID, sample volume, purge volume, dilution factor, the validated time of sample receipt (VTSR), analysis date, and pH. Forms for any reanalyses or diluted analyses must be included.
9. Modified CLP Form 1E for all environmental samples which lists tentatively identified compounds. The modified CLP Form 1E must report the CAS number, compound name, RT, the estimated concentration, and the laboratory qualifier for each TIC reported. The header information should be the same as that provided in CLP Form 1, listed above.
10. Raw sample data must include the following items:
  - a) Reconstructed ion chromatograms (RICs) labeled with the following information: EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID. Each internal standard and surrogate compound must be labeled on the RIC by name.
  - b) Copies of raw mass spectra and background-subtracted mass spectra of detected target compounds labeled with the compound name, EPA sample number, laboratory file ID, analysis date and time, and instrument ID. For TICs, copies of mass spectra with associated 3 best-match spectra must be provided and labeled as above.
  - c) GC/MS quantitation report/data system printouts containing the following information: EPA sample number, date and time of analysis, RT or scan numbers of identified target compounds, ion used for quantitation, copy of area table from data system, GC/MS instrument ID, laboratory file ID, and analyst ID.
  - d) Extracted ion current profile (EICPs) displaying any manual integrations
11. Modified CLP Form 2A which lists the percent recovery (%R) values, rounded to the nearest whole number, and the QC limits for each system monitoring compound in all samples and laboratory method blanks. Flag all recoveries outside the QC limits with an asterisk.
12. Modified CLP Form 3A, a matrix spike/matrix spike duplicate (MS/MSD) results summary, which reports percent recoveries and RPD values to the nearest whole number for the spike compounds. List the QC limits for percent recovery and RPD for each spike compound and flag all values outside of the QC limits with an asterisk. Tabulate the concentration of analytes added to the sample, the sample concentration, and the MS and MSD concentrations for each analyte spiked. Include the EPA sample number in the header.
13. Modified CLP Form 4A which summarizes the samples that were analyzed with each laboratory method blank. The EPA sample

- number, the laboratory sample ID, and analysis time must be reported for each sample associated with the referenced blank. Include the following additional information in the header: the laboratory method blank EPA sample number, laboratory file ID, laboratory sample ID, method blank analysis date and time, the GC column ID (i.e. liquid phase, length, diameter), and instrument ID.
14. CLP Form 5A which summarizes the data for BFB instrument performance checks. The ion, the abundance criteria, and the percent relative abundance for each ion must be reported. The modified CLP Form 5A must include the EPA sample number, laboratory sample ID, laboratory file ID, and date and time of analysis for each sample associated with the referenced BFB instrument performance check. Include the following additional information in the header: laboratory file ID, BFB injection date and time, instrument ID, and GC column ID.
15. Instrument calibration data which include the following items:
- a) Initial Calibration
- 1) Modified CLP Form 6A which includes: RRFs for each analyte and surrogate for each concentration level, and average RRFs and %RSDs for each target and surrogate compound
  - 2) Include the following additional information in the form header: date and time of calibration, instrument ID, and GC analytical column ID
  - 3) Raw data which include: RICs and quantitation reports/data system printouts. RICs must contain the following information: EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID. Each internal standard and surrogate compound must be labeled on the RIC by name. Quantitation reports/data system printouts must contain the following information: EPA sample number, date and time of analysis, RT or scan numbers of identified target compounds, ion used for quantitation, copy of area table from data system, GC/MS instrument ID, laboratory file ID, and analyst ID.
  - 4) Extracted ion current profile (EICPs) displaying any manual edits and integrations.
- b) Continuing Calibration
- 1) Modified CLP Form 7A which includes: average RRFs (from the initial calibration), RRFs (for the continuing calibration standard), and %D values for each target and surrogate compound.
  - 2) Include the following additional information in the header: laboratory file ID, date and time of the continuing calibration, instrument ID, GC

analytical column ID, and the date and time of the referenced initial calibration

- 3) Raw data which include: RICs must contain the following information: EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID. Each internal standard and surrogate compound must be labeled on the RIC by name. Quantitation reports/data system printouts must contain the following information: EPA sample number, date and time of analysis, RT or scan numbers of identified target compounds, ion used for quantitation, copy of area table from data system, GC/MS instrument ID, laboratory file ID, and analyst ID.
  - 4) Extracted ion current profile (EICPs) displaying any manual edits and integrations.
16. Modified CLP Form 8A which summarizes areas and RTs of the internal standards for all samples, blanks, QC samples associated with the referenced continuing calibration standard. Clearly specify the QC limits for the internal standard response and RT and flag all results outside the QC limits. Include the following information in the header: laboratory file ID for the standard, date and time of analysis, instrument ID, and GC column ID.
17. Raw QC data which include the following items:
- a) Blank data, in chronological order:
    - 1) Tabulated results on a modified CLP Form 1A and 1E
    - 2) RIC, mass spectra, and best-match spectra labeled as specified above
    - 3) GC/MS quantitation report/data system printout labeled as specified above
    - 4) EICPs displaying any manual integrations or edits
  - b) MS/MSD data, in chronological order:
    - 1) Tabulated results on a modified CLP Form 1A
    - 2) RIC labeled as specified above
    - 3) GC/MS quantitation report/data system printout labeled as specified above
    - 4) EICPs displaying any manual integrations or edits
  - c) BFB Instrument Performance Check data, in chronological order:
    - 1) Bar graph spectrum labelled with EPA sample number, date and time of analysis, GC/MS

instrument identifier, laboratory file ID, and analyst ID.

- 2) Mass listing labeled with EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID
- 3) RICs labeled with EPA sample number, date and time of analysis, GC/MS instrument identifier, laboratory file ID, and analyst ID.

18. Bench sheets for pH determination and dilutions
19. Bench sheets for sample analysis including instrument ID and analysis date and time
20. Bench sheets for sample preparation which include spike solution and SMC solution description (i.e. concentrations and volume added)
21. Standards preparation logs, for all standards used for either calibration or spiking, which include source, traceable lot number, and concentrations of all compounds
22. Any internal laboratory sample or sample extract transfer records and tracking sheets

8. Other (use additional sheets or attach supplementary information, as needed):

If a copy of the "U.S. EPA Region 9 Laboratory QC Summary Report" form is attached, complete the form by following the directions on the first page of the form.

Initial and continuing calibration criteria for 1,2-dibromoethane and 1,2-dibromo-3-chloropropane are as follows:

|                             | <u>min. RRF</u> | <u>max % RSD</u> | <u>max % D (continuing calibration)</u> |
|-----------------------------|-----------------|------------------|---|
| 1,2-dibromoethane           | 0.100           | 30.0             | ± 30.0                                  |
| 1,2-dibromo-3-chloropropane | 0.010           | 30.0             | ± 30.0                                  |

1,2-dibromoethane criteria are from SAM LCO. 1,2-dibromo-3-chloropropane criteria are specific for this project. Four point initial calibration for 1,2-dibromo-3-chloropropane is acceptable if five point calibration does not meet the above criteria due to poor response at the 1 µg/l level.

9. Data Requirements:

The required target compounds with corresponding contract required quantitation limits (CRQLs) are provided below.

| <u>Analyte</u>             | <u>CAS Number</u> | <u>(CRQL) <math>\mu\text{g/L}</math></u> |
|----------------------------|-------------------|--|
| Chloromethane              | 74-87-3           | 1  |
| Bromomethane               | 74-83-9           | 1  |
| Vinyl chloride             | 75-01-4           | 0.5                                      |
| Chloroethane               | 75-00-3           | 1  |
| Methylene chloride         | 75-09-2           | 1  |
| Acetone                    | 67-64-1           | 10                                       |
| Carbon disulfide           | 75-15-0           | 1  |
| 1,1-Dichloroethene         | 75-35-4           | 1  |
| 1,1-Dichloroethane         | 75-34-3           | 1  |
| 1,2-Dichloroethene (total) | 540-59-0          | 1  |
| Chloroform                 | 67-66-3           | 1  |
| 1,2-Dichloroethane         | 107-06-2          | 0.5                                      |
| 2-Butanone                 | 78-87-5           | 10                                       |
| 1,1,1-Trichloroethane      | 71-55-6           | 1  |
| Carbon tetrachloride       | 56-23-5           | 0.5                                      |
| Bromodichloromethane       | 75-27-4           | 1  |
| 1,2-Dichloropropane        | 78-87-5           | 1  |
| cis-1,3-Dichloropropene    | 10061-01-5        | 0.5                                      |
| Trichloroethene            | 79-01-6           | 1  |
| Dibromochloromethane       | 124-48-1          | 1  |
| 1,1,2-Trichloroethane      | 79-00-5           | 1  |
| Benzene                    | 71-43-2           | 1  |
| trans-1,3-Dichloropropene  | 10061-02-6        | 0.5                                      |
| Bromoform                  | 75-25-2           | 1  |
| 4-Methyl-2-pentanone       | 108-10-1          | 10                                       |
| 2-Hexanone                 | 591-78-6          | 10                                       |
| Tetrachloroethene          | 127-18-4          | 1  |
| 1,1,2,2-Tetrachloroethane  | 79-34-5           | 1  |
| Toluene                    | 108-88-3          | 1  |
| Chlorobenzene              | 108-90-7          | 1  |
| Ethylbenzene               | 100-41-4          | 1  |
| Styrene                    | 100-42-5          | 1  |
| Xylenes (total)            | 1330-20-7         | 1  |

U. S. EPA REGION 9  
LABORATORY QC SUMMARY REPORT

LABORATORY: \_\_\_\_\_

SAS #:

SUBMITTED BY: \_\_\_\_\_

# SAMPLES:

TITLE: \_\_\_\_\_

MATRIX:

ANALYSIS: VOLATILE ORGANIC COMPOUNDS  
IN WATER FOLLOWING CONTRACT  
LABORATORY PROGRAM (CLP)  
STATEMENT OF WORK (SOW) FOR  
ORGANICS ANALYSIS (OLM02.1)  
PROTOCOLS USING A 25 ML PURGE  
VOLUME

DATE:

QC SUMMARY TABLE

| QC PARAMETER   | QC LIMITS   | FREQUENCY   |
|--|---|---|
| Laboratory method blank (concentration of target analytes)       | <CRQL   | each 12 hour period during sample analysis              |
| Initial and continuing calibration (4-bromofluorobenzene tuning) | as per requirements listed in Table 1 of Exhibit D, Section 17 of CLP SOW (OLM02.1)                   | beginning of each 12 hour period during sample analysis |
| Initial calibration (%RSD)                                       | <30% for all analytes (in 5 point calibration)  | as needed   |
| Initial calibration (minimum relative response factor [RRF])     | as per requirements listed in Table D-3 of SAMICO (10/92) or on page 9                                | as needed   |
| Initial calibration (minimum relative response factor [RRF])     | 0.05 (criterion applies to sixth initial calibration standard containing analytes with 0.5 g/L CRQLs) | with every initial calibration performed                |
| Continuing calibration (%D)                                      | 30% for all analytes  | every 12 hours during sample analysis                   |

*9 of SAS CRF for EDB and DCP*

QC SUMMARY TABLE (Continued)

| QC PARAMETER                                | QC LIMITS  | FREQUENCY  |
|---|--|--|
| Continuing calibration<br>(minimum RRF)     | as per requirements listed in Table D-3 of Exhibit D, Section V of SAMICO (10/92) or as listed | every 12 hours during sample analysis<br>of SWS CRF<br>on page 9 for EOB and 10/94 |
| System monitoring compounds (IR)            | as per requirements listed in Table 7 of Exhibit D, Section 17 of SOW (OLM02.1)                | all standards, blanks, environmental samples, and QC samples                       |
| Internal standards (areas, retention times) | as per requirements listed in Exhibit D, Sections 11.3.5 and 11.3.6 of SOW (OLM02.1)           | all blanks, environmental samples, and QC samples                                  |
| MS/MSD (IR)                                 | as per requirements listed in Table 8 of Exhibit D, Section 17 of SOW (OLM02.1)                | 1 per SDG  |
| MS/MSD (RPD)                                | as per requirements listed in Table 8 of Exhibit D, Section 17 of SOW (OLM02.1)                | 1 per SDG  |

1. Were all samples analyzed within the contract required holding time of 10 days from the date of sample receipt? YES NO
- a. If no, list the samples that were analyzed outside of the holding time.
- b. How many days outside of the holding time were these samples analyzed?
2. Were all samples analyzed within the technical holding time of 14 days (for preserved samples) or 7 days (for unpreserved samples) from the date of collection? YES NO
- a. If no, list the samples that were analyzed outside of the holding time
- b. How many days outside of the holding time were these samples analyzed?

3. Were all samples received intact and in good condition? YES NO
4. Was the data package sent within 35 days from the receipt of the last samples in the SDG? YES NO
- a. If no, how many days late was the data package sent?
5. Were the protocols outlined in the Contract Laboratory Program (CLP) Statement of Work (SOW) for Organics Analysis (OLM02.1 or more recent version) followed for the analysis of these samples? YES NO
- a. If no, specify which method was used.
- b. If no, why was this method used and who authorized its use?
- c. Was the approved method followed without modifications or deviations? YES NO
- d. If no, specify what the modification or deviations were and who approved them.
6. Was a BFB tune performed every 12 hours during sample analysis? YES NO
- a. If no, why not?
7. Were the ion abundance criteria specified in Table 1 of Exhibit D, Section 17 of the SOW (OLM02.1) met for all BFB tunes that were performed? YES NO
- a. If no, why not?
8. Was a 5-point initial calibration curve analyzed prior to sample analyses? YES NO

- a. If yes, when?
- b. If no, why not?
9. Did the initial calibration standards contain all target analytes at the concentrations specified in Table D-1 of Exhibit D, Section II, Part B; Item 7.5 of the Superfund Analytical Methods for Low Concentration Water for Organics Analysis (SAMCO, 10/92)? YES NO
- a. If no, why not?
10. Did the initial calibration curve meet the QC requirement of <30% RSD for all target compounds? YES NO
- a. If no, specify the analyte(s) and %RSD(s) that were outside of the QC limits.
11. Was a sixth initial calibration standard containing carbon tetrachloride, 1,2-dichloroethane, cis and trans-1,3-dichloropropene, and vinyl chloride at a concentration of 0.5 g/L analyzed as part of the initial calibration? YES NO
- a. If no, why not?
12. Was a minimum RRF of 0.05 achieved for all analytes in the sixth initial calibration standard? YES NO
- a. If no, why not?
13. Was a continuing calibration standard analyzed every 12-hour time period in which samples were analyzed? YES NO
- a. If no, why not?
14. Were percent differences (%D) for YES NO

target compounds in the continuing calibration standards within the QC requirement of 30%?

a. If no, specify the analyte(s) and ID(s) that were outside of the QC limits.

15. Were the RRFs for analytes in the initial calibration within the requirements specified Table D-3 of Exhibit D, Section V of the SAMLCO (10/92)?

YES NO

In continuing calibrations?

YES NO

a. If no, why not?

16. Was a 25-mL purge volume used for the analysis of all samples?

YES NO

a. If no, why not?

17. Were the area counts of all internal standards within -50% to +100% of the area count for each respective internal standard in the associated continuing calibration?

YES NO

a. If no, specify the sample(s) and internal standard(s) that were outside of the QC limits.

18. Were the retention times of all the internal standards within 30 seconds of the retention time for each respective internal standard in the associated continuing calibration?

YES NO

a. If no, specify the sample(s) and internal standard(s) that were outside of the QC limits.

19. Were laboratory method blanks analyzed at a minimum frequency of 1 per each 12 hour period during sample analysis? YES NO

a. If no, at what frequency were method blank analyses performed.

20. Were laboratory method blanks free of contamination at concentrations greater than the CRQL for all analytes? YES NO

a. If no, specify contaminant(s) and at what level(s) it was (they were) present.

21. Were recoveries for system monitoring compounds within the requirements specified in Table 7 of Exhibit D, Section 17 of the CLP SOW (OLM02.1) for all samples? YES NO

a. If no, list the recoveries and the associated samples that were above this range.

b. If no, list the recoveries and the associated samples that were below this range.

c. How many noncompliant recoveries were caused by dilutions?

d. How many noncompliant recoveries were caused by matrix interferences?

e. Were samples with noncompliant recoveries reanalyzed? YES NO

If no, why not?

22. Were MS/MSD analyses performed at a minimum frequency of 1 per SDG? YES NO

a. If no, why not?

23. Were MS/MSD recoveries within the requirements specified in Table 8 of Exhibit D, Section 17, of the CLP SOW (OLM02.1) for all spike compounds? YES NO

a. If no, specify the analyte(s) and %R(s) that were outside of the QC limits.

24. Were the relative percent differences (RPDs) between MS/MSD recoveries within the requirements specified in Table 8 of Exhibit D, Section 17 of the SOW (OLM02.1) for all spike compounds? YES NO

a. If no, specify the analyte(s) and %RPD(s) that were outside of the QC limits.

25. Were the CROQLs met for all analytes? YES NO

a. If no, why not?

26. Was it possible to analyze all target compounds within the range of the initial calibration curve? YES NO

a. If no, were these samples diluted according to the instructions in Section 8.b.5 of the SAS Client Request Form? YES NO

b. List these samples, the analytes above range, and the analyte concentration.

c. If no, explain why not and list the samples.