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# WP-60 Final Report

**ERA Laboratory Code: C0535-01A EPA ID: CA00112 State ID: 1664** **Report Issued: 03/28/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Warning Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|---|
| ***Base Neutrals*** | Acenaphthene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Acenaphthylene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Aniline | µg/l | | < 10.0 | | | | |
| | Anthracene | µg/l | 7.96 | 13.7 | 1.56 - 22.4 | 5.04 - 19.0 | Acceptable | EPA610 |
| | Benzidine | µg/l | | < 10.0 | | | | |
| | Benzo(a)anthracene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Benzo(b)fluoranthene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Benzo(k)fluoranthene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Benzo(g,h,i)perylene | µg/l | 20.0 | 21.8 | D.L. - 38.7 | 2.36 - 31.4 | Acceptable | EPA610 |
| | Benzo(a)pyrene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Benzyl alcohol | µg/l | | < 10.0 | | | | |
| | 4-Bromophenyl-phenylether | µg/l | | < 10.0 | | | | |
| | Butylbenzylphthalate | µg/l | | < 10.0 | | | | |
| | Carbazole | µg/l | | < 10.0 | | | | |
| | 4-Chloroaniline | µg/l | | < 10.0 | | | | |
| | bis(2-Chloroethoxy)methane | µg/l | | < 10.0 | | | | |
| | bis(2-Chloroethyl)ether | µg/l | | < 10.0 | | | | |
| | 1-Chloronaphthalene | µg/l | | < 10.0 | | | | |
| | 2-Chloronaphthalene | µg/l | | 84.0 | 10.5 - 114 | 27.4 - 97.7 | | |
| | 4-Chlorophenyl-phenylether | µg/l | | < 10.0 | | | | |
| | 2,2'-oxybis(1-chloropropane) | µg/l | | < 10.0 | | | | |
| | Chrysene | µg/l | 36.4 | 40.4 | 11.0 - 57.2 | 18.7 - 49.5 | Acceptable | EPA610 |
| | Dibenz(a,h)anthracene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Dibenzofuran | µg/l | | 86.5 | 27.4 - 110 | 41.1 - 95.9 | | |
| | Di-n-butylphthalate | µg/l | | < 10.0 | | | | |
| | 1,2-Dichlorobenzene | µg/l | | 47.9 | D.L. - 67.1 | 6.60 - 55.0 | | |
| | 1,3-Dichlorobenzene | µg/l | | < 10.0 | | | | |
| | 1,4-Dichlorobenzene | µg/l | | 119 | D.L. - 170 | 9.90 - 141 | | |
| | 3,3'-Dichlorobenzidine | µg/l | | < 10.0 | | | | |
| | Diethylphthalate | µg/l | | 96.5 | D.L. - 149 | 23.1 - 124 | | |
| | Dimethylphthalate | µg/l | | < 10.0 | | | | |
| | 2,4-Dinitrotoluene | µg/l | | 60.7 | 14.3 - 81.2 | 25.4 - 70.0 | | |
| | 2,6-Dinitrotoluene | µg/l | | 158 | 41.9 - 203 | 68.6 - 179 | | |
| | Di-n-octylphthalate | µg/l | | < 10.0 | | | | |
| | bis(2-ethylhexyl)phthalate | µg/l | | 129 | 16.7 - 203 | 47.8 - 172 | | |
| | Fluoranthene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Fluorene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Hexachlorobenzene | µg/l | | < 10.0 | | | | |
| | Hexachlorobutadiene | µg/l | | < 10.0 | | | | |
| | Hexachlorocyclopentadiene | µg/l | | < 10.0 | | | | |
| | Hexachloroethane | µg/l | | 95.2 | D.L. - 156 | D.L. - 123 | | |
| | Indeno(1,2,3-cd)pyrene | µg/l | 0.00 | < 10.0 | | | Acceptable | EPA610 |
| | Isophorone | µg/l | | < 10.0 | | | | |
| | 1-Methylnaphthalene | µg/l | | < 10.0 | | | | |
| | 2-Methylnaphthalene | µg/l | | < 10.0 | | | | |

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| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Warning Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|---|
| ***Acids*** | Benzoic acid | µg/l | | < 10.0 | | | | |
| | 4-Chloro-3-methylphenol | µg/l | | 138 | 40.3 - 184 | 64.2 - 160 | | |
| | 2-Chlorophenol | µg/l | | 29.9 | 5.07 - 40.5 | 11.0 - 34.6 | | |
| | 2,4-Dichlorophenol | µg/l | | < 10.0 | | | | |
| | 2,6-Dichlorophenol | µg/l | | < 10.0 | | | | |
| | 2,4-Dimethylphenol | µg/l | | < 10.0 | | | | |
| | 4,6-Dinitro-2-methylphenol | µg/l | | < 10.0 | | | | |
| | 2,4-Dinitrophenol | µg/l | | < 10.0 | | | | |
| | 2-Methylphenol | µg/l | | 80.2 | 7.60 - 110 | 22.2 - 92.8 | | |
| | 3-Methylphenol | µg/l | | < 10.0 | | | | |
| | 4-Methylphenol | µg/l | | < 10.0 | | | | |
| | 2-Nitrophenol | µg/l | | < 10.0 | | | | |
| | 3-Nitrophenol | µg/l | | < 10.0 | | | | |
| | 4-Nitrophenol | µg/l | | < 10.0 | | | | |
| | Pentachlorophenol | µg/l | | 134 | 10.1 - 200 | 41.7 - 168 | | |
| | Phenol | µg/l | | 71.7 | D.L. - 111 | D.L. - 88.7 | | |
| | 2,4,5-Trichlorophenol | µg/l | | < 10.0 | | | | |
| | 2,4,6-Trichlorophenol | µg/l | | 120 | 30.3 - 160 | 51.8 - 138 | | |

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# WP-60 Score and Overall Assessment

**ERA Laboratory Code: C0535-01A EPA ID: CA00112 State ID: 1664** **Report Issued: 03/28/00**

| Number of Acceptable Analytes | Number of Reported Analytes | InterLaB™ Score | Overall Assessment |
|---|---|---|---|
| 99 | 105 | 94.3% | Very Good |

**Scoring Definitions:**

- The **Number of Acceptable Analytes** is equal to any reported values that resulted in an "Acceptable" performance evaluation for the laboratory. An acceptable performance evaluation falls with in the Warning Limits.

- The **Number of Reported Analytes** is equal to any evaluated values reported during this study.

- The **InterLaB™ Score** is equal to the sum of the Number of Acceptable Analytes divided by the total Number of Reported Analytes.

- The **Overall Assessment:**
  **Excellent** = InterLaB™ Score falls within 95.0% and 100%.
  **Very Good** = InterLaB™ Score falls within 85.0% and 94.9%.
  **Good** = InterLaB™ Score falls within 75.0% and 84.9%.
  **Acceptable** = InterLaB™ Score falls within 65.0% and 74.9%.
  **Needs Improvement** = InterLaB™ Score is equal to or below 64.9%.

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# WP-60 Definitions & Study Discussion

**ERA Laboratory Code: C0535-01B EPA ID: CA00112 State ID: 1664 Report Issued: 03/28/00**

## InterLaB WatR™ Pollution Definitions:

The **Reported Value** is the value that the laboratory reported to ERA.

The **ERA Assigned Value** is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. A parameter not added to the standard is given an **Assigned Value** of **"Zero"** per the guidelines contained in the EPA's National Standards for Water Proficiency Testing Criteria Document, December 1998 as applicable.

The **Acceptance Limits** and **Warning Limits** are established per the guidelines contained in the EPA's National Standards for Water Proficiency Testing Criteria Document, December 1998 as applicable.

The **Performance Evaluation:**

**Acceptable =** Reported Value falls within the Acceptance Limits.

**Not Acceptable =** Reported Value falls outside the Acceptance Limits.

**Check for Error =** Reported Value falls within the Acceptance Limits and outside of the Warning Limits.

**No Evaluation =** Reported Values that can not be evaluated.

The **Method Description** is the method the laboratory reported to ERA.

**D.L.** equals the Detection Limit.

## InterLaB WatR™ Pollution Study Discussion:

ERA WatR™ Pollution Proficiency Testing Study 60 has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and those contained in the National Institute for Standards and Technologies Handbooks 150 and 150-19.

Per the requirements of the USEPA's Criteria Document and the NIST NVLAP Handbooks, the WatR™ Pollution 60 (WP60) results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WP60 standards met the acceptance criteria contained in the US EPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program, Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing, June 1999.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no study anomolies were found and all Acceptance Limits for the InterLaB WatR™ Pollution study, WP60, were calculated based on the US EPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, as applicable.

If you have any questions regarding WatR™ Pollution Study, WP 60, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Roland P. Bruggeman, InterLaB™ Chemist, at 1-800-372-0122.

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# WP-60 Final Report

**ERA Laboratory Code: C0535-01B EPA ID: CA00112 State ID: 1664 Report Issued: 03/28/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Warning Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|---|
| ***Minerals*** | Total Solids at 105°C | mg/l | | 244 | 166 - 310 | 190 - 286 | | |
| | Total Dissolved Solids | mg/l | | 228 | 168 - 288 | 188 - 268 | | |
| | Conductivity at 25°C | µmhos | | 315 | 291 - 339 | 299 - 331 | | |
| | Alkalinity as CaCO3 | mg/l | | 55.3 | 49.2 - 61.7 | 51.3 - 59.6 | | |
| | Chloride | mg/l | 34.4 | 39.5 | 34.8 - 43.6 | 36.3 - 42.2 | Not Acceptable | SM4110B |
| | Fluoride | mg/l | 2.43 | 2.54 | 2.21 - 2.84 | 2.32 - 2.73 | Acceptable | SM4110B |
| | Potassium | mg/l | 26.6 | 24.5 | 21.1 - 28.0 | 22.2 - 26.8 | Acceptable | SM3120B |
| | Sodium | mg/l | 50.2 | 50.4 | 45.4 - 55.6 | 47.1 - 53.9 | Acceptable | SM3120B |
| | Sulfate | mg/l | 21.2 | 23.7 | 18.7 - 28.1 | 20.2 - 26.6 | Acceptable | SM4110B |
| ***pH*** | pH | S.U. | | 9.00 | 8.73 - 9.27 | 8.82 - 9.18 | | |
| ***Hardness*** | Total Suspended Solids | mg/l | | 36.3 | 26.7 - 38.7 | 28.7 - 36.7 | | |
| | Calcium | mg/l | 43.5 | 42.4 | 37.8 - 48.2 | 39.5 - 46.4 | Acceptable | SM3120B |
| | Magnesium | mg/l | 30.1 | 29.8 | 26.1 - 33.4 | 27.3 - 32.2 | Acceptable | SM3120B |
| | Calcium hardness (CaCO3) | mg/l | 109 | 106 | 83.8 - 128 | 91.2 - 121 | Acceptable | SM3120B CALC |
| | Total Hardness (CaCO3) | mg/l | 230 | 228 | 208 - 249 | 215 - 242 | Acceptable | SM3120B CALC |
| ***Demand*** | BOD | mg/l | | 37.1 | 18.5 - 55.7 | 24.7 - 49.5 | | |
| | CBOD | mg/l | | 32.0 | 14.3 - 49.7 | 20.2 - 43.8 | | |
| | COD | mg/l | | 59.8 | 41.6 - 73.2 | 46.9 - 67.9 | | |
| | TOC | mg/l | | 23.6 | 19.6 - 27.5 | 20.9 - 26.2 | | |
| ***Nutrients - Simple*** | Ammonia as N | mg/l | | 1.30 | 0.926 - 1.69 | 1.05 - 1.56 | | |
| | Nitrate as N | mg/l | 13.5 | 16.0 | 12.7 - 19.0 | 13.7 - 17.9 | Check for Error | SM4100B |
| | Ortho-phosphate as P | mg/l | | 3.31 | 2.82 - 3.83 | 2.99 - 3.66 | | |
| ***Nutrients - Complex*** | Total phosphorus as P | mg/l | | 4.34 | 3.30 - 5.09 | 3.60 - 4.79 | | |
| | Total kjeldahl nitrogen | mg/l | | 5.78 | 3.97 - 7.51 | 4.56 - 6.92 | | |
| ***Cyanide*** | Cyanide, total | mg/l | | 0.729 | 0.506 - 0.937 | 0.578 - 0.865 | | |
| ***Phenolics*** | Phenolics, total | mg/l | | 0.748 | 0.408 - 1.09 | 0.522 - 0.974 | | |
| ***Grease & Oil*** | Grease & Oil (Gravimetric) | mg/l | | 29.7 | 18.7 - 34.9 | 21.4 - 32.2 | | |
| ***TRC*** | Total Residual Chlorine | mg/l | | 1.33 | 1.05 - 1.61 | 1.14 - 1.52 | | |

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# WP-60 Score and Overall Assessment

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| Number of Acceptable Analytes | Number of Reported Analytes | InterLaB™ Score | Overall Assessment |
|---|---|---|---|
| 27 | 30 | 90.0% | Very Good |

**Scoring Definitions:**

- The **Number of Acceptable Analytes** is equal to any reported values that resulted in an "Acceptable" performance evaluation for the laboratory. An acceptable performance evaluation falls with in the Warning Limits.

- The **Number of Reported Analytes** is equal to any evaluated values reported during this study.

- The **InterLaB™ Score** is equal to the sum of the Number of Acceptable Analytes divided by the total Number of Reported Analytes.

- The **Overall Assessment:**
  **Excellent** = InterLaB™ Score falls within 95.0% and 100%.
  **Very Good** = InterLaB™ Score falls within 85.0% and 94.9%.
  **Good** = InterLaB™ Score falls within 75.0% and 84.9%.
  **Acceptable** = InterLaB™ Score falls within 65.0% and 74.9%.
  **Needs Improvement** = InterLaB™ Score is equal to or below 64.9%.

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# WP-60 Definitions & Study Discussion

**ERA Laboratory Code: C0535-01C EPA ID: CA00112 State ID: 1664 Report Issued: 03/28/00**

## InterLaB WatR™ Pollution Definitions:

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The **Acceptance Limits** and **Warning Limits** are established per the guidelines contained in the EPA's National Standards for Water Proficiency Testing Criteria Document, December 1998 as applicable.

The **Performance Evaluation:**

**Acceptable =** Reported Value falls within the Acceptance Limits.

**Not Acceptable =** Reported Value falls outside the Acceptance Limits.

**Check for Error =** Reported Value falls within the Acceptance Limits and outside of the Warning Limits.

**No Evaluation =** Reported Values that can not be evaluated.

The **Method Description** is the method the laboratory reported to ERA.

**D.L.** equals the Detection Limit.

## InterLaB WatR™ Pollution Study Discussion:

ERA WatR™ Pollution Proficiency Testing Study 60 has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and those contained in the National Institute for Standards and Technologies Handbooks 150 and 150-19.

Per the requirements of the USEPA's Criteria Document and the NIST NVLAP Handbooks, the WatR™ Pollution 60 (WP60) results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WP60 standards met the acceptance criteria contained in the US EPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program, Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing, June 1999.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no study anomolies were found and all Acceptance Limits for the InterLaB WatR™ Pollution study, WP60, were calculated based on the US EPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, as applicable.

If you have any questions regarding WatR™ Pollution Study, WP 60, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Roland P. Bruggeman, InterLaB™ Chemist, at 1-800-372-0122.

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| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Warning Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|---|
| ***Minerals*** | Total Solids at 105°C | mg/l | | 244 | 166 - 310 | 190 - 286 | | |
| | Total Dissolved Solids | mg/l | | 228 | 168 - 288 | 188 - 268 | | |
| | Conductivity at 25°C | µmhos | | 315 | 291 - 339 | 299 - 331 | | |
| | Alkalinity as CaCO3 | mg/l | | 55.3 | 49.2 - 61.7 | 51.3 - 59.6 | | |
| | Chloride | mg/l | | 39.5 | 34.8 - 43.6 | 36.3 - 42.2 | | |
| | Fluoride | mg/l | 2.78 | 2.54 | 2.21 - 2.84 | 2.32 - 2.73 | Check for Error | SM4500F-C |
| | Potassium | mg/l | | 24.5 | 21.1 - 28.0 | 22.2 - 26.8 | | |
| | Sodium | mg/l | | 50.4 | 45.4 - 55.6 | 47.1 - 53.9 | | |
| | Sulfate | mg/l | | 23.7 | 18.7 - 28.1 | 20.2 - 26.6 | | |
| ***pH*** | pH | S.U. | | 9.00 | 8.73 - 9.27 | 8.82 - 9.18 | | |
| ***Hardness*** | Total Suspended Solids | mg/l | | 36.3 | 26.7 - 38.7 | 28.7 - 36.7 | | |
| | Calcium | mg/l | | 42.4 | 37.8 - 48.2 | 39.5 - 46.4 | | |
| | Magnesium | mg/l | | 29.8 | 26.1 - 33.4 | 27.3 - 32.2 | | |
| | Calcium hardness (CaCO3) | mg/l | | 106 | 83.8 - 128 | 91.2 - 121 | | |
| | Total Hardness (CaCO3) | mg/l | | 228 | 208 - 249 | 215 - 242 | | |
| ***Demand*** | BOD | mg/l | | 37.1 | 18.5 - 55.7 | 24.7 - 49.5 | | |
| | CBOD | mg/l | | 32.0 | 14.3 - 49.7 | 20.2 - 43.8 | | |
| | COD | mg/l | | 59.8 | 41.6 - 73.2 | 46.9 - 67.9 | | |
| | TOC | mg/l | | 23.6 | 19.6 - 27.5 | 20.9 - 26.2 | | |
| ***Nutrients - Simple*** | Ammonia as N | mg/l | | 1.30 | 0.926 - 1.69 | 1.05 - 1.56 | | |
| | Nitrate as N | mg/l | 15.6 | 16.0 | 12.7 - 19.0 | 13.7 - 17.9 | Acceptable | SM4500NO3-F |
| | Ortho-phosphate as P | mg/l | | 3.31 | 2.82 - 3.83 | 2.99 - 3.66 | | |
| ***Nutrients - Complex*** | Total phosphorus as P | mg/l | | 4.34 | 3.30 - 5.09 | 3.60 - 4.79 | | |
| | Total kjeldahl nitrogen | mg/l | | 5.78 | 3.97 - 7.51 | 4.56 - 6.92 | | |
| ***Cyanide*** | Cyanide, total | mg/l | | 0.729 | 0.506 - 0.937 | 0.578 - 0.865 | | |
| ***Phenolics*** | Phenolics, total | mg/l | | 0.748 | 0.408 - 1.09 | 0.522 - 0.974 | | |
| ***Grease & Oil*** | Grease & Oil (Gravimetric) | mg/l | | 29.7 | 18.7 - 34.9 | 21.4 - 32.2 | | |
| ***TRC*** | Total Residual Chlorine | mg/l | | 1.33 | 1.05 - 1.61 | 1.14 - 1.52 | | |

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# WP-60 Score and Overall Assessment

**ERA Laboratory Code: C0535-01C EPA ID: CA00112 State ID: 1664 Report Issued: 03/28/00**

| Number of Acceptable Analytes | Number of Reported Analytes | InterLaB™ Score | Overall Assessment |
|---|---|---|---|
| 9 | 13 | 69.2% | Acceptable |

**Scoring Definitions:**

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- The **InterLaB™ Score** is equal to the sum of the Number of Acceptable Analytes divided by the total Number of Reported Analytes.

- The **Overall Assessment:**
  **Excellent** = InterLaB™ Score falls within 95.0% and 100%.
  **Very Good** = InterLaB™ Score falls within 85.0% and 94.9%.
  **Good** = InterLaB™ Score falls within 75.0% and 84.9%.
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# WP-60 Definitions & Study Discussion

**ERA Laboratory Code: C0535-01D EPA ID: CA00112 State ID: 1664** **Report Issued: 03/28/00**

## InterLaB WatR™ Pollution Definitions:

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The **Acceptance Limits** and **Warning Limits** are established per the guidelines contained in the EPA's National Standards for Water Proficiency Testing Criteria Document, December 1998 as applicable.

The **Performance Evaluation:**

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**Not Acceptable =** Reported Value falls outside the Acceptance Limits.

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The **Method Description** is the method the laboratory reported to ERA.

D.L. equals the Detection Limit.

## InterLaB WatR™ Pollution Study Discussion:

ERA WatR™ Pollution Proficiency Testing Study 60 has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and those contained in the National Institute for Standards and Technologies Handbooks 150 and 150-19.

Per the requirements of the USEPA's Criteria Document and the NIST NVLAP Handbooks, the WatR™ Pollution 60 (WP60) results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WP60 standards met the acceptance criteria contained in the US EPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program, Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing, June 1999.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no study anomolies were found and all Acceptance Limits for the InterLaB WatR™ Pollution study, WP60, were calculated based on the US EPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, as applicable.

If you have any questions regarding WatR™ Pollution Study, WP 60, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Roland P. Bruggeman, InterLaB™ Chemist, at 1-800-372-0122.

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|---|---|---|---|---|---|---|---|---|
| ***Minerals*** | Total Solids at 105°C | mg/l | | 244 | 166 - 310 | 190 - 286 | | |
| | Total Dissolved Solids | mg/l | | 228 | 168 - 288 | 188 - 268 | | |
| | Conductivity at 25°C | µmhos | | 315 | 291 - 339 | 299 - 331 | | |
| | Alkalinity as CaCO3 | mg/l | | 55.3 | 49.2 - 61.7 | 51.3 - 59.6 | | |
| | Chloride | mg/l | | 39.5 | 34.8 - 43.6 | 36.3 - 42.2 | | |
| | Fluoride | mg/l | | 2.54 | 2.21 - 2.84 | 2.32 - 2.73 | | |
| | Potassium | mg/l | | 24.5 | 21.1 - 28.0 | 22.2 - 26.8 | | |
| | Sodium | mg/l | | 50.4 | 45.4 - 55.6 | 47.1 - 53.9 | | |
| | Sulfate | mg/l | | 23.7 | 18.7 - 28.1 | 20.2 - 26.6 | | |
| ***pH*** | pH | S.U. | | 9.00 | 8.73 - 9.27 | 8.82 - 9.18 | | |
| ***Hardness*** | Total Suspended Solids | mg/l | | 36.3 | 26.7 - 38.7 | 28.7 - 36.7 | | |
| | Calcium | mg/l | | 42.4 | 37.8 - 48.2 | 39.5 - 46.4 | | |
| | Magnesium | mg/l | | 29.8 | 26.1 - 33.4 | 27.3 - 32.2 | | |
| | Calcium hardness (CaCO3) | mg/l | | 106 | 83.8 - 128 | 91.2 - 121 | | |
| | Total Hardness (CaCO3) | mg/l | | 228 | 208 - 249 | 215 - 242 | | |
| ***Demand*** | BOD | mg/l | | 37.1 | 18.5 - 55.7 | 24.7 - 49.5 | | |
| | CBOD | mg/l | | 32.0 | 14.3 - 49.7 | 20.2 - 43.8 | | |
| | COD | mg/l | | 59.8 | 41.6 - 73.2 | 46.9 - 67.9 | | |
| | TOC | mg/l | | 23.6 | 19.6 - 27.5 | 20.9 - 26.2 | | |
| ***Nutrients - Simple*** | Ammonia as N | mg/l | | 1.30 | 0.926 - 1.69 | 1.05 - 1.56 | | |
| | Nitrate as N | mg/l | | 16.0 | 12.7 - 19.0 | 13.7 - 17.9 | | |
| | Ortho-phosphate as P | mg/l | | 3.31 | 2.82 - 3.83 | 2.99 - 3.66 | | |
| ***Nutrients - Complex*** | Total phosphorus as P | mg/l | | 4.34 | 3.30 - 5.09 | 3.60 - 4.79 | | |
| | Total kjeldahl nitrogen | mg/l | | 5.78 | 3.97 - 7.51 | 4.56 - 6.92 | | |
| ***Cyanide*** | Cyanide, total | mg/l | | 0.729 | 0.506 - 0.937 | 0.578 - 0.865 | | |
| ***Phenolics*** | Phenolics, total | mg/l | | 0.748 | 0.408 - 1.09 | 0.522 - 0.974 | | |
| ***Grease & Oil*** | Grease & Oil (Gravimetric) | mg/l | | 29.7 | 18.7 - 34.9 | 21.4 - 32.2 | | |
| ***TRC*** | Total Residual Chlorine | mg/l | | 1.33 | 1.05 - 1.61 | 1.14 - 1.52 | | |

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# WP-60 Score and Overall Assessment

**ERA Laboratory Code: C0535-01D EPA ID: CA00112 State ID: 1664** **Report Issued: 03/28/00**

| Number of Acceptable Analytes | Number of Reported Analytes | InterLaB™ Score | Overall Assessment |
|---|---|---|---|
| 8 | 11 | 72.7% | Acceptable |

**Scoring Definitions:**

- The **Number of Acceptable Analytes** is equal to any reported values that resulted in an "Acceptable" performance evaluation for the laboratory. An acceptable performance evaluation falls with in the Warning Limits.

- The **Number of Reported Analytes** is equal to any evaluated values reported during this study.

- The **InterLaB™ Score** is equal to the sum of the Number of Acceptable Analytes divided by the total Number of Reported Analytes.

- The **Overall Assessment:**
  **Excellent** = InterLaB™ Score falls within 95.0% and 100%.
  **Very Good** = InterLaB™ Score falls within 85.0% and 94.9%.
  **Good** = InterLaB™ Score falls within 75.0% and 84.9%.
  **Acceptable** = InterLaB™ Score falls within 65.0% and 74.9%.
  **Needs Improvement** = InterLaB™ Score is equal to or below 64.9%.

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# WP-60 Definitions & Study Discussion

**ERA Laboratory Code: C0535-01E EPA ID: CA00112 State ID: 1664 Report Issued: 03/28/00**

## InterLaB WatR™ Pollution Definitions:

The **Reported Value** is the value that the laboratory reported to ERA.

The **ERA Assigned Value** is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. A parameter not added to the standard is given an **Assigned Value** of **"Zero"** per the guidelines contained in the EPA's National Standards for Water Proficiency Testing Criteria Document, December 1998 as applicable.

The **Acceptance Limits** and **Warning Limits** are established per the guidelines contained in the EPA's National Standards for Water Proficiency Testing Criteria Document, December 1998 as applicable.

The **Performance Evaluation:**

**Acceptable =** Reported Value falls within the Acceptance Limits.

**Not Acceptable =** Reported Value falls outside the Acceptance Limits.

**Check for Error =** Reported Value falls within the Acceptance Limits and outside of the Warning Limits.

**No Evaluation =** Reported Values that can not be evaluated.

The **Method Description** is the method the laboratory reported to ERA.

**D.L.** equals the Detection Limit.

## InterLaB WatR™ Pollution Study Discussion:

ERA WatR™ Pollution Proficiency Testing Study 60 has been reviewed by ERA Senior Management and certified compliant with the requirements of the USEPA's National Standards for Water Proficiency Testing Studies Criteria Document, December 1998, and those contained in the National Institute for Standards and Technologies Handbooks 150 and 150-19.

Per the requirements of the USEPA's Criteria Document and the NIST NVLAP Handbooks, the WatR™ Pollution 60 (WP60) results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the WP60 standards met the acceptance criteria contained in the US EPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, and the National Voluntary Laboratory Accreditation Program, Handbook 150-19 for Chemical Calibration for Providers of Proficiency Testing, June 1999.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no study anomolies were found and all Acceptance Limits for the InterLaB WatR™ Pollution study, WP60, were calculated based on the US EPA's National Criteria Document for Water Proficiency Testing Studies, December 1998, as applicable.

If you have any questions regarding WatR™ Pollution Study, WP 60, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Roland P. Bruggeman, InterLaB™ Chemist, at 1-800-372-0122.

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# WP-60 Final Report

**ERA Laboratory Code: C0535-01E EPA ID: CA00112 State ID: 1664** **Report Issued: 03/28/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Warning Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|---|
| ***Minerals*** | Total Solids at 105°C | mg/l | | 244 | 166 - 310 | 190 - 286 | | |
| | Total Dissolved Solids | mg/l | | 228 | 168 - 288 | 188 - 268 | | |
| | Conductivity at 25°C | µmhos | | 315 | 291 - 339 | 299 - 331 | | |
| | Alkalinity as CaCO3 | mg/l | | 55.3 | 49.2 - 61.7 | 51.3 - 59.6 | | |
| | Chloride | mg/l | | 39.5 | 34.8 - 43.6 | 36.3 - 42.2 | | |
| | Fluoride | mg/l | | 2.54 | 2.21 - 2.84 | 2.32 - 2.73 | | |
| | Potassium | mg/l | | 24.5 | 21.1 - 28.0 | 22.2 - 26.8 | | |
| | Sodium | mg/l | | 50.4 | 45.4 - 55.6 | 47.1 - 53.9 | | |
| | Sulfate | mg/l | | 23.7 | 18.7 - 28.1 | 20.2 - 26.6 | | |
| ***pH*** | pH | S.U. | | 9.00 | 8.73 - 9.27 | 8.82 - 9.18 | | |
| ***Hardness*** | Total Suspended Solids | mg/l | | 36.3 | 26.7 - 38.7 | 28.7 - 36.7 | | |
| | Calcium | mg/l | | 42.4 | 37.8 - 48.2 | 39.5 - 46.4 | | |
| | Magnesium | mg/l | | 29.8 | 26.1 - 33.4 | 27.3 - 32.2 | | |
| | Calcium hardness (CaCO3) | mg/l | | 106 | 83.8 - 128 | 91.2 - 121 | | |
| | Total Hardness (CaCO3) | mg/l | | 228 | 208 - 249 | 215 - 242 | | |
| ***Demand*** | BOD | mg/l | | 37.1 | 18.5 - 55.7 | 24.7 - 49.5 | | |
| | CBOD | mg/l | | 32.0 | 14.3 - 49.7 | 20.2 - 43.8 | | |
| | COD | mg/l | | 59.8 | 41.6 - 73.2 | 46.9 - 67.9 | | |
| | TOC | mg/l | | 23.6 | 19.6 - 27.5 | 20.9 - 26.2 | | |
| ***Nutrients - Simple*** | Ammonia as N | mg/l | | 1.30 | 0.926 - 1.69 | 1.05 - 1.56 | | |
| | Nitrate as N | mg/l | | 16.0 | 12.7 - 19.0 | 13.7 - 17.9 | | |
| | Ortho-phosphate as P | mg/l | | 3.31 | 2.82 - 3.83 | 2.99 - 3.66 | | |
| ***Nutrients - Complex*** | Total phosphorus as P | mg/l | | 4.34 | 3.30 - 5.09 | 3.60 - 4.79 | | |
| | Total kjeldahl nitrogen | mg/l | | 5.78 | 3.97 - 7.51 | 4.56 - 6.92 | | |
| ***Cyanide*** | Cyanide, total | mg/l | | 0.729 | 0.506 - 0.937 | 0.578 - 0.865 | | |
| ***Phenolics*** | Phenolics, total | mg/l | | 0.748 | 0.408 - 1.09 | 0.522 - 0.974 | | |
| ***Grease & Oil*** | Grease & Oil (Gravimetric) | mg/l | | 29.7 | 18.7 - 34.9 | 21.4 - 32.2 | | |
| ***TRC*** | Total Residual Chlorine | mg/l | | 1.33 | 1.05 - 1.61 | 1.14 - 1.52 | | |

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# WP-60 Score and Overall Assessment

**ERA Laboratory Code: C0535-01E EPA ID: CA00112 State ID: 1664 Report Issued: 03/28/00**

| Number of Acceptable Analytes | Number of Reported Analytes | InterLaB™ Score | Overall Assessment |
|---|---|---|---|
| 2 | 2 | 100.0% | Excellent |

**Scoring Definitions:**

- The **Number of Acceptable Analytes** is equal to any reported values that resulted in an "Acceptable" performance evaluation for the laboratory. An acceptable performance evaluation falls with in the Warning Limits.

- The **Number of Reported Analytes** is equal to any evaluated values reported during this study.

- The **InterLaB™ Score** is equal to the sum of the Number of Acceptable Analytes divided by the total Number of Reported Analytes.

- The **Overall Assessment:**
  **Excellent** = InterLaB™ Score falls within 95.0% and 100%.
  **Very Good** = InterLaB™ Score falls within 85.0% and 94.9%.
  **Good** = InterLaB™ Score falls within 75.0% and 84.9%.
  **Acceptable** = InterLaB™ Score falls within 65.0% and 74.9%.
  **Needs Improvement** = InterLaB™ Score is equal to or below 64.9%.

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Caltest Analytical Laboratory
EPA code: CA00112


# CORRECTIVE ACTION REPORT
## FOR ERA's InterLab WatR™ Supply WP-60

| Parameters | Reported Value | True Value: | Acceptance Limits |
|---|---|---|---|
| Total Dissolved (EPA 160.1, SM 2540C) Solids | 147 mg/L | 228 | 168 – 288 mg/L |

The analytical and quality control measurements were reviewed and found to acceptable. The glassware used might have been chipped during analysis. All QC criteria for this method were met. Blank was OK, LCS (102% recovery), RPD ( 0%). A single blind sample was analyzed before and after analyzing WP 60 PE sample with acceptable result. Checking of glassware during analysis for chips is now being performed and will be incorporated in the SOP.

| Analyte | Reported Value | True Value: | Acceptance Limits |
|---|---|---|---|
| Chloride (EPA 300.0) | 34.4 mg/L | 39.5 mg/L | 34.8 – 43.6 |

The analytical and quality control measurements and calculations were reviewed and found to be acceptable. Blank was OK, LCS (96% recovery) and RPD was 1.5%. The reported result was 87.1% of the true result which is within Caltest's QC criteria (80-120%) for this procedure.

| Analyte | Reported Value | True Value: | Acceptance Limits |
|---|---|---|---|
| Oil and Grease (EPA 1664) | 36.1 mg/L | 29.7 mg/L | 18.7 – 34.9 |

The analytical and analytical measurements and calculations were reviewed and found to be acceptable. Possibly water or sodium sulfate got through the filter. Analytical balance used was calibrated. Blank was OK, LCS (108% recovery), LCSD (102% recovery) and RPD (5.25%).Checking of filters before and during analysis is now being performed and will be incorporated in the SOP. A single blind sample was analyzed with acceptable result.

InterLaB Soil Proficiency Testing Program
by Environmental Resource Associates

Soil-29 Performance Evaluation

Report Issue date: 04/10/00

| | |
|---|---|
| Total Reported Analytes: | 135 |
| Acceptable Analytes: | 135 |
| InterLaB Score: | 100% |
| Overall Assessment: | Excellent |

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April 10, 2000

Carmelita Oliveros.
Caltest Analytical Laboratory
1885 North Kelly Road
Napa, CA 94558

Dear Carmelita Oliveros. :

Enclosed is the ERA final statistical summary for the InterLaB™ Soil, SOIL-29 Proficiency Testing (PT) Program. The opening date of the InterLaB™ Soil, SOIL-29 study was 01/21/00 and the closing date was 03/06/00. This concludes the SOIL-29 study.

Your requested final report for SOIL-29 has not been sent to the State of California. ERA is currently compiling your results to the evaluation criteria required by the State of California. The final report you have received does not contain the California evaluation criteria. A final report for the State of California will be mailed to both you and the State of California prior to the deadline for the submittal.

ERA recommends that your lab demonstrates corrective action for any parameter that is reported as a "Not Acceptable". Corrective action is demonstrated by identifying root causes of error, alleviating the error, and successfully analyzing a Quality Control Standard. An excellent way to demonstrate the effectiveness of your corrective action is to participate in the next ERA InterLaB™ Soil round robin study. For your convenience ERA has an InterLaB™ Soil round robin study tentatively scheduled to begin shipping the second week of April. If you would like to place an order for the next available study, please contact me at 1-800-372-0122.

Thank you for your participation in the InterLaB™ Soil, SOIL-29, Performance Evaluation Program. If you have any questions, please do not hesitate to call Roland P. Bruggeman, InterLaB™ Chemist, or me.

Sincerely,

Shawn Kassner

Shawn Kassner
InterLaB™ Administrator

attachments
smk

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April 10, 2000

Carmelita Oliveros.
Caltest Analytical Laboratory
1885 North Kelly Road
Napa, CA 94558

Dear Carmelita Oliveros. :

I would like to introduce myself; I am Curtis J. Wood, Quality Assurance Manager for ERA. I have been a chemist at ERA for eleven years and have experience in every aspect of ERA's PT programs. My job is to ensure that our PT programs meet all National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, July 1999 program requirements as well as those of our ISO 9001 Registered Quality System. In addition, I believe a critical aspect of overall quality is responsive and professional customer service.

Over the past month we have conducted an extensive review of all aspects of our InterLaB™ program. I am sure you can appreciate that complying with the myriad of federal and state requirements presents a significant challenge. Our review as well as our policy of making our PT programs compliant with imminent new requirements caused us to make some changes effective with the SOIL-29 reports. We apologize for the delay that these changes caused.

Thank you for participating in ERA's SOIL-29 Soil Proficiency Testing study. All aspects of the SOIL-29 study, from standard manufacture to final report generation, were completed by ERA in accordance with the NELAC, Proficiency Testing Program, Chapter 2, July 1999. ERA has reviewed all of the data that is contained in this report and has made every possible effort to make it complete, accurate and compliant. However, if you find anything in this report that you feel is incomplete or inaccurate or have any quality related issues, please call me directly at 1-800-372-0122.

Sincerely,

Curtis J. Wood
Quality Assurance Manager

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# SOIL-29 Definitions & Study Discussion

**ERA Laboratory Code: C0535-01 EPA ID: CA00112 State ID: 1664 Report Issued: 04/10/00**

## InterLaB™ Soil Study Definitions:

The **Reported Value** is the value that the laboratory reported to ERA.

The **ERA Assigned Value** for the **Organic Proficiency Testing Standards** is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable. The **ERA Assigned Value** for the **Inorganic Proficiency Testing Standards** is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation plus the concentration of the analytes found in the background soil as determined by the applicable EPA methodoligies. A parameter not added to a standard is given an **Assigned Value** of less than a minimum verified concentration as determined in the background soil by applicable EPA methodoligies.

The **Acceptance Limits** are established per the guidelines contained in the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, July 1999. The Acceptance Limits are equal to the study mean plus or minus three standard deviations as determined by robust statistical analysis.

The **Performance Evaluation:**

**Acceptable =** Reported Value falls within the Acceptance Limits.

**Not Acceptable =** Reported Value falls outside the Acceptance Limits.

**No Evaluation =** Reported Values that can not be evaluated.

The **Method Description** and **Prep Code** are the methods the laboratory reported to ERA.

**D.L.** equals the Detection Limit.

## InterLaB™ Soil Study Discussion:

ERA Soil Proficiency Testing Study 29 has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, July 1999.

Per the requirements of the NELAC Proficiency Testing Program, the SOIL 29 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the SOIL 29 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, July 1999.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no study anomolies were found and all Acceptance Limits for the InterLaB™ Soil study, SOIL 29, were calculated based on the NELAC, Proficiency Testing Program, Chapter 2, July 1999, as applicable.

If you have any questions regarding Soil Study, SOIL 29, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Curtis Wood, Quality Assurance Manager, at 1-800-372-0122.

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# SOIL-29 Final Report

**ERA Laboratory Code: C0535-01** **EPA ID: CA00112** **State ID: 1664** **Report Issued: 04/10/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Performance Evaluation | Prep Code | Method Description |
|---|---|---|---|---|---|---|---|---|
| *Trace Metals - in Soil* | Aluminum | mg/Kg | 5720 | 9690 | 430 - 16100 | Acceptable | A | 6010B |
| | Antimony | mg/Kg | 6.65 | 87.3 | D.L. - 87.3 | Acceptable | A | 6010B |
| | Arsenic | mg/Kg | 179 | 221 | 123 - 249 | Acceptable | A | 6010B |
| | Barium | mg/Kg | 160 | 189 | 115 - 241 | Acceptable | A | 6010B |
| | Beryllium | mg/Kg | 47.4 | 57.9 | 32.7 - 58.3 | Acceptable | A | 6010B |
| | Boron | mg/Kg | 34.4 | 83.0 | 12.9 - 90.9 | Acceptable | A | 6010B |
| | Cadmium | mg/Kg | 64.9 | 73.9 | 47.3 - 82.3 | Acceptable | A | 6010B |
| | Calcium | mg/Kg | 10700 | 11300 | 8150 - 13100 | Acceptable | A | 6010B |
| | Chromium | mg/Kg | 145 | 165 | 102 - 186 | Acceptable | A | 6010B |
| | Cobalt | mg/Kg | 216 | 257 | 153 - 275 | Acceptable | A | 6010B |
| | Copper | mg/Kg | 86.5 | 101 | 66.1 - 119 | Acceptable | A | 6010B |
| | Iron | mg/Kg | 9480 | 14200 | 3510 - 21700 | Acceptable | A | 6010B |
| | Lead | mg/Kg | 121 | 134 | 83.8 - 158 | Acceptable | A | 6010B |
| | Magnesium | mg/Kg | 2480 | 3100 | 1510 - 3970 | Acceptable | A | 6010B |
| | Manganese | mg/Kg | 376 | 416 | 281 - 501 | Acceptable | A | 6010B |
| | Mercury | mg/Kg | 1.44 | 1.54 | 0.683 - 2.24 | Acceptable | A | 7471A |
| | Molybdenum | mg/Kg | 38.9 | 67.4 | 29.9 - 67.4 | Acceptable | A | 6010B |
| | Nickel | mg/Kg | 73.6 | 75.8 | 54.9 - 95.4 | Acceptable | A | 6010B |
| | Potassium | mg/Kg | 2720 | 3980 | 1760 - 5450 | Acceptable | A | 6010B |
| | Selenium | mg/Kg | 153 | 174 | 99.8 - 204 | Acceptable | A | 6010B |
| | Silver | mg/Kg | 64.9 | 106 | 58.8 - 121 | Acceptable | A | 6010B |
| | Sodium | mg/Kg | 999 | 1090 | 629 - 1390 | Acceptable | A | 6010B |
| | Strontium | mg/Kg | | 130 | 75.8 - 168 | | | |
| | Thallium | mg/Kg | | 93.2 | 48.3 - 116 | | | |
| | Tin | mg/Kg | | 194 | 44.1 - 194 | | | |
| | Titanium | mg/Kg | | 278 | D.L. - 563 | | | |
| | Vanadium | mg/Kg | 158 | 193 | 100 - 244 | Acceptable | A | 6010B |
| | Zinc | mg/Kg | 259 | 304 | 182 - 366 | Acceptable | A | 6010B |
| *Cyanide - in Soil* | Cyanide, total | mg/Kg | 89.3 | 312 | D.L. - 580 | Acceptable | | 9010A |

**Prep Codes:**

- **A** - Hot plate with HNO3, HCl and H2O2. (SW-846 Method 3050 for FLAA & ICP-OES)
- **B** - Hot plate with HNO3 and H2O2. (SW-846 Method 3050 for GFAA & ICP-MS)
- **C** - SW-846 Method 3050b optional section 7.5.
- **D** - Microwave with HNO3 only. (SW-846 Method 3051)
- **E** - Microwave with HNO3 and HCl. (SW-846 method 3051 modified)
- **F** - Other if not listed above.

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# SOIL-29 Final Report

**ERA Laboratory Code: C0535-01** **EPA ID: CA00112** **State ID: 1664** **Report Issued: 04/10/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|
| ***Semivolatiles Base / Neutrals and Acids in Soil*** | Acenaphthene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Acenaphthylene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Aniline | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Anthracene | µg/Kg | 4260 | 5460 | 1630 - 6730 | Acceptable | 8270C |
| | Benzidine | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Benzoic acid | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Benzo(a)anthracene | µg/Kg | 1010 | 1160 | 516 - 1430 | Acceptable | 8270C |
| | Benzo(b)fluoranthene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Benzo(k)fluoranthene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Benzo(g,h,i)perylene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Benzo(a)pyrene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Benzyl alcohol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 4-Bromophenyl-phenylether | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Butylbenzylphthalate | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Carbazole | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 4-Chloroaniline | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | bis(2-Chloroethoxy)methane | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | bis(2-Chloroethyl)ether | µg/Kg | 6990 | 9910 | D.L. - 12800 | Acceptable | 8270C |
| | 4-Chloro-3-methylphenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 1-Chloronaphthalene | µg/Kg | | < 300 | | | |
| | 2-Chloronaphthalene | µg/Kg | | < 300 | | | |
| | 2-Chlorophenol | µg/Kg | 7160 | 9590 | 670 - 10900 | Acceptable | 8270C |
| | 4-Chlorophenyl-phenylether | µg/Kg | 5060 | 5650 | 2020 - 7340 | Acceptable | 8270C |
| | 2,2'-oxybis(1-Chloropropane) | µg/Kg | | < 300 | | | |
| | Chrysene | µg/Kg | 3240 | 3860 | 1400 - 5020 | Acceptable | 8270C |
| | Dibenz(a,h)anthracene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Dibenzofuran | µg/Kg | 6480 | 7110 | 2450 - 8750 | Acceptable | 8270C |
| | Di-n-butylphthalate | µg/Kg | 2700 | 3270 | 996 - 4580 | Acceptable | 8270C |
| | 1,2-Dichlorobenzene | µg/Kg | 6990 | 10000 | 30.0 - 12200 | Acceptable | 8270C |
| | 1,3-Dichlorobenzene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 1,4-Dichlorobenzene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 3,3'-Dichlorobenzidine | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 2,4-Dichlorophenol | µg/Kg | 6230 | 7950 | 1450 - 8950 | Acceptable | 8270C |
| | 2,6-Dichlorophenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Diethylphthalate | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 2,4-Dimethylphenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Dimethylphthalate | µg/Kg | 7520 | 8520 | 760 - 11900 | Acceptable | 8270C |
| | 4,6-Dinitro-2-methylphenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 2,4-Dinitrophenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 2,4-Dinitrotoluene | µg/Kg | 5490 | 6820 | D.L. - 12600 | Acceptable | 8270C |
| | 2,6-Dinitrotoluene | µg/Kg | 7620 | 9050 | 3290 - 10700 | Acceptable | 8270C |
| | Di-n-octylphthalate | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | bis(2-Ethylhexyl)phthalate | µg/Kg | 7020 | 7770 | 2840 - 10500 | Acceptable | 8270C |
| | Fluoranthene | µg/Kg | 3920 | 4460 | 1760 - 5920 | Acceptable | 8270C |
| | Fluorene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |

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# SOIL-29 Final Report

**ERA Laboratory Code: C0535-01 EPA ID: CA00112 State ID: 1664 Report Issued: 04/10/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|
| ***Semivolatiles Base / Neutrals and Acids in Soil (cont.)*** | Hexachlorobenzene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Hexachlorobutadiene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Hexachlorocyclopentadiene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Hexachloroethane | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Indeno(1,2,3-cd)pyrene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Isophorone | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 2-Methylnaphthalene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 2-Methylphenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 3-Methylphenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 4-Methylphenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Naphthalene | µg/Kg | 2320 | 3150 | 491 - 3930 | Acceptable | 8270C |
| | 2-Nitroaniline | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 3-Nitroaniline | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 4-Nitroaniline | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Nitrobenzene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 2-Nitrophenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 3-Nitrophenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 4-Nitrophenol | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | N-Nitrosodimethylamine | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | N-Nitrosodiphenylamine | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | N-Nitroso-di-n-propylamine | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Pentachlorophenol | µg/Kg | 6180 | 8120 | 10.0 - 10200 | Acceptable | 8270C |
| | Phenanthrene | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | Phenol | µg/Kg | 4530 | 6460 | 130 - 7810 | Acceptable | 8270C |
| | Pyrene | µg/Kg | 3960 | 4520 | 1600 - 5980 | Acceptable | 8270C |
| | Pyridine | µg/Kg | 0.00 | < 300 | | Acceptable | 8270C |
| | 1,2,4-Trichlorobenzene | µg/Kg | 2170 | 2880 | 541 - 3440 | Acceptable | 8270C |
| | 2,4,5-Trichlorophenol | µg/Kg | 4180 | 4830 | 584 - 6360 | Acceptable | 8270C |
| | 2,4,6-Trichlorophenol | µg/Kg | 3720 | 4500 | 733 - 5590 | Acceptable | 8270C |

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# SOIL-29 Final Report

**ERA Laboratory Code: C0535-01** **EPA ID: CA00112** **State ID: 1664** **Report Issued: 04/10/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|
| ***Semivolatiles Pesticides in Soil*** | Aldrin | µg/Kg | 108 | 122 | 11.8 - 200 | Acceptable | 8081A |
| | alpha-BHC | µg/Kg | 215 | 268 | 24.6 - 401 | Acceptable | 8081A |
| | beta-BHC | µg/Kg | 136 | 180 | D.L. - 343 | Acceptable | 8081A |
| | delta-BHC | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| | gamma-BHC(Lindane) | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| | alpha-Chlordane | µg/Kg | 264 | 285 | 63.2 - 403 | Acceptable | 8081A |
| | gamma-Chlordane | µg/Kg | 195 | 225 | 49.4 - 327 | Acceptable | 8081A |
| | 4,4'-DDD | µg/Kg | 248 | 284 | 58.5 - 428 | Acceptable | 8081A |
| | 4,4'-DDE | µg/Kg | 179 | 195 | 47.4 - 289 | Acceptable | 8081A |
| | 4,4'-DDT | µg/Kg | 102 | 106 | 28.9 - 151 | Acceptable | 8081A |
| | Dieldrin | µg/Kg | 146 | 165 | 23.5 - 255 | Acceptable | 8081A |
| | Endrin | µg/Kg | 186 | 199 | 49.8 - 316 | Acceptable | 8081A |
| | Endrin aldehyde | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| | Endrin ketone | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| | Endosulfan I | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| | Endosulfan II | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| | Endosulfan sulfate | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| | Heptachlor | µg/Kg | 212 | 230 | 46.2 - 332 | Acceptable | 8081A |
| | Heptachlor epoxide | µg/Kg | 232 | 265 | 41.0 - 389 | Acceptable | 8081A |
| | Methoxychlor | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| | Toxaphene | µg/Kg | 0.00 | < 5.00 | | Acceptable | 8081A |
| ***PCBs in Soil*** | Aroclor 1248 | mg/Kg | 163 | 202 | 16.3 - 310 | Acceptable | 8082 |
| | PCB Aroclor Identity | | 1248 | 1248 | 1248 | Acceptable | 8082 |

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# SOIL-29 Final Report

**ERA Laboratory Code: C0535-01** **EPA ID: CA00112** **State ID: 1664** **Report Issued: 04/10/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|
| ***PAHs - in Soil*** | Acenaphthene | µg/Kg | 435 | 788 | D.L. - 1220 | Acceptable | 8310 |
| | Acenaphthylene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |
| | Anthracene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |
| | Benzo(a)anthracene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |
| | Benzo(b)fluoranthene | µg/Kg | 299 | 386 | 139 - 463 | Acceptable | 8310 |
| | Benzo(k)fluoranthene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |
| | Benzo(g,h,i)perylene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |
| | Benzo(a)pyrene | µg/Kg | 241 | 316 | 75.7 - 394 | Acceptable | 8310 |
| | Chrysene | µg/Kg | 230 | 263 | 41.8 - 440 | Acceptable | 8310 |
| | Dibenz(a,h)anthracene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |
| | Fluoranthene | µg/Kg | 666 | 787 | 256 - 1050 | Acceptable | 8310 |
| | Fluorene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |
| | Indeno(1,2,3-cd)pyrene | µg/Kg | 263 | 371 | 41.0 - 527 | Acceptable | 8310 |
| | Naphthalene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |
| | Phenanthrene | µg/Kg | 656 | 895 | 196 - 1290 | Acceptable | 8310 |
| | Pyrene | µg/Kg | 0.00 | < 10.0 | | Acceptable | 8310 |

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# SOIL-29 Score and Overall Assessment

**ERA Laboratory Code: C0535-01** **EPA ID: CA00112** **State ID: 1664** **Report Issued: 04/10/00**

| Number of Acceptable Analytes | Number of Reported Analytes | InterLaB™ Score | Overall Assessment |
|---|---|---|---|
| 135 | 135 | 100.0% | Excellent |

**Scoring Definitions:**

- The **Number of Acceptable Analytes** is equal to any reported values that resulted in an "Acceptable" performance evaluation for the laboratory. An acceptable performance evaluation falls with in the Acceptance Limits.
- The **Number of Reported Analytes** is equal to any evaluated values reported during this study.
- The **InterLaB™ Score** is equal to the sum of the Number of Acceptable Analytes divided by the total Number of Reported Analytes.
- The **Overall Assessment:**
  **Excellent** = InterLaB™ Score falls within 95.0% and 100%.
  **Very Good** = InterLaB™ Score falls within 85.0% and 94.9%.
  **Good** = InterLaB™ Score falls within 75.0% and 84.9%.
  **Acceptalbe** = InterLaB™ Score falls within 65.0% and 74.9%.
  **Needs Improvement** = InterLaB™ Score is equal to or below 64.9%.

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InterLaBUnderground Storage Tank, Proficiency Testing Program
by Environmental Resource Associates

UST-29 Performance Evaluation

Report Issue date: 04/26/00

| | |
|---|---|
| Total Reported Analytes: | 2 |
| Acceptable Analytes: | 2 |
| InterLaB Score: | 100% |
| Overall Assessment: | Excellent |

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The Industry Standard™

April 26, 2000

Carmelita P. Oliveros
Caltest Analytical Laboratory
1885 North Kelly Road
Napa, CA 94558

Dear Carmelita P. Oliveros :

Enclosed is the ERA final statistical summary for the InterLaB™ Underground Storage Tank, UST-29 Proficiency Testing (PT) Program. The opening date of the InterLaB™ UST-29 study was 02/11/00 and the closing date was 03/27/00. This concludes the UST-29 study.

Your requested final report for UST-29 has not been sent to the State of California. ERA is currently compiling your results to the evaluation criteria required by the State of California. The final report you have received does not contain the California evaluation criteria. A final report for the State of California will be mailed to both you and the State of California prior to the deadline for the submittal.

ERA recommends that your lab demonstrates corrective action for any parameter that is reported as a "Not Acceptable". Corrective action is demonstrated by identifying root causes of error, alleviating the error, and successfully analyzing a Quality Control Standard. An excellent way to demonstrate the effectiveness of your corrective action is to participate in the next ERA InterLaB™ Underground Storage Tank round robin study. For your convenience ERA has an InterLaB Underground Storage Tank round robin study tentatively scheduled to begin shipping the first week of May. If you would like to place an order for the next available study, please contact me at 1-800-372-0122.

Thank you for your participation in the InterLaB™ Underground Storage Tank, UST-29, Performance Evaluation Program. If you have any questions, please do not hesitate to call Roland P. Bruggeman, InterLaB™ Chemist, or me.

Sincerely,

Shawn Kassner

Shawn Kassner
InterLaB™ Administrator

attachments
smk

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RESOURCE ASSOCIATES®
The Industry Standard™

April 26, 2000

Carmelita P. Oliveros
Caltest Analytical Laboratory
1885 North Kelly Road
Napa, CA 94558

Dear Carmelita P. Oliveros :

I would like to introduce myself; I am Curtis J. Wood, Quality Assurance Manager for ERA. I have been a chemist at ERA for eleven years and have experience in every aspect of ERA's PT programs. My job is to ensure that our PT programs meet all National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, July 1999 program requirements as well as those of our ISO 9001 Registered Quality System. In addition, I believe a critical aspect of overall quality is responsive and professional customer service.

Over the past month we have conducted an extensive review of all aspects of our InterLaB™ program. I am sure you can appreciate that complying with the myriad of federal and state requirements presents a significant challenge. Our review as well as our policy of making our PT programs compliant with imminent new requirements caused us to make some changes effective with the UST-29 reports. We apologize for the delay that these changes caused.

Thank you for participating in ERA's UST-29 Soil Proficiency Testing study. All aspects of the UST-29 study, from standard manufacture to final report generation, were completed by ERA in accordance with the NELAC, Proficiency Testing Program, Chapter 2, July 1999. ERA has reviewed all of the data that is contained in this report and has made every possible effort to make it complete, accurate and compliant. However, if you find anything in this report that you feel is incomplete or inaccurate or have any quality related issues, please call me directly at 1-800-372-0122.

Sincerely,

Curtis J. Wood
Quality Assurance Manager

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# UST-29 Definitions & Study Discussion

**ERA Laboratory Code: C0535-01 EPA ID: CA00112 State ID: Report Issued: 04/26/00**

## InterLaB™ UST Study Definitions:

The Reported Value is the value that the laboratory reported to ERA.

The ERA Assigned Value for the Proficiency Testing Standards is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation as applicable.

The Acceptance Limits are established per the guidelines contained in the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, July 1999. The Acceptance Limits are equal to the study mean plus or minus three standard deviations as determined by robust statistical analysis.

The Performance Evaluation:

Acceptable = Reported Value falls within the Acceptance Limits.

Not Acceptable = Reported Value falls outside the Acceptance Limits.

No Evaluation = Reported Value that can not be evaluated.

The Method Description is the method the laboratory reported to ERA.

D.L. equals the Detection Limit.

## InterLaB™ UST Study Discussion:

ERA UST Proficiency Testing Study 29 has been reviewed by ERA Senior Management and certified compliant with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC), Proficiency Testing Program, Chapter 2, July 1999.

Per the requirements of the NELAC Proficiency Testing Program, the UST 29 results were examined for any study anomalies. A full review of all homogeneity, stability, and accuracy verification data was completed. All analytical verification data for all analytes in the UST 29 standards met the acceptance criteria contained in the NELAC Proficiency Testing Program, Chapter 2, July 1999.

The data submitted by participating laboratories was also examined for study anomalies. Based upon ERA's review of the data, no study anomolies were found and all Acceptance Limits for the InterLaB™ UST study, UST 29, were calculated based on the NELAC, Proficiency Testing Program, Chapter 2, July 1999, as applicable.

If you have any questions regarding UST Study, UST 29, please contact Shawn Kassner, InterLaB™ Program Coordinator, or Curtis Wood, Quality Assurance Manager, at 1-800-372-0122.

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# UST-29 Final Report

**ERA Laboratory Code: C0535-01** **EPA ID: CA00112** **State ID:** **Report Issued: 04/26/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|
| ***Gasoline in Soil*** | Unleaded Gasoline | mg/Kg | 939 | 991 | 53.0 - 1700 | Acceptable | EPA 8015 |
| | Benzene | mg/Kg | | 4.83 | D.L. - 16.3 | | |
| | Ethylbenzene | mg/Kg | | 18.7 | 6.16 - 39.7 | | |
| | Toluene | mg/Kg | | 52.8 | D.L. - 137 | | |
| | Xylenes, total | mg/Kg | | 102 | 27.1 - 176 | | |
| ***Diesel in Soil*** | No. 2 Diesel | mg/Kg | 914 | 1210 | 284 - 1780 | Acceptable | EPA8015 |
| ***TPH in Soil*** | TPH (gravimetric) | mg/Kg | | 1860 | 506 - 3240 | | |
| | TPH (IR) | mg/Kg | | 2230 | 606 - 3880 | | |
| ***BTEX in Soil*** | Benzene | µg/Kg | | 128 | 56.2 - 186 | | |
| | Ethylbenzene | µg/Kg | | 54.9 | 24.6 - 87.0 | | |
| | Toluene | µg/Kg | | 153 | 59.0 - 215 | | |
| | Xylenes, total | µg/Kg | | 510 | 241 - 769 | | |
| ***MTBE in Soil*** | t-Butylmethylether (MTBE) | µg/Kg | | 32.5 | 6.52 - 60.3 | | |
| ***Gasoline in Water*** | Unleaded Gasoline | µg/l | | 3780 | 840 - 8280 | | |
| | Benzene | µg/l | | 40.6 | 14.7 - 71.3 | | |
| | Ethylbenzene | µg/l | | 86.8 | 53.8 - 157 | | |
| | Toluene | µg/l | | 289 | 159 - 529 | | |
| | Xylenes, total | µg/l | | 469 | 227 - 616 | | |
| ***Diesel in Water*** | No. 2 Diesel | µg/l | | 1720 | D.L. - 3010 | | |
| ***TPH in Water*** | TPH (gravimetric) | mg/bttl | | 175 | 79.3 - 349 | | |
| | TPH (IR) | mg/bttl | | 210 | 95.1 - 419 | | |
| ***BTEX in Water*** | Benzene | µg/l | | 103 | 46.5 - 146 | | |
| | Ethylbenzene | µg/l | | 120 | 44.7 - 177 | | |
| | Toluene | µg/l | | 67.6 | 25.2 - 92.4 | | |
| | Xylenes, total | µg/l | | 306 | 120 - 444 | | |
| ***MTBE in Water*** | t-Butylmethylether (MTBE) | µg/l | | 62.1 | 35.1 - 86.7 | | |

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# UST-29 Score and Overall Assessment

**ERA Laboratory Code: C0535-01** **EPA ID: CA00112** **State ID:** **Report Issued: 04/26/00**

| Number of Acceptable Analytes | Number of Reported Analytes | InterLaB™ Score | Overall Assessment |
|---|---|---|---|
| 2 | 2 | 100.0% | Excellent |

**Scoring Definitions:**

- The **Number of Acceptable Analytes** is equal to any reported values that resulted in an "Acceptable" performance evaluation for the laboratory.

- The **Number of Reported Analytes** is equal to any evaluated values reported during this study.

- The **InterLaB™ Score** is equal to the sum of the Number of Acceptable Analytes divided by the total Number of Reported Analytes.

- The **Overall Assessment:**
  **Excellent =** InterLaB™ Score falls within 95.0% and 100%.
  **Very Good =** InterLaB™ Score falls within 85.0% and 94.9%.
  **Good =** InterLaB™ Score falls within 75.0% and 84.9%.
  **Acceptalbe =** InterLaB™ Score falls within 65.0% and 74.9%.
  **Needs Improvement =** InterLaB™ Score is equal to or below 64.9%.

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InterLaB Soil Proficiency Testing Program
by Environmental Resource Associates

Soil-24 Performance Evaluation

Report Issue date: 12/4/98

ENVIRONMENTAL
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December 4, 1998

Carmelita Oliveros
Caltest Analytical Laboratory
1885 North Kelly Road
Napa, CA 94558

Dear Carmelita,

Enclosed is the ERA final statistical summary for the InterLaB™ Soil, Soil-24, Performance Evaluation Program. This concludes the Soil-24 study. For any parameter in which the performance evaluation is reported as a "Check For Error" or "Not Acceptable", we recommend that your lab demonstrates corrective action.

Demonstrating corrective action is accomplished by first identifying what caused the error, taking the necessary steps to correct the problem and then demonstrating that your actions are effective by analyzing a Quality Control Standard. ERA's next InterLaB™ Soil, Soil-25, is tentatively scheduled to begin on January 6, 1999 and is an excellent way to demonstrate the effectiveness of your corrective action. If you would like to place an order for the Soil-25 study, please contact the order department or me.

Thank you for your participation in the InterLaB™ Soil, Soil-24, Performance Evaluation Program. If you have any questions, please do not hesitate to call me at 1-800-372-0122.

Sincerely,

Shawn Kassner

Shawn Kassner
InterLaB™ Administrator

smk
attachments

5540 MARSHALL STREET, ARVADA, COLORADO 80002
TEL (303) 431-8454 FAX (303) 421-0159

ENVIRONMENTAL RESOURCE ASSOCIATES

InterLaB™ Soil
Statistical Summary

# Trace Metals
# SOIL-24
## Method 3050 FLAA/ICP-AES Digestion

**Laboratory Code:** C0535-01 **Report Issued:** 12/1/98

| Parameter | Reported Value (mg/Kg) | ERA Made To Value (mg/Kg) | Mean Recovery (mg/Kg) | n | Acceptance Limits (mg/Kg) | Warning Limits (mg/Kg) | Performance Evaluation |
|---|---|---|---|---|---|---|---|
| Aluminum | 4650 | 56300 | 5250 | 23 | 2090 - 8410 | 3140 - 7360 | Acceptable |
| Antimony | 24.4 | 85.2 | 33.0 | 18 | D.L. - 73.7 | 5.87 - 60.2 | Acceptable |
| Arsenic | 94.6 | 107 | 93.9 | 14 | 71.2 - 117 | 78.7 - 109 | Acceptable |
| Barium | 338 | 1110 | 330 | 22 | 222 - 437 | 258 - 401 | Acceptable |
| Beryllium | 40.6 | 47.6 | 42.7 | 15 | 33.7 - 51.7 | 36.7 - 48.7 | Acceptable |
| Boron | 61.4 | 111 | 71.8 | 17 | 30.9 - 113 | 44.5 - 99.0 | Acceptable |
| Cadmium | 94.3 | 107 | 97.2 | 23 | 75.1 - 119 | 82.5 - 112 | Acceptable |
| Calcium | 1180 | 9630 | 1320 | 22 | 857 - 1780 | 1010 - 1630 | Acceptable |
| Chromium | 47.3 | 79.4 | 46.0 | 22 | 30.2 - 61.7 | 35.5 - 56.5 | Acceptable |
| Cobalt | 115 | 131 | 120 | 18 | 86.4 - 154 | 97.7 - 143 | Acceptable |
| Copper | 143 | 166 | 147 | 22 | 126 - 168 | 133 - 161 | Acceptable |
| Iron | 7370 | 21700 | 10200 | 21 | 3770 - 16600 | 5910 - 14500 | Acceptable |
| Lead | 135 | 158 | 135 | 22 | 92.6 - 177 | 107 - 163 | Acceptable |
| Magnesium | 2160 | 3340 | 2340 | 21 | 1790 - 2890 | 1970 - 2710 | Acceptable |
| Manganese | 144 | 366 | 166 | 23 | 107 - 224 | 127 - 205 | Acceptable |
| Molybdenum | 37.6 | 44.6 | 40.4 | 16 | 26.7 - 54.1 | 31.3 - 49.5 | Acceptable |
| Nickel | 135 | 157 | 138 | 10 | 124 - 152 | 128 - 147 | Acceptable |
| Potassium | 1300 | 3170 | 1480 | 21 | 769 - 2190 | 1010 - 1950 | Acceptable |
| Selenium | 94.4 | 110 | 96.0 | 17 | 64.9 - 127 | 75.3 - 117 | Acceptable |
| Silver | 83.8 | 99.7 | 86.7 | 20 | 61.7 - 112 | 70.0 - 103 | Acceptable |
| Sodium | 729 | 15400 | 845 | 21 | 374 - 1320 | 531 - 1160 | Acceptable |
| Strontium | 116 | 293 | 102 | 13 | 76.9 - 127 | 85.2 - 119 | Acceptable |

ENVIRONMENTAL RESOURCE ASSOCIATES

InterLaB™ Soil Statistical Summary

# Trace Metals
# SOIL-24

## Method 3050 FLAA/ICP-AES Digestion

**Page 2**

**Laboratory Code:** C0535-01

**Report Issued:** 12/3/98

| Parameter | Reported Value (mg/Kg) | ERA Certified Value (mg/Kg) | Mean Recovery (mg/Kg) | n | Acceptance Limits (mg/Kg) | Warning Limits (mg/Kg) | Performance Evaluation |
|---|---|---|---|---|---|---|---|
| Thallium | 50.6 | 81.3 | 45.7 | 10 | 11.9 - 79.5 | 23.2 - 68.3 | Acceptable |
| Tin | 109 | 135 | 140 | 14 | 54.4 - 226 | 83.0 - 197 | Acceptable |
| Titanium | 266 | 2140 | 319 | 17 | 108 - 530 | 179 - 460 | Acceptable |
| Vanadium | 59.1 | 97.9 | 65.1 | 19 | 38.6 - 91.6 | 47.4 - 82.8 | Acceptable |
| Zinc | 69.5 | 112 | 75.0 | 23 | 56.1 - 93.9 | 62.4 - 87.6 | Acceptable |

**Definitions:**

- The ERA Certified Value is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation.
- The Mean Recovery has been determined from all data reported to ERA for the parameter during this study.
- The Acceptance Limits are equal to the Mean Recovery plus and minus 3.0 standard deviations.
- The Warning Limits are equal to the Mean Recovery plus and minus 2.0 standard deviations.
- The Performance Evaluation:

  Acceptable = Reported Result falls within the Warning Limits.

  Not Acceptable = Reported Result falls outside of the Acceptance Limits.

  Check for Error = Reported Result falls within the Acceptance Limits and outside of the Warning Limits.
- n = number of data points used to calculate Mean Recovery, Acceptance and Warning Limits.

**Additional Comments:**

D.L. - Detection Limit

ENVIRONMENTAL RESOURCE ASSOCIATES

InterLaB™ Soil
Statistical Summary

# Trace Metals
# SOIL-24
## Misc

**Laboratory Code:** C0535-01 **Report Issued:** 12/1/98

| Parameter | Reported Value (mg/Kg) | ERA Made To Value (mg/Kg) | Mean Recovery (mg/Kg) | n | Acceptance Limits (mg/Kg) | Warning Limits (mg/Kg) | Performance Evaluation |
|---|---|---|---|---|---|---|---|
| Mercury | 1.99 | 3.25 | 2.17 | 22 | 0.979 - 3.36 | 1.38 - 2.97 | Acceptable |

**Definitions:**

- The ERA Certified Value is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation.
- The Mean Recovery has been determined from all data reported to ERA for the parameter during this study.
- The Acceptance Limits are equal to the Mean Recovery plus and minus 3.0 standard deviations.
- The Warning Limits are equal to the Mean Recovery plus and minus 2.0 standard deviations.
- The Performance Evaluation:
  - Acceptable = Reported Result falls within the Warning Limits.
  - Not Acceptable = Reported Result falls outside of the Acceptance Limits.
  - Check for Error = Reported Result falls within the Acceptance Limits and outside of the Warning Limits.
- n = number of data points used to calculate Mean Recovery, Acceptance and Warning Limits.

**Additional Comments:**

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InterLaB™ Soil Statistical Summary

# Cyanide
# SOIL-24

**Laboratory Code:** C0635-01

**Report Issued:** 12/1/98

| Parameter | Reported Value (mg/Kg) | ERA Made To Value (mg/Kg) | Mean Recovery (mg/Kg) | n | Acceptance Limits (mg/Kg) | Warning Limits (mg/Kg) | Performance Evaluation |
|---|---|---|---|---|---|---|---|
| Cyanide, total | 76.5 | 100 | 82.5 | 13 | 37.8 - 127 | 52.7 - 112 | Acceptable |

**Definitions:**

- The ERA Certified Value is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation.
- The Mean Recovery has been determined from all data reported to ERA for the parameter during this study.
- The Acceptance Limits are equal to the Mean Recovery plus and minus 3.0 standard deviations.
- The Warning Limits are equal to the Mean Recovery plus and minus 2.0 standard deviations.
- The Performance Evaluation:

  Acceptable = Reported Result falls within the Warning Limits.

  Not Acceptable = Reported Result falls outside of the Acceptance Limits.

  Check for Error = Reported Result falls within the Acceptance Limits and outside of the Warning Limits.
- n = number of data points used to calculate Mean Recovery, Acceptance and Warning Limits.

**Additional Comments:**

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InterLaB™ Soil
Statistical Summary

# BNAs
# SOIL-24

**Laboratory Code:** C0535-01 **Report Issued:** 12/1/98

| Parameter | Reported Value (µg/Kg) | ERA Certified Value (µg/Kg) | Mean Recovery (µg/Kg) | n | Acceptance Limits (µg/Kg) | Warning Limits (µg/Kg) | Performance Evaluation |
|---|---|---|---|---|---|---|---|
| Acenaphthene | 5060 | 6280 | 4800 | 21 | 2420 - 7180 | 3210 - 6390 | Acceptable |
| Anthracene | 744 | 990 | 727 | 21 | 123 - 1330 | 324 - 1130 | Acceptable |
| Benzo(b)fluoranthene | 4190 | 6240 | 4750 | 22 | 2030 - 7470 | 2940 - 6560 | Acceptable |
| Benzo(a)pyrene | 3810 | 5860 | 4410 | 22 | 1950 - 6870 | 2770 - 6050 | Acceptable |
| 4-Chlorophenyl-phenylether | 5320 | 6170 | 5110 | 22 | 2440 - 7780 | 3330 - 6890 | Acceptable |
| Chrysene | 969 | 1150 | 1110 | 22 | 310 - 1910 | 576 - 1640 | Acceptable |
| Dibenzofuran | 3640 | 4570 | 3560 | 23 | 1410 - 5710 | 2130 - 4990 | Acceptable |
| 1,2-Dichlorobenzene | 9260 | 11900 | 7220 | 22 | 2020 - 12400 | 3750 - 10700 | Acceptable |
| 1,4-Dichlorobenzene | 7690 | 10100 | 5720 | 22 | 1600 - 9840 | 2970 - 8470 | Acceptable |
| 2,4-Dinitrotoluene | 10180 | 12500 | 9190 | 20 | 3550 - 14800 | 5430 - 13000 | Acceptable |
| 2,6-Dinitrotoluene | 4720 | 6040 | 4720 | 22 | 1740 - 7700 | 2730 - 6710 | Acceptable |
| bis(2-Ethylhexyl)phthalate | 5140 | 5330 | 4680 | 21 | 2100 - 7260 | 2960 - 6400 | Acceptable |
| Fluorene | 2260 | 2720 | 2200 | 23 | 633 - 3770 | 1160 - 3240 | Acceptable |
| Hexachlorobenzene | 2330 | 2670 | 2180 | 23 | 752 - 3610 | 1230 - 3130 | Acceptable |
| Naphthalene | 3700 | 4550 | 3270 | 22 | 1370 - 5170 | 2000 - 4540 | Acceptable |
| N-Nitrosodimethylamine | 9060 | 9810 | 6760 | 17 | 1490 - 12000 | 3250 - 10300 | Acceptable |
| Pyrene | 5480 | 6640 | 5610 | 23 | 2200 - 9020 | 3340 - 7880 | Acceptable |
| 1,2,4-Trichlorobenzene | 8690 | 11600 | 7790 | 20 | 4170 - 11400 | 5370 - 10200 | Acceptable |

ENVIRONMENTAL RESOURCE ASSOCIATES

InterLaB™ Soil Statistical Summary

# BNAs
# SOIL-24

## Page 2

**Laboratory Code:** C0535-01

| Parameter | Reported Value (µg/Kg) | ERA Certified Value (µg/Kg) | Mean Recovery (µg/Kg) | n | Acceptance Limits (µg/Kg) | Warning Limits (µg/Kg) | Performance Evaluation |
|---|---|---|---|---|---|---|---|
| 2-Chlorophenol | 5630 | 7770 | 4993 | 22 | 533 - 9450 | 2020 - 7970 | Acceptable |
| 2-Methylphenol | 5220 | 8820 | 4849 | 21 | 936 - 8760 | 2240 - 7460 | Acceptable |
| Pentachlorophenol | 5630 | 8800 | 5661 | 23 | 158 - 11200 | 1990 - 9330 | Acceptable |
| Phenol | 1140 | 1520 | 1016 | 20 | 221 - 1810 | 486 - 1550 | Acceptable |
| 2,4,5-Trichlorophenol | 2500 | 3880 | 2778 | 22 | 357 - 5200 | 1160 - 4390 | Acceptable |
| 2,4,6-Trichlorophenol | 6870 | 10200 | 6919 | 22 | 2820 - 11000 | 4190 - 9650 | Acceptable |

**Definitions:**

- The ERA Certified Value is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation.
- The Mean Recovery has been determined from all data reported to ERA for the parameter during this study.
- The Acceptance Limits are equal to the Mean Recovery plus and minus 3.0 standard deviations.
- The Warning Limits are equal to the Mean Recovery plus and minus 2.0 standard deviations.
- The Performance Evaluation:

  Acceptable = Reported Result falls within the Warning Limits.

  Not Acceptable = Reported Result falls outside of the Acceptance Limits.

  Check for Error = Reported Result falls within the Acceptance Limits and outside of the Warning Limits.
- n = number of data points used to calculate Mean Recovery, Acceptance and Warning Limits.

**Additional Comments:**

ENVIRONMENTAL RESOURCE ASSOCIATES

InterLaB™ Soil
Statistical Summary

# Volatiles
# SOIL-24

**Laboratory Code:** C0535-01 **Report Issued:** 12/3/98

| Parameter | Reported Value (µg/Kg) | ERA Certified Value (µg/Kg) | Mean Recovery (µg/Kg) | n | Acceptance Limits (µg/Kg) | Warning Limits (µg/Kg) | Performance Evaluation |
|---|---|---|---|---|---|---|---|
| Benzene | 28.9 | 39.2 | 37.9 | 24 | 26.3 - 49.6 | 30.2 - 45.7 | Check for Error |
| Bromodichloromethane | 43.3 | 45.5 | 46.6 | 23 | 35.8 - 57.4 | 39.4 - 53.8 | Acceptable |
| Bromoform | 140 | 126 | 131 | 24 | 98.1 - 165 | 109 - 154 | Acceptable |
| Carbon tetrachloride | 57.5 | 80.0 | 77.2 | 23 | 49.7 - 105 | 58.9 - 95.5 | Check for Error |
| Chlorobenzene | 86.1 | 89.9 | 89.4 | 24 | 67.3 - 111 | 74.7 - 104 | Acceptable |
| Chlorodibromomethane | 57.0 | 58.0 | 57.4 | 23 | 44.3 - 70.5 | 48.6 - 66.1 | Acceptable |
| Chloroform | 39.3 | 46.6 | 45.7 | 24 | 34.4 - 56.9 | 38.2 - 53.1 | Acceptable |
| 1,2-Dichlorobenzene | 110 | 101 | 104 | 23 | 81.3 - 126 | 88.7 - 118 | Acceptable |
| 1,3-Dichlorobenzene | 29.1 | 28.4 | 28.3 | 23 | 21.7 - 34.8 | 23.9 - 32.6 | Acceptable |
| 1,4-Dichlorobenzene | 44.5 | 43.4 | 44.2 | 23 | 33.4 - 55.0 | 37.0 - 51.4 | Acceptable |
| 1,1-Dichloroethane | 50.8 | 69.6 | 67.7 | 24 | 44.0 - 91.3 | 51.9 - 83.4 | Check for Error |
| 1,2-Dichloroethane | 86.2 | 101 | 99.5 | 24 | 67.1 - 132 | 77.9 - 121 | Acceptable |
| Ethylbenzene | 60.4 | 61.2 | 61.2 | 24 | 47.4 - 75.0 | 52.0 - 70.4 | Acceptable |
| Methylene chloride | 25.2 | 39.4 | 37.7 | 23 | 23.1 - 52.2 | 28.0 - 47.4 | Check for Error |

ENVIRONMENTAL RESOURCE ASSOCIATES

InterLaB™ Soil Statistical Summary

# Volatiles
# SOIL-24

## Page 2

**Laboratory Code:** C0535-01

**Report Issued:** 12/1/98

| Parameter | Reported Value (µg/Kg) | ERA Certified Value (µg/Kg) | Mean Recovery (µg/Kg) | n | Acceptance Limits (µg/Kg) | Warning Limits (µg/Kg) | Performance Evaluation |
|---|---|---|---|---|---|---|---|
| 4-Methyl-2-pentanone (MIBK) | 20.3 | 23.4 | 22.4 | 22 | 12.6 - 32.1 | 15.9 - 28.9 | Acceptable |
| 1,1,2,2-Tetrachloroethane | 59.5 | 59.9 | 59.1 | 24 | 34.8 - 83.3 | 42.9 - 75.2 | Acceptable |
| Tetrachloroethylene | 51.9 | 56.9 | 56.6 | 24 | 41.2 - 72.0 | 46.4 - 66.9 | Acceptable |
| Toluene | 140 | 168 | 159 | 24 | 109 - 209 | 125 - 193 | Acceptable |
| 1,1,1-Trichloroethane | 17.2 | 24.4 | 23.5 | 24 | 14.2 - 32.9 | 17.3 - 29.7 | Check for Error |
| Trichloroethylene | 89.1 | 110 | 106 | 24 | 73.7 - 138 | 84.4 - 127 | Acceptable |
| Total, Xylenes | 110 | 116 | 116 | 24 | 78.7 - 152 | 91.0 - 140 | Acceptable |

**Definitions:**

- The **ERA Certified Value** is equal to 100% of the parameter present in the standard as determined by gravimetric and/or volumetric measurements made during standard preparation.
- The **Mean Recovery** has been determined from all data reported to ERA for the parameter during this study.
- The **Acceptance Limits** are equal to the Mean Recovery plus and minus 3.0 standard deviations.
- The **Warning Limits** are equal to the Mean Recovery plus and minus 2.0 standard deviations.
- The **Performance Evaluation:**
  - **Acceptable** = Reported Result falls within the Warning Limits.
  - **Not Acceptable** = Reported Result falls outside of the Acceptance Limits.
  - **Check for Error** = Reported Result falls within the Acceptance Limits and outside of the Warning Limits.
- **n** = number of data points used to calculate Mean Recovery, Acceptance and Warning Limits.

**Additional Comments:**

# ONSITE ENVIRONMENTAL LABORATORIES

Department of Health Services
ELAP (Environmental Laboratory Accreditation Program) Certification

| | |
|---|---|
| Certificate No. | 2077 |
| Issued at: | Berkeley, CA. |
| Date Issued: | Feb. 14, 2000 |

STATE OF CALIFORNIA—HEALTH AND HUMAN SERVICES AGENCY

GRAY DAVIS, Governor

**DEPARTMENT OF HEALTH SERVICES**
2151 BERKELEY WAY
BERKELEY, CA 94704-1011

(510) 540-2800

FEB 18 2000

February 14, 2000

Certificate No.:2077

VINH P. LE
ONSITE ENVIRONMENTAL LABORATORIES, INC.
5500 BOSCELL COMMON
FREMONT, CA 94538-

Dear VINH P. LE:

This is to advise you that the laboratory named above has been certified as an environmental testing laboratory pursuant to the provisions of the California Environmental Laboratory Improvement Act of 1988 (Health and Safety Code (HSC), Division 101, Part 1, Chapter 4, commencing with Section 100825).

The Fields of Testing for which this laboratory has been certified under this Act are indicated in the enclosed "List of Approved Fields of Testing and Analytes." Certification shall remain in effect until **August 31, 2001** unless revoked. This certificate is subject to an annual fee as prescribed by Section 100860(a), HSC, due on August 31, 2000.
Your application for renewal must be received 90 days before the expiration of your certificate to remain in force according to the California Code of Regulations, Title 22, Division 4, Chapter 19, Section 64801 through 64827.

Please note that your laboratory is required to notify the Environmental Laboratory Accreditation Program of any major changes in the laboratory such as the transfer of ownership, change of laboratory director, change in location, or structural alterations which may affect adversely the quality of analyses (Section 100845(b) and (d), HSC).

Your continued cooperation is essential in order to maintain a reputation for the high quality of the data produced by environmental laboratories certified by the State of California.

If you have any questions, please contact Paramjit Behniwal at (510) 540-2800.

Sincerely,

George C. Kulasingam

George C. Kulasingam, Ph.D.
Program Chief
Environmental Laboratory Accreditation Program

Enclosure

# CALIFORNIA DEPARTMENT OF HEALTH SERVICES
## ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
**List of Approved Fields of Testing and Analytes**

| | | | |
|---|---|---|---|
| ONSITE ENVIRONMENTAL LABORATORIES, INC. | MOBILELAB 4C55999 | Certificate No. | 2077 |
| MOBILE LAB #6 | TZE063V100283 | | |
| 5500 BOSCELL COMMON | PHONE No. (510) 490-8571 | Expiration Date | 08/31/2001 |
| FREMONT, CA | COUNTY ALAMEDA | | |

12 **Organic Chemistry of Hazardous Waste by GC/MS**

| | |
|---|---|
| 12.03 | EPA Method 8270B |
| 12.06A | EPA Method 8260B |
| 12.07 | Total Petroleum Hydrocarbons - Gasoline |

13 **Organic Chemistry of Hazardous Waste (excluding GC/MS)**

| | |
|---|---|
| 13.02A | EPA Method 8015B |
| 13.03 | EPA Method 8020A |
| 13.15 | Total Petroleum Hydrocarbons - Gasoline |
| 13.16 | Total Petroleum Hydrocarbons - Diesel |
| 13.17 | TRPH - Screening by IR |
| 13.19B | EPA Method 8021B Aromatic Volatiles only |
| 13.19C | EPA Method 8021B BTEX and MTBE only |

Department of Health Services
ELAP (Environmental Laboratory Accreditation Program) Certification

Certificate No. 2317

Issued at: Berkeley, CA.

Date Issued: 08/07/2000

STATE OF CALIFORNIA—HEALTH AND HUMAN SERVICES AGENCY

GRAY DAVIS, Governor

**DEPARTMENT OF HEALTH SERVICES**
2151 BERKELEY WAY
BERKELEY, CA 94704-1011

(510) 540-2800

AUG 2 9 2000

August 7, 2000

Certificate No.: 2317

JOHN HAWKINS
ONSITE ENVIRONMENTAL LABORATORIES, INC.
5500 BOSCELL COMMON
FREMONT, CA 94538-

Dear JOHN HAWKINS:

This is to advise you that the laboratory named above has been certified as an environmental testing laboratory pursuant to the provisions of the California Environmental Laboratory Improvement Act of 1988 (Health and Safety Code (HSC), Division 101, Part 1, Chapter 4, commencing with Section 100825).

The Fields of Testing for which this laboratory has been certified under this Act are indicated in the enclosed "List of Approved Fields of Testing and Analytes." Certification shall remain in effect until **September 30, 2002** unless revoked. This certificate is subject to an annual fee as prescribed by Section 100860(a), HSC, due on September 30, 2001.
Your application for renewal must be received 90 days before the expiration of your certificate to remain in force according to the California Code of Regulations, Title 22, Division 4, Chapter 19, Section 64801 through 64827.

Please note that your laboratory is required to notify the Environmental Laboratory Accreditation Program of any major changes in the laboratory such as the transfer of ownership, change of laboratory director, change in location, or structural alterations which may affect adversely the quality of analyses (Section 100845(b) and (d), HSC).

Your continued cooperation is essential in order to maintain a reputation for the high quality of the data produced by environmental laboratories certified by the State of California.

If you have any questions, please contact Nelson Lan at (510) 540-2800.

Sincerely,

Fred Seto for George Kulasingam

George C. Kulasingam, Ph.D.
Program Chief
Environmental Laboratory Accreditation Program

Enclosure

# CALIFORNIA DEPARTMENT OF HEALTH SERVICES
## ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
List of Approved Fields of Testing and Analytes

| | | | |
|---|---|---|---|
| ONSITE ENVIRONMENTAL LABORATORIES, INC.<br>MOBILE #3 | MOBILELAB G89552<br>4AGHU28D2PC018594 | Certificate No. | 2317 |
| 5500 BOSCELL COMMON<br>FREMONT, CA | PHONE No. (510) 490-8571<br>COUNTY ALAMEDA | Expiration Date | 09/30/2002 |

**12 Organic Chemistry of Hazardous Waste by GC/MS**

| | |
|---|---|
| 12.01 | EPA Method 8240B |
| 12.06 | EPA Method 8260A |
| 12.99 | Gasoline by GC/MS |

**13 Organic Chemistry of Hazardous Waste (excluding GC/MS)**

| | |
|---|---|
| 13.15 | Total Petroleum Hydrocarbons - Gasoline |
| 13.16 | Total Petroleum Hydrocarbons - Diesel |
| 13.17 | TRPH - Screening by IR |
| 13.24C | EPA Method 8082 PCBs only |

As of 08/21/2000, this list supersedes all previous lists for this certificate number.

Department of Health Services
ELAP (Environmental Laboratory Accreditation Program) Certification

Certificate No. 2317

Issued at: Berkeley, CA.

Date Issued: Sept. 1, 1998

STATE OF CALIFORNIA—HEALTH AND WELFARE AGENCY

PETE WILSON, Governor

DEPARTMENT OF HEALTH SERVICES
2151 BERKELEY WAY
BERKELEY, CA 94704-1011
(510) 540-2800

September 1, 1998

Certificate No.: 2317

MICHAEL YUROVSKY
ONSITE ENVIRONMENTAL LABORATORIES
5500 BOSCELL COMMON
FREMONT, CA 94538-

Dear MICHAEL YUROVSKY:

This is to advise you that the laboratory named above has been certified as an environmental testing laboratory pursuant to the provisions of the California Environmental Laboratory Improvement Act of 1988 (Health and Safety Code, Division 1, Part 2, Chapter 7.5, commencing with Section 100825).

The Fields of Testing for which this laboratory has been certified under this Act are indicated in the enclosed "List of Approved Fields of Testing and Analytes." Certification shall remain in effect until **September 30, 2000** unless revoked. This certificate is subject to an annual fee as prescribed by Section 100860(a), Health and Safety Code, on September 30, 1999.
Your application for renewal must be received 90 days before the expiration of your certificate to remain in force according to the California Code of Regulations, Title 22, Division 4, Chapter 19, Section 64801 through 64827.

Please note that your laboratory is required to notify the Environmental Laboratory Accreditation Program of any major changes in the laboratory such as the transfer of ownership, change of laboratory director, change in location, or structural alterations which may affect adversely the quality of analyses (Section 100845(b) and (d), California Health and Safety Code).

Your continued cooperation is essential in order to establish a reputation for the high quality of the data produced by environmental laboratories certified by the State of California.

If you have any questions, please contact Amanda Vidal at (510) 540-2800.

Sincerely,

George C. Kulasingam

George C. Kulasingam, Ph.D.
Program Chief
Environmental Laboratory
Accreditation Program

Enclosure

# CALIFORNIA DEPARTMENT OF HEALTH SERVICES
## ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
**List of Approved Fields of Testing and Analytes**

| | | | |
|---|---|---|---|
| ONSITE ENVIRONMENTAL LABORATORIES | MOBILELAB G89552 | Certificate No. | 2317 |
| MOBILE #3 | 4AGHU28D2PC018594 | | |
| 5500 BOSCELL COMMON | PHONE No. (510) 490-8571 | Expiration Date | 09/30/2000 |
| FREMONT, CA | COUNTY ALAMEDA | | |

12 Organic Chemistry of Hazardous Waste by GC/MS

- 12.01 EPA Method 8240B
- 12.06 EPA Method 8260A
- 12.99 Gasoline/BTEX/MTBE by GC/MS

13 Organic Chemistry of Hazardous Waste (excluding GC/MS)

- 13.15 Total Petroleum Hydrocarbons - Gasoline
- 13.16 Total Petroleum Hydrocarbons - Diesel
- 13.17 TRPH - Screening by IR

Department of Health Services
ELAP (Environmental Laboratory Accreditation Program) Certification

Certificate No. 2077

Issued at: Berkeley, CA.

Date Issued: 08/01/1999

**STATE OF CALIFORNIA**
**DEPARTMENT OF HEALTH SERVICES**

# ENVIRONMENTAL LABORATORY CERTIFICATION

is hereby granted to

## ONSITE ENVIRONMENTAL LABORATORIES, INC.

MOBILE LAB #6

5600 BOSCELL COMMON

FREMONT, CALIFORNIA

to conduct analyses of environmental samples as specified in the "List of Approved Fields of Testing and Analytes" which accompanies this Certificate.

This Certificate is granted in accordance with provisions of Section 1010, et seq. (New Section 100825) of the Health and Safety Code.

Certificate No.: **2077**

Expiration Date: **08/31/2001**

Issued on: **08/01/1999**

at Berkeley, California,
subject to forfeiture or revocation.

George C. Kulasingam, Ph.D.
Manager
Environmental Laboratory Accreditation Program

# CALIFORNIA DEPARTMENT OF HEALTH SERVICES
## ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
### List of Approved Fields of Testing and Analytes

| | | | |
|---|---|---|---|
| ONSITE ENVIRONMENTAL LABORATORIES, INC. | MOBILELAB 4C55999 | Certificate No. | 2077 |
| MOBILE LAB #6 | TZE063V100283 | | |
| 5500 BOSCELL COMMON | PHONE No. (510) 490-8571 | Expiration Date | 08/31/2001 |
| FREMONT, CA | COUNTY ALAMEDA | | |

**12 Organic Chemistry of Hazardous Waste by GC/MS**

| | |
|---|---|
| 12.03 | EPA Method 8270B |
| 12.06A | EPA Method 8260B |
| 12.07 | Total Petroleum Hydrocarbons - Gasoline |

**13 Organic Chemistry of Hazardous Waste (excluding GC/MS)**

| | |
|---|---|
| 13.02A | EPA Method 8015B |
| 13.03 | EPA Method 8020A |
| 13.15 | Total Petroleum Hydrocarbons - Gasoline |
| 13.16 | Total Petroleum Hydrocarbons - Diesel |
| 13.17 | TRPH - Screening by IR |
| 13.19B | EPA Method 8021B Aromatic Volatiles only |
| 13.19C | EPA Method 8021B BTEX and MTBE only |

As of 02/14/2000, this list supersedes all previous lists for this certificate number.

Department of Health Services
ELAP (Environmental Laboratory Accreditation Program) Certification

Certificate No. 1774

Issued at: Berkeley, CA.

Date Issued: Jan. 22, 1999

STATE OF CALIFORNIA—HEALTH AND WELFARE AGENCY

PETE WILSON, Governor

DEPARTMENT OF HEALTH SERVICES
2151 BERKELEY WAY
BERKELEY, CA 94704-1011
(510) 540-2800

January 22, 1999

Certificate No.: 1774

VINH P. LE
ONSITE ENVIRONMENTAL LABORATORIES
5500 BOSCELL COMMON
FREMONT, CA 94538-

Dear VINH P. LE:

This is to advise you that the laboratory named above has been certified as an environmental testing laboratory pursuant to the provisions of the California Environmental Laboratory Improvement Act of 1988 (Health and Safety Code, Division 1, Part 2, Chapter 7.5, commencing with Section 100825).

The Fields of Testing for which this laboratory has been certified under this Act are indicated in the enclosed "List of Approved Fields of Testing and Analytes." Certification shall remain in effect until **July 31, 2000** unless revoked. This certificate is subject to an annual fee as prescribed by Section 100860(a), Health and Safety Code, on July 31, 1999.
Your application for renewal must be received 90 days before the expiration of your certificate to remain in force according to the California Code of Regulations, Title 22, Division 4, Chapter 19, Section 64801 through 64827.

Please note that your laboratory is required to notify the Environmental Laboratory Accreditation Program of any major changes in the laboratory such as the transfer of ownership, change of laboratory director, change in location, or structural alterations which may affect adversely the quality of analyses (Section 100845(b) and (d), California Health and Safety Code).

Your continued cooperation is essential in order to establish a reputation for the high quality of the data produced by environmental laboratories certified by the State of California.

If you have any questions, please contact Paramjit Behniwal at (510) 540-2800.

Sincerely,

George C. Kulasingam

George C. Kulasingam, Ph.D.
Program Chief
Environmental Laboratory
Accreditation Program

Enclosure

# CALIFORNIA DEPARTMENT OF HEALTH SERVICES
# ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
**List of Approved Fields of Testing and Analytes**

| | | | |
|---|---|---|---|
| **ONSITE ENVIRONMENTAL LABORATORIES** | MOBILELAB OEL 1 | Certificate No. | 1774 |
| MOBILE LAB. #1 | 4AG4U33D3NC016846 | | |
| 5500 BOSCELL COMMON | PHONE No. (510) 490-8571 | Expiration Date | 07/31/2000 |
| FREMONT, CA | COUNTY ALAMEDA | | |

**12** **Organic Chemistry of Hazardous Waste by GC/MS**

| | |
|---|---|
| 12.01 | EPA Method 8240B |
| 12.06A | EPA Method 8260B |
| 12.99 | Gasoline by GC/MS |

**13** **Organic Chemistry of Hazardous Waste (excluding GC/MS)**

| | |
|---|---|
| 13.03 | EPA Method 8020A |
| 13.07B | EPA Method 8081 |
| 13.15 | Total Petroleum Hydrocarbons - Gasoline |
| 13.16 | Total Petroleum Hydrocarbons - Diesel |
| 13.17 | TRPH - Screening by IR |

Department of The Army
Corps of Engineers, Omaha District
HTRW Center of Expertise

Laboratory Validated for the following methods:

| | |
|---|---|
| 8260B | Volatile Organics |
| 8270C | Semivolatile Organics |
| 8081A | Organochlorine Pesticides |
| 8082 | Polychlorinated Biphenyls |
| 8330 | Explosives |
| 8015M | TPH |

Issued at: Omaha, NB

Date Issued Sept. 3, 1998

[illegible]
[illegible] CENTER ROAD
OMAHA, NEBRASKA [illegible]

REPLY TO
ATTENTION OF

September 3, 1998

Hazardous, Toxic and Radioactive Waste
Center of Expertise

ONSITE Environmental Laboratories Inc.
First Street, Building 82
Dover, New Jersey 07801

Gentlemen:

This correspondence addresses the ongoing validation status of your laboratory by the U.S. Army Corps of Engineers (USACE) for chemical analysis in support of the USACE Hazardous, Toxic, and Radioactive Waste Program.

With the inclusion of the recent successful analysis for Semi-Volatile Organics in Sediment by Method 8270C and Explosives in Soil by Method 8330, your laboratory is validated for the parameters listed below:

| METHOD | PARAMETERS | MATRIX |
|---|---|---|
| 8260B | Volatile Organics | Water[2] |
| 8260B | Volatile Organics | Solids |
| 8270C | Semi-Volatile Organics | Water[2] |
| 8270C | Semi-Volatile Organics | Solids[2] |
| 8081A | Organochlorine Pesticides | Water[2] |
| 8081A | Organochlorine Pesticides | Solids |
| 8082 | PCBs | Water[2] |
| 8082 | PCBs | Solids[2] |
| 8330 | Explosives | Solids[2] |
| 8015M | Total Petroleum Hydrocarbons-DRO | Water[1] |
| 8015M | Total Petroleum Hydrocarbons-DRO | Solids[1] |

Remarks: 1) Validation granted based on Standard Operating Procedure review only. No performance evaluation samples were sent to the laboratory by USACE. DRO - Diesel Range Organics.

2) The laboratory has successfully analyzed a performance evaluation sample for this method/matrix.

3) 'Solids' includes soils, sediments, and solid waste.

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Based on the successful analysis of the performance evaluation samples and the results of the laboratory inspection, your laboratory is validated for sample analysis by the methods listed above. Please note this validation applies to projects within the Picatinny Arsenal, New Jersey only. The period of validation is 18 months and expires on December 3, 1999.

USACE reserves the right to conduct additional laboratory auditing or to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any analytical samples from a USACE Contracting Officer Representative.

If you have any questions or comments please contact the Laboratory Validation Coordinator at (402) 697-2574.

Sincerely,

for Marcia C. Davies, Ph.D.
Director, USACE Hazardous,
Toxic and Radioactive Waste
Center of Expertise

Enclosure

# State of New Jersey

Christine Todd Whitman
Governor

Department of Environmental Protection
Office of Quality Assurance
P. O. Box 424
9 Ewing Street - 2nd Floor
Trenton, NJ 08625
Telephone: (609)292-3950
Facsimile: (609)777-1774

Robert C. Shinn, Jr.
Commissioner

JAN 24 2000

ONSITE ENVIRONMENTAL LABS INC
1915 PICKRELL RD
REIDSVILLE, NC 27320
Attn: JOHN V HAWKINS
[illegible]

Dear Laboratory Manager:

A Certificate and an Annual Certified Parameter List (ACPL) that reflects the current status of your facility are enclosed. If there are any discrepancies, please contact your Laboratory Certification Officer to verify information and make arrangements for a new ACPL. Effective with the receipt of this letter, your facility's certification status is valid through June 30, 2000. Both the ACPL and Certificate should be conspicuously displayed at your facility in a location on the premises that is visible to the public.

As always, we are available to discuss any comments or questions. Please do not hesitate to contact your laboratory certification officer or me.

Sincerely,

Joseph F. Aiello, Chief

Enclosure(s)

New Jersey is an Equal Opportunity Employer
Recycled Paper

# STATE OF NEW JERSEY

## DEPARTMENT OF ENVIRONMENTAL PROTECTION

*Certifies That*

ONSITE ENVIRONMENTAL LABORATORIES, INCORPORATED

(Laboratory Name)

14008

(Laboratory I.D. Number)

*having duly met the requirements of the*

Regulations Governing The Certification Of

Laboratories And Environmental Measurements N.J.A.C. 7:18 et.seq.

*is hereby approved as a*

**State Certified Environmental Laboratory**

*To perform the analyses as indicated on the Annual Certified Parameter List*

*which must accompany this certificate to be valid*

Expiration Date: June 30, 2000

Joseph F. Aiello, Chief
Office of Quality Assurance

THIS CERTIFICATE IS TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY WITH THE ANNUAL CERTIFIED PARAMETER LIST IN A LOCATION ON THE PREMISES VISIBLE TO THE PUBLIC

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320

Lab ID 14008

Effective Date: 07/01/99 Expiration Date: 06/30/00

| Status | Code | Parameter | EPA | ASTM | SM18 | USG9 | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW05.0100 | SEMIVOLATILE ORGANICS | | | | | 3510C, REV 3, 12/96 | |
| C | SHW05.0500 | SEMIVOLATILE ORGANICS | | | | | 3550B, REV 2, 12/96 | |
| C | SHW05.0600 | ORGANICS | | | | | 3580A, REV 1, 7/92 | |
| C | SHW05.0700 | VOLATILE ORGANICS | | | | | 5030B, REV 2, 12/96 | |
| A | SHW05.0730 | VOLATILE ORGANICS | | | | | 5035, REV 0, 12/96 | |
| A | SHW05.0731 | VOLATILE ORGANICS HIGH CONC. | | | | | 5035, REV0 12/96 | |
| C | SHW05.0910 | STACK GASES | | | | | 5041, REV 0, 9/94 | |
| C | SHW05.1000 | SEMIVOLATILE ORGANICS | | | | | 3510B, REV 3, 12/96 | |
| C | SHW05.1100 | SEMIVOLATILE ORGANICS | | | | | 3511B, REV 2, 12/96 | |
| C | SHW05.1200 | SEMIVOLATILE ORGANICS | | | | | 3520B, REV 2, 12/96 | |
| C | SHW05.1300 | SEMIVOLATILE ORGANICS | | | | | 3530C, REV 3, 12/96 | |
| C | SHW05.1600 | SEMIVOLATILE ORGANICS | | | | | 3660, REV 2, 12/96 | |
| C | SHW05.1700 | SEMIVOLATILE ORGANICS | | | | | 3665A, REV 1, 12/96 | |
| C | SHW06.1200 | ORGANOCHLOROPESTICIDES | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1201 | ALDRIN | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1202 | ALPHA-BHC | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1203 | BETA-BHC | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1204 | DELTA-BHC | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1205 | GAMMA-BHC (LINDANE) | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1206 | CHLORDANE (TECHNICAL) | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1207 | ALPHA-CHLORDANE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1208 | GAMMA-CHLORDANE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1209 | 4,4'-DDD | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1210 | 4,4'-DDE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1211 | 4,4'-DDT | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1212 | DIELDRIN | | | | | 8081A, REV 1, 12/96 | |

Joseph F. Aiello, Chief

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14006

Effective Date: 07/01/99 Expiration Date: 06/30/00

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW06.1213 | ENDOSULFAN I | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1214 | ENDOSULFAN II | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1215 | ENDOSULFAN SULFATE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1216 | ENDRIN | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1217 | ENDRIN ALDEHYDE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1218 | ENDRIN KETONE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1219 | HEPTACHLOR | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1220 | HEPTACHLOR EPOXIDE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1221 | METHOXYCHLOR | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1222 | TOXAPHENE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1310 | PCB's | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1311 | PCB-1016 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1312 | PCB-1221 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1313 | PCB-1232 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1314 | PCB-1242 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1315 | PCB-1248 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1316 | PCB-1254 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1317 | PCB-1260 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.2800 | NITROAROMATICS, NITRAMINES | | | | | 8330, REV 0, 9/94 | |
| C | SHW07.0400 | VOLATILE ORGANICS | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0401 | BENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0402 | CHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0403 | 1,2-DICHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0404 | 1,3-DICHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0405 | 1,4-DICHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0406 | ETHYLBENZENE | | | | | 8260B, REV 2, 12/96 | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/99 Expiration Date: 06/30/00

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW07.0407 | TOLUENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0408 | TOTAL XYLENES | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0409 | BROMODICHLOROMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0410 | BROMOFORM | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0411 | BROMOMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0412 | CARBON TETRACHLORIDE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0413 | CHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0414 | CHLOROETHYLVINYL ETHER | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0415 | CHLOROFORM | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0416 | CHLOROMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0417 | TRANS, 3-DICHLOROPROPENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0418 | DIBROMOCHLOROMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0419 | DICHLORODIFLUOROMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0420 | 1,1-DICHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0421 | 1,2-DICHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0422 | 1,1-DICHLOROETHENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0423 | 1,2-DICHLOROETHENE (TOTAL) | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0424 | 1,2-DICHLOROPROPANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0425 | CIS 1,3-DICHLOROPROPENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0426 | METHYLENE CHLORIDE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0427 | 1,1,2,2-TETRACHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0428 | TETRACHLOROETHENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0429 | 1,1,1-TRICHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0430 | 1,1,2-TRICHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0431 | TRICHLOROETHENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0432 | TRICHLOROFLUOROMETHANE | | | | | 8260B, REV 2, 12/96 | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/99 Expiration Date: 06/30/00

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW07.0433 | VINYL CHLORIDE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0434 | ACETONE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0435 | CARBON DISULFIDE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0436 | 2-BUTANONE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0437 | 2-HEXANONE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0438 | 4-METHYL-2-PENTANONE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0439 | METHYL-TERT-BUTYL ETHER | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0440 | ACROLEIN | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0441 | ACRYLONITRILE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0500 | SEMIVOLATILE ORGANICS | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0501 | N-NITROSODIPHENYLAMINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0502 | DIPHENYLAMINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0503 | CARBAZOLE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0504 | 3,3'-DICHLOROBENZIDINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0505 | 4-CHLORANILINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0506 | 2-NITROANILINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0507 | 2-CHLORONAPHTHALENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0508 | HEXACHLOROBENZENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0509 | HEXACHLOROBUTADIENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0510 | HEXACHLOROCYCLOPENTADIENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0511 | HEXACHLOROETHANE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0512 | 1,2,4-TRICHLOROBENZENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0513 | BIS (2-CHLOROETHOXY) METHANE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0514 | BIS (2-CHLOROISOPROPYL) ETHER (2,2-OXYBIS) (1-CHLOROPROPANE) | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0515 | 4-CHLOROPHENYL-PHENYLETHER | | | | | 8270C, REV 3, 12/96 | |

# State of New Jersey

Christine Todd Whitman
Governor

Department of Environmental Protection
Office of Quality Assurance
P.O. Box 424
9 Ewing Street – 2nd Floor
Trenton, NJ 08625
Telephone: (609)292-3950
Facsimile: (609)777-1774

Robert C. Shinn, Jr.
Commissioner

SEP 27 2000

ONSITE ENVIRONMENTAL LABS, INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
ATTN: JOHN V. HAWKINS
LAB ID # 14008

Dear Laboratory Manager:

A Certificate and an Annual Certified Parameter List (ACPL) that reflects the current status of your facility are enclosed. If there are any discrepancies, please contact your Laboratory Certification Officer to verify information and make arrangements for a new ACPL. Effective with the receipt of this letter, your facility's certification status is valid through June 30, 2001. Both the ACPL and Certificate should be conspicuously displayed at your facility in a location on the premises that is visible to the public.

As always, we are available to discuss any comments or questions. Please do not hesitate to contact your laboratory certification officer or me.

Sincerely,

Joseph F. Aiello, Chief

Enclosure(s)

New Jersey is an Equal Opportunity Employer
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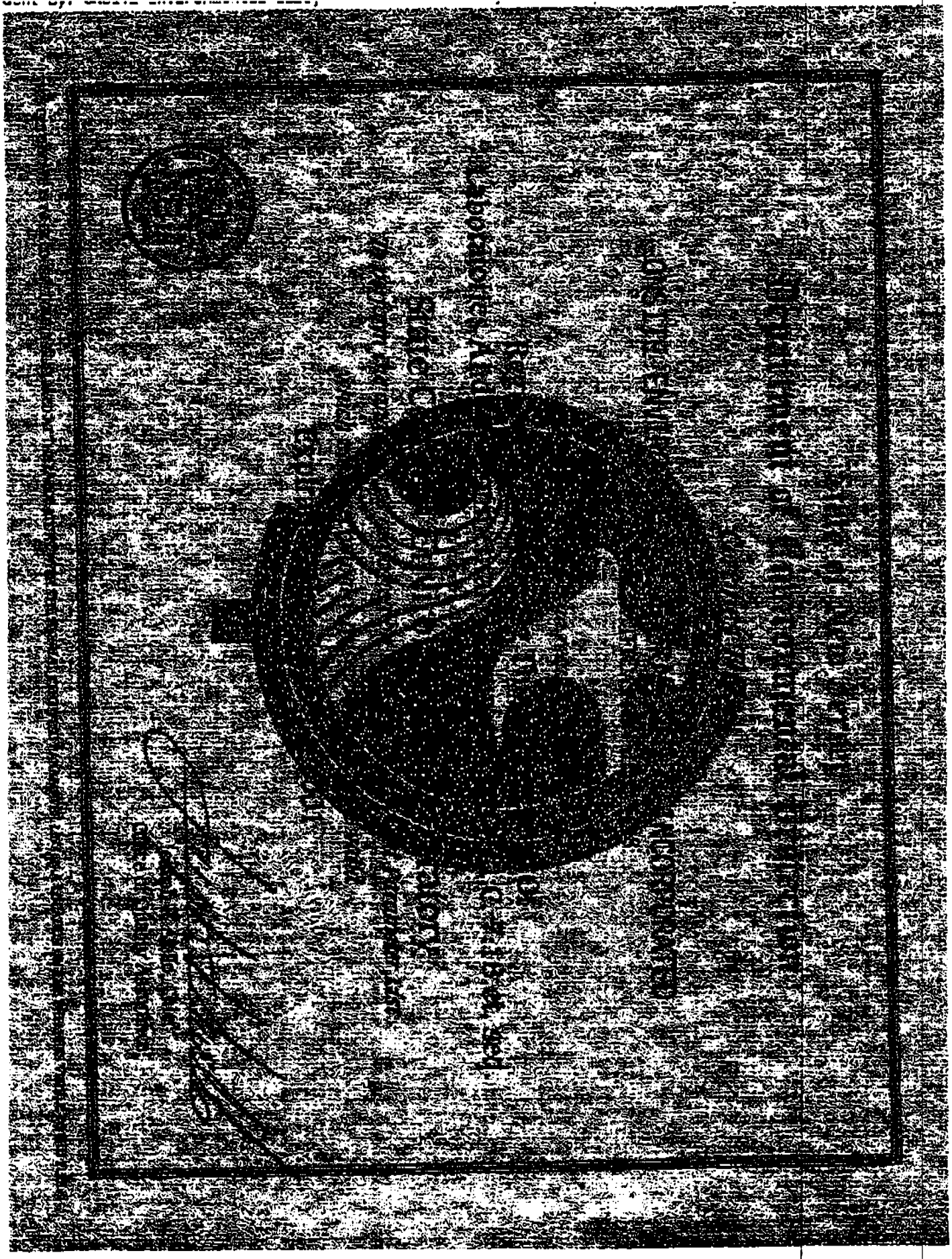

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW05.0100 | SEMIVOLATILE ORGANICS | | | | | 3510C, REV 3, 12/96 | |
| C | SHW05.0500 | SEMIVOLATILE ORGANICS | | | | | 3510B, REV 2, 12/96 | |
| C | SHW05.0600 | ORGANICS | | | | | 3580A, REV 1, 7/92 | |
| C | SHW05.0700 | VOLATILE ORGANICS | | | | | 5030B, REV 2, 12/96 | |
| A | SHW05.0730 | VOLATILE ORGANICS | | | | | 5035, REV 0, 12/96 | |
| A | SHW05.0731 | VOLATILE ORGANICS HIGH CONC. | | | | | 5035, REV0 12/96 | |
| C | SHW05.0910 | STACK GASES | | | | | 5041, REV 0, 9/94 | |
| C | SHW05.1000 | SEMIVOLATILE ORGANICS | | | | | 3610B, REV 3, 12/96 | |
| C | SHW05.1100 | SEMIVOLATILE ORGANICS | | | | | 3611B, REV 2, 12/96 | |
| C | SHW05.1200 | SEMIVOLATILE ORGANICS | | | | | 3620B, REV 2, 12/96 | |
| C | SHW05.1300 | SEMIVOLATILE ORGANICS | | | | | 3630C, REV 3, 12/96 | |
| C | SHW05.1600 | SEMIVOLATILE ORGANICS | | | | | 3660, REV 2, 12/96 | |
| C | SHW05.1700 | SEMIVOLATILE ORGANICS | | | | | 3665A, REV 1, 12/96 | |
| C | SHW06.1200 | ORGANOCHLOROPESTICIDES | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1201 | ALDRIN | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1202 | ALPHA-BHC | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1203 | BETA-BHC | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1204 | DELTA-BHC | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1205 | GAMMA-BHC (LINDANE) | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1206 | CHLORDANE (TECHNICAL) | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1207 | ALPHA-CHLORDANE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1208 | GAMMA-CHLORDANE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1209 | 4,4'-DDD | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1210 | 4,4'-DDE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1211 | 4,4'-DDT | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1212 | DIELDRIN | | | | | 8081A, REV 1, 12/96 | |

Joseph F. Aiello, Chief

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW06.1213 | ENDOSULFAN I | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1214 | ENDOSULFAN II | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1215 | ENDOSULFAN SULFATE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1216 | ENDRIN | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1217 | ENDRIN ALDEHYDE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1218 | ENDRIN KETONE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1219 | HEPTACHLOR | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1220 | HEPTACHLOR EPOXIDE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1221 | METHOXYCHLOR | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1222 | TOXAPHENE | | | | | 8081A, REV 1, 12/96 | |
| C | SHW06.1310 | PCB's | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1311 | PCB-1016 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1312 | PCB-1221 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1313 | PCB-1232 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1314 | PCB-1242 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1315 | PCB-1248 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1316 | PCB-1254 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.1317 | PCB-1260 | | | | | 8082, REV 0, 12/96 | |
| C | SHW06.2800 | NITROAROMATICS, NITRAMINES | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2801 | HMX | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2802 | RDX | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2803 | 1,3,5-TNB | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2804 | 1,3-DNB | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2805 | TETRYL | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2806 | NB | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2807 | 2,4,6-TNT | | | | | 8330, REV 0, 9/94 | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW06.2808 | 4-AM-DNT | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2809 | 2-AM-DNT | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2810 | 2,4-DNT | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2811 | 2,6-DNT | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2812 | 2-NT | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2813 | 3-NT | | | | | 8330, REV 0, 9/94 | |
| C | SHW06.2814 | 4-NT | | | | | 8330, REV 0, 9/94 | |
| C | SHW07.0400 | VOLATILE ORGANICS | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0401 | BENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0402 | CHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0403 | 1,2-DICHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0404 | 1,3-DICHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0405 | 1,4-DICHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0406 | ETHYLBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0407 | TOLUENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0408 | TOTAL XYLENES | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0409 | BROMODICHLOROMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0410 | BROMOFORM | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0411 | BROMOMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0412 | CARBON TETRACHLORIDE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0413 | CHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0414 | 2-CHLOROETHYL VINYL ETHER | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0415 | CHLOROFORM | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0416 | CHLOROMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0417 | TRANS, 3-DICHLOROPROPENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0418 | DIBROMOCHLOROMETHANE | | | | | 8260B, REV 2, 12/96 | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW07.0419 | DICHLORODIFLUOROMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0420 | 1,1-DICHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0421 | 1,2-DICHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0422 | 1,1-DICHLOROETHENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0423 | 1,2-DICHLOROETHENE (TOTAL) | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0424 | 1,2-DICHLOROPROPANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0425 | CIS 1,3-DICHLOROPROPENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0426 | METHYLENE CHLORIDE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0427 | 1,1,2,2-TETRACHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0428 | TETRACHLOROETHENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0429 | 1,1,1-TRICHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0430 | 1,1,2-TRICHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0431 | TRICHLOROETHENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0432 | TRICHLOROFLUOROMETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0433 | VINYL CHLORIDE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0434 | ACETONE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0435 | CARBON DISULFIDE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0436 | 2-BUTANONE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0437 | 2-HEXANONE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0438 | 4-METHYL-2-PENTANONE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0439 | METHYL-TERT-BUTYL ETHER | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0440 | ACROLEIN | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0441 | ACRYLONITRILE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0450 | HEXACHLOROBUTADIENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0453 | HEXACHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0454 | NAPTHALENE | | | | | 8260C, REV 2, 12/96 | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

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1918 PICKRELL RD
REIDSVILLE, NC 27320
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|---|---|---|---|---|---|---|---|---|
| C | SHW07.0455 | STYRENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0456 | 1,1,1,2-TETRACHLOROETHANE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0457 | 1,2,4-TRICHLOROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0458 | NITROBENZENE | | | | | 8260B, REV 2, 12/96 | |
| C | SHW07.0500 | SEMIVOLATILE ORGANICS | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0501 | N-NITROSODIPHENYLAMINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0502 | DIPHENYLAMINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0503 | CARBAZOLE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0504 | 3,3'-DICHLOROBENZIDINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0505 | 4-CHLORANILINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0506 | 2-NITROANILINE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0507 | 2-CHLORONAPHTHALENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0508 | HEXACHLOROBENZENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0509 | HEXACHLOROBUTADIENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0510 | HEXACHLOROCYCLOPENTADIENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0511 | HEXACHLOROETHANE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0512 | 1,2,4-TRICHLOROBENZENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0513 | BIS (2-CHLOROETHOXY) METHANE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0514 | BIS (2-CHLOROISOPROPYL) ETHER (2,2-OXYBIS) (1-CHLOROPROPANE) | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0515 | 4-CHLOROPHENYL-PHENYLETHER | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0516 | 4-BROMOPHENYL-PHENYLETHER | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0517 | 2,4-DINITROTOLUENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0518 | 2,6-DINITROTOLUENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0519 | ISOPHORONE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0520 | NITROBENZENE | | | | | 8270C, REV 3, 12/96 | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW07.0521 | BUTYL BENZYL PHTHALATE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0522 | BIS (2-ETHYLHEXYL) PHTHALATE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0523 | DIETHYL PHTHALATE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0524 | DIMETHYL PHTHALATE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0525 | DI-N-BUTYL PHTHALATE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0526 | DI-N-OCTYL PHTHALATE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0527 | ACENAPHTHENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0528 | ANTHRACENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0529 | ACENAPHTHYLENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0530 | BENZO(A)ANTHRACENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0531 | BENZO(A)PYRENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0532 | BENZO(B)FLUORANTHENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0533 | BENZO(GHI)PERYLENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0534 | BENZO(K)FLUORANTHENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0535 | CHRYSENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0536 | DIBENZO(A,H)ANTHRACENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0537 | FLUORANTHENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0538 | FLUORENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0539 | INDENO(1,2,3-CD)PYRENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0540 | 2-METHYLNAPHTHALENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0541 | NAPHTHALENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0542 | PHENANTHRENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0543 | PYRENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0544 | 4-CHLORO-3-METHYL-PHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0545 | 2-CHLOROPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0546 | 2,4-DICHLOROPHENOL | | | | | 8270C, REV 3, 12/96 | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| C | SHW07.0547 | 2,4-DIMETHYLPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0548 | 2,4-DINITROPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0549 | 2-METHYL-4,6-DINITROPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0550 | 2-METHYLPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0551 | 4-METHYLPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0552 | 2-NITROPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0553 | 4-NITROPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0554 | PENTACHLOROPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0555 | PHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0556 | 2,4,5-TRICHLOROPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0557 | 2,4,6-TRICHLOROPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0559 | 3-METHYLPHENOL | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0560 | DIBENZOFURAN | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0570 | 1,4-DICHLOROBENZENE | | | | | 8270C, REV 3, 12/96 | |
| C | SHW07.0575 | PYRIDINE | | | | | 8270C, REV 3 12/96 | |
| C | WPP05.0900 | ORGANOCHLORINE PESTICIDES | 608 | | | | | |
| C | WPP05.0901 | ALDRIN | 608 | | | | | |
| C | WPP05.0902 | ALPHA-BHC | 608 | | | | | |
| C | WPP05.0903 | BETA-BHC | 608 | | | | | |
| C | WPP05.0904 | DELTA-BHC | 608 | | | | | |
| C | WPP05.0905 | GAMMA-BHC | 608 | | | | | |
| C | WPP05.0906 | CHLORDANE (TECHNICAL) | 608 | | | | | |
| C | WPP05.0907 | 4,4'-DDD | 608 | | | | | |
| C | WPP05.0908 | 4,4'-DDE | 608 | | | | | |
| C | WPP05.0909 | 4,4'-DDT | 608 | | | | | |
| C | WPP05.0910 | DIELDRIN | 608 | | | | | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

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|---|---|---|---|---|---|---|---|---|
| C | WPP05.0911 | ENDOSULFAN I | 608 | | | | | |
| C | WPP05.0912 | ENDOSULFAN II | 608 | | | | | |
| C | WPP05.0913 | ENDOSULFAN SULFATE | 608 | | | | | |
| C | WPP05.0914 | ENDRIN | 608 | | | | | |
| C | WPP05.0915 | ENDRIN ALDEHYDE | 608 | | | | | |
| C | WPP05.0916 | ENDRIN KETONE | 608 | | | | | |
| C | WPP05.0917 | HEPTACHLOR | 608 | | | | | |
| C | WPP05.0918 | HEPTACHLOR EPOXIDE | 608 | | | | | |
| C | WPP05.0919 | METHOXYCHLOR | 608 | | | | | |
| C | WPP05.0920 | TOXAPHENE | 608 | | | | | |
| C | WPP05.1101 | PCB-1016 | 608 | | | | | |
| C | WPP05.1102 | PCB-1221 | 608 | | | | | |
| C | WPP05.1103 | PCB-1232 | 608 | | | | | |
| C | WPP05.1104 | PCB-1242 | 608 | | | | | |
| C | WPP05.1105 | PCB-1248 | 608 | | | | | |
| C | WPP05.1106 | PCB-1254 | 608 | | | | | |
| C | WPP05.1107 | PCB-1260 | 608 | | | | | |
| A | WPP06.0200 | VOLATILE ORGANICS | 624 | | | | | |
| A | WPP06.0201 | BENZENE | 624 | | | | | |
| A | WPP06.0202 | BROMODICHLOROMETHANE | 624 | | | | | |
| A | WPP06.0203 | BROMOFORM | 624 | | | | | |
| A | WPP06.0204 | BROMOMETHANE | 624 | | | | | |
| A | WPP06.0205 | CARBON TETRACHLORIDE | 624 | | | | | |
| A | WPP06.0206 | CHLOROBENZENE | 624 | | | | | |
| A | WPP06.0207 | CHLOROETHANE | 624 | | | | | |
| A | WPP06.0208 | 2-CHLOROETHYLVINYL ETHER | 624 | | | | | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| A | WPP06.0209 | CHLOROFORM | 624 | | | | | |
| A | WPP06.0210 | CHLOROMETHANE | 624 | | | | | |
| A | WPP06.0211 | DIBROMOCHLOROMETHANE | 624 | | | | | |
| A | WPP06.0212 | 1,2-DICHLOROBENZENE | 624 | | | | | |
| A | WPP06.0213 | 1,3-DICHLOROBENZENE | 624 | | | | | |
| A | WPP06.0214 | 1,4-DICHLOROBENZENE | 624 | | | | | |
| A | WPP06.0215 | 1,1-DICHLOROETHANE | 624 | | | | | |
| A | WPP06.0216 | 1,2-DICHLOROETHANE | 624 | | | | | |
| A | WPP06.0217 | 1,1-DICHLOROETHENE | 624 | | | | | |
| A | WPP06.0218 | TRANS-1,2-DICHLOROETHENE | 624 | | | | | |
| A | WPP06.0219 | 1,2-DICHLOROPROPANE | 624 | | | | | |
| A | WPP06.0220 | CIS-1,3-DICHLOROPROPENE | 624 | | | | | |
| A | WPP06.0221 | TRANS-1,3-DICHLOROPROPENE | 624 | | | | | |
| A | WPP06.0222 | ETHYLBENZENE | 624 | | | | | |
| A | WPP06.0223 | METHYLENE CHLORIDE | 624 | | | | | |
| A | WPP06.0224 | 1,1,2,2-TETRACHLOROETHANE | 624 | | | | | |
| A | WPP06.0225 | TETRACHLOROETHENE | 624 | | | | | |
| A | WPP06.0226 | TOLUENE | 624 | | | | | |
| A | WPP06.0227 | 1,1,1-TRICHLOROETHANE | 624 | | | | | |
| A | WPP06.0228 | 1,1,2-TRICHLOROETHANE | 624 | | | | | |
| A | WPP06.0229 | TRICHLOROETHENE | 624 | | | | | |
| A | WPP06.0230 | TRICHLOROFLUOROMETHANE | 624 | | | | | |
| A | WPP06.0231 | VINYL CHLORIDE | 624 | | | | | |
| A | WPP06.0300 | BASE/NEUTRALS AND ACIDS | 625 | | | | | |
| A | WPP06.0301 | ACENAPHTHENE | 625 | | | | | |
| A | WPP06.0302 | ACENAPHTHYLENE | 625 | | | | | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

| Status | Code | Parameter | EPA | ASTM | SM18 | USGS | SW846 | Other |
|---|---|---|---|---|---|---|---|---|
| A | WPP06.0303 | ANTHRACENE | 625 | | | | | |
| A | WPP06.0304 | BENZO(A)ANTHRACENE | 625 | | | | | |
| A | WPP06.0305 | BENZO(B)FLUORANTHENE | 625 | | | | | |
| A | WPP06.0306 | BENZO(K)FLUORANTHENE | 625 | | | | | |
| A | WPP06.0307 | BENZO(K)PYRENE | 625 | | | | | |
| A | WPP06.0308 | BENZO(GHI)PERYLENE | 625 | | | | | |
| A | WPP06.0309 | BUTYL BENZYL PHTHALATE | 625 | | | | | |
| A | WPP06.0310 | BIS (2-CHLOROETHYL) ETHER | 625 | | | | | |
| A | WPP06.0311 | BIS (2-CHLOROETHOXY)METHANE | 625 | | | | | |
| A | WPP06.0312 | BIS (2-ETHYLHEXYL) PHTHALATE | 625 | | | | | |
| A | WPP06.0313 | BIS (2-CHLOROISOPROPYL) ETHER | 625 | | | | | |
| A | WPP06.0314 | 4-BROMOPHENYL-PHENYL ETHER | 625 | | | | | |
| A | WPP06.0315 | 2-CHLORONAPHTHALENE | 625 | | | | | |
| A | WPP06.0316 | 4-CHLOROPHENYL-PHENYL ETHER | 625 | | | | | |
| A | WPP06.0317 | CHRYSENE | 625 | | | | | |
| A | WPP06.0318 | DIBENZO(A,H)ANTHRACENE | 625 | | | | | |
| A | WPP06.0319 | DI-N-BUTYL PHTHALATE | 625 | | | | | |
| A | WPP06.0320 | 1,3-DICHLOROBENZENE | 625 | | | | | |
| A | WPP06.0321 | 1,2-DICHLOROBENZENE | 625 | | | | | |
| A | WPP06.0322 | 1,4-DICHLOROBENZENE | 625 | | | | | |
| A | WPP06.0323 | 3,3'-DICHLOROBENZIDINE | 625 | | | | | |
| A | WPP06.0324 | DIETHYL PHTHALATE | 625 | | | | | |
| A | WPP06.0325 | DIMETHYL PHTHALATE | 625 | | | | | |
| A | WPP06.0326 | 2,4-DINITROTOLUENE | 625 | | | | | |
| A | WPP06.0327 | 2,6-DINITROTOLUENE | 625 | | | | | |
| A | WPP06.0328 | DI-N-OCTYL PHTHALATE | 625 | | | | | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1918 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008

Effective Date: 07/01/2000 Expiration Date: 06/30/2001

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|---|---|---|---|---|---|---|---|---|
| A | WPP06.0329 | FLUORANTHENE | 625 | | | | | |
| A | WPP06.0330 | FLUORENE | 625 | | | | | |
| A | WPP06.0331 | HEXACHLOROBENZENE | 625 | | | | | |
| A | WPP06.0332 | HEXACHLOROBUTADIENE | 625 | | | | | |
| A | WPP06.0333 | HEXACHLOROETHANE | 625 | | | | | |
| A | WPP06.0334 | INDENO(1,2,3-CD)PYRENE | 625 | | | | | |
| A | WPP06.0335 | ISOPHORONE | 625 | | | | | |
| A | WPP06.0336 | NAPHTHALENE | 625 | | | | | |
| A | WPP06.0337 | NITROBENZENE | 625 | | | | | |
| A | WPP06.0338 | N-NITROSODI-N-PROPYLAMINE | 625 | | | | | |
| A | WPP06.0339 | PHENANTHRENE | 625 | | | | | |
| A | WPP06.0340 | PYRENE | 625 | | | | | |
| A | WPP06.0341 | 1,2,4-TRICHLOROBENZENE | 625 | | | | | |
| A | WPP06.0342 | 4-CHLORO-3-METHYL-PHENOL | 625 | | | | | |
| A | WPP06.0343 | 2-CHLOROPHENOL | 625 | | | | | |
| A | WPP06.0344 | 2,4-DICHLOROPHENOL | 625 | | | | | |
| A | WPP06.0345 | 2,4-DIMETHYLPHENOL | 625 | | | | | |
| A | WPP06.0346 | 2,4-DINITROPHENOL | 625 | | | | | |
| A | WPP06.0347 | 2-METHYL-4,6-DINITROPHENOL | 625 | | | | | |
| A | WPP06.0348 | 2-NITROPHENOL | 625 | | | | | |
| A | WPP06.0349 | 4-NITROPHENOL | 625 | | | | | |
| A | WPP06.0350 | PENTACHLOROPHENOL | 625 | | | | | |
| A | WPP06.0351 | PHENOL | 625 | | | | | |
| A | WPP06.0352 | 2,4,6-TRICHLOROPHENOL | 625 | | | | | |
| A | WPP06.0353 | BENZOIC ACID | 625 | | | | | |
| A | WPP06.0354 | P-CRESOL | 625 | | | | | |

# New Jersey Department of Environmental Protection
## Environmental Laboratory Certification Program
## Annual Certified Parameter List and Current Status

ONSITE ENVIRONMENTAL LABS INC
1916 PICKRELL RD
REIDSVILLE, NC 27320
Lab ID 14008
Effective Date: 07/01/2000 Expiration Date: 06/30/2001

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|---|---|---|---|---|---|---|---|---|
| A | WPP06.0355 | ACETOPHENONE | 625 | | | | | |
| A | WPP06.0356 | ALPHA-TERPINEOL | 625 | | | | | |
| A | WPP06.0357 | ANILINE | 625 | | | | | |
| A | WPP06.0358 | BENZIDINE | 625 | | | | | |
| A | WPP06.0359 | CARBAZOLE | 625 | | | | | |
| A | WPP06.0360 | 2,3-DICHLOROANILINE | 625 | | | | | |
| A | WPP06.0362 | N-DECANE | 625 | | | | | |
| A | WPP06.0363 | N-DOCOSANE | 625 | | | | | |
| A | WPP06.0364 | N-DODECANE | 625 | | | | | |
| A | WPP06.0365 | N-EICOSANE | 625 | | | | | |
| A | WPP06.0366 | HEXACHLOROCYCLOPENTADIENE | 625 | | | | | |
| A | WPP06.0367 | N-HEXADECANE | 625 | | | | | |
| A | WPP06.0368 | N-NITROSODIMETHYLAMINE | 625 | | | | | |
| A | WPP06.0369 | N-NITROSODIPHENYLAMINE | 625 | | | | | |
| A | WPP06.0370 | N-OCTADECANE | 625 | | | | | |
| A | WPP06.0371 | N-TETRADECANE | 625 | | | | | |
| A | WPP06.0372 | PYRIDINE | 625 | | | | | |
| A | WPP06.0373 | 1-METHYLPHENANTHRENE | 625 | | | | | |

Key: A Applied, C Certified, D Dropped by Lab, S Suspended, T Temporary Certification

E.S. BABCOCK & SONS LABORATORIES

Department of Health Services
Sanitation and Radiation Laboratories Branch

Approval for Perchlorate Determinations in Water

Issued at: Los Angeles, CA.

Date Issued: Jun. 23, 1997

State of California

Department of Health Services

# Memorandum

Date : June 23, 1997

To : Clifford Sharpe, P.E., Chief
Drinking Water Field Operations Branch
601 North 7th Street MS 92
Post Office Box 942732
Sacramento, CA 94704

From : Sanitation and Radiation Laboratories Branch
1449 West Temple Street, Room 101
Los Angeles, CA 90026
(213) 580-5795

Subject : Laboratory Approval for Perchlorate Determinations in Drinking Water
E.S. Babcock & Sons, Incorporated (ELAP No. 1156), Riverside, CA

We are pleased to inform the Drinking Water Field Operations Branch that the laboratory identified above successfully underwent a voluntary on-site audit on June 30, 1997, by the Environmental Laboratory Accreditation Program staff, of its readiness to analyze Drinking Water Samples for the presence of the Perchlorate ion.

The laboratory demonstrated it competency for the procedure through satisfactory completion of Method Detection Limit studies, through a written Standard Operating Procedure, a Quality Control procedure, presentation of an acceptable data package, and by the successful analysis of an unknown Performance Evaluation Sample.

Please inform your District Engineers that the E.S. Babcock & Sons, Incorporated has been evaluated as acceptable for perchlorate determinations in drinking water. District staff may recommend the laboratory's services to their water systems.

F. J. Baumann

Frank J. Baumann, P.E., Chief

cc: E.S. Babcock, Incorporated

ENVIRONMENTAL RESOURCE ASSOCIATES®

# WS-44 Final Customer Report

**ERA Laboratory Code: E3038-01** **EPA ID: CA00102** **State ID:** **Report Issued: 06/09/00**

| ERA Standard | Analyte | Units | Reported Value | Assigned Value | Acceptance Limits | Performance Evaluation | Method Description |
|---|---|---|---|---|---|---|---|
| *Corrosivity* | Corrosivity | S.I. | 1.82 | 1.49 | 0.969 - 2.01 | Acceptable | SM 2330B |
| *MBAS* | MBAS | mg/l | 0.433 | 0.401 | 0.301 - 0.501 | Acceptable | SM 5540C |
| *Silica* | Silica as SiO2 | mg/l | 210 | 203 | 173 - 233 | Acceptable | EPA 200.7 |
| *Perchlorate* | Perchlorate | mg/l | 0.853 | 0.880 | 0.660 - 1.10 | Acceptable | EPA 314 |
| *UV 254* | UV 254 | cm -1 | 0.585 | 0.713 | 0.535 - 0.891 | Acceptable | SM 5910 |
| *GAD* | Ethyl-t-butylether (ETBE) | µg/l | 0 | ZERO | | Acceptable | EPA 524.2 |
| | T-amylmethylether (TAME) | µg/l | 0 | ZERO | | Acceptable | EPA 524.2 |
| | Di-isopropylether (DIPE) | µg/l | 0 | ZERO | | Acceptable | EPA 524.2 |
| | Trichlorotrifluoroethane | µg/l | 0 | ZERO | | Acceptable | EPA 524.2 |
| | MTBE | µg/l | 25.0 | 19.1 | 15.3 - 22.9 | Not Acceptable | EPA 524.2 |
| | 1-Phenylpropane | µg/l | 9.19 | 9.43 | 5.66 - 13.2 | Acceptable | EPA 524.2 |

NVLAP®

FRONTIER GEOSCIENCES, INC.

# The State of Washington
## Department of Ecology

This is to certify that

**Frontier Geosciences, Incorporated**
**Seattle, Washington**

has complied with provisions set forth in Chapter 173-50 WAC and is hereby recognized by the Department of Ecology as an **ACCREDITED LABORATORY** for the analytical parameters listed on the accompanying Scope of Accreditation. This certificate is effective on the **3rd day of June 2000,** and shall expire on the **2nd day of June 2001.**

Witnessed under my hand this 7th day of September 2000.

Perry F. Brake, Chemist
Lab Accreditation Unit Supervisor

LAB ACCREDITATION NUMBER
**C239**

COPY

# SCOPE OF ACCREDITATION

## Frontier Geosciences, Incorporated
## Seattle, Washington

is accredited by the State of Washington Department of Ecology to perform analyses for the parameters listed below using the analytical methods indicated. This Scope of Accreditation applies to non-potable water analyses only. Accreditation for all parameters is final unless otherwise indicated in a note. EPA refers to the U.S. Environmental Protection Agency. "SM" refers to APHA *Standard Methods for the Examination of Water and Wastewater*, 19th Edition. "FGS" refers to methods referencing Standard Operating Procedures of Frontier Geosciences.

| PARAMETER | METHOD | NOTES |
|---|---|---|
| Aluminum by GFAA | FGS-021 | 2 |
| Aluminum by ICP-MS | EPA 1638 | |
| Antimony by GFAA | FGS-021 | 2 |
| Antimony by ICP-MS | EPA 1638 | |
| Arsenic by GFAA | FGS-021 | 2 |
| Arsenic Species-AA Hydride | FGS-022 | 1 |
| Arsenic by ICP-MS | EPA 1638 | |
| Barium by GFAA | FGS-021 | 2 |
| Barium by ICP-MS | EPA 1638 | |
| Beryllium by GFAA | FGS-021 | 2 |
| Beryllium by ICP-MS | EPA 1638 | |
| Cadmium by GFAA | FGS-021 | 2 |
| Cadmium by ICP-MS | EPA 1638 | |
| Chromium by GFAA | FGS-021 | 2 |
| Chromium by ICP-MS | EPA 1638 | |
| Cobalt by GFAA | FGS-021 | 2 |
| Cobalt by ICP-MS | EPA 1638 | |
| Copper by GFAA | FGS-021 | 2 |
| Copper by ICP-MS | EPA 1638 | |
| Iron by GFAA | FGS-021 | 2 |
| Iron by ICP-MS | EPA 1638 | |
| Lead by GFAA | FGS-021 | 2 |
| Lead by ICP-MS | EPA 1638 | |
| Manganese by GFAA | FGS-021 | 2 |
| Manganese by ICP-MS | EPA 1638 | |
| Mercury, Total by CVAF | EPA 1631 B | |

Frontier Geosciences, Incorporated Scope of Accreditation (Cont'd)

| PARAMETER | METHOD | NOTES |
|---|---|---|
| Methyl Mercury, Total | EPA 1630 | 1 |
| Mercury, Volatile Species | FGS-015 | 1 |
| Molybdenum by GFAA | FGS-021 | 2 |
| Molybdenum by ICP-MS | EPA 1638 | |
| Nickel by GFAA | FGS-021 | 2 |
| Nickel by ICP-MS | EPA 1638 | |
| Selenium by GFAA | FGS-021 | 2 |
| Selenium Species-AA Hydride | FGS-037 | 1 |
| Selenium by ICP-MS | EPA 1638 | |
| Silver by GFAA | FGS-021 | 2 |
| Silver by ICP-MS | EPA 1638 | |
| Thallium by GFAA | FGS-021 | 2 |
| Thallium by ICP-MS | EPA 1638 | |
| Tin by GFAA | FGS-021 | 2 |
| Vanadium by GFAA | FGS-021 | 2 |
| Vanadium by ICP-MS | EPA 1638 | |
| Zinc by GFAA | FGS-021 | 2 |
| Zinc by ICP-MS | EPA 1638 | |

NOTES:
1. Interim due to inability of Department of Ecology to locate suitable readily available PE sample (WAC 173-50-100).
2. Provisional pending submittal of acceptable performance evaluation sample results. (WAC 173-50-110).

AUTHENTICATION: _____
Perry F. Brake
Lab Accreditation Unit Supervisor

June 2, 2001
Expiration Date

Frontier Geosciences, Incorporated Scope of Accreditation (Cont'd)

## PARAMETERS DENIED ACCREDITATION

| PARAMETER | METHOD | NOTES |
|---|---|---|
| Solids, Total Suspended | FGS-016 | 1 |
| Iron | SM 3500-Fe D | 1 |
| Tin | FGS-021 | 1 |

NOTES: 1. Denied pending receipt of acceptable performance evaluation results.

**STATE OF CALIFORNIA**
**DEPARTMENT OF HEALTH SERVICES**

# ENVIRONMENTAL LABORATORY CERTIFICATION

is hereby granted to

**FRONTIER GEOSCIENCES, INC.**

414 PONTIUS AVEN

SEATTLE, WASHINGTON

to conduct analyses of environmental samples as specified in the "List of Approved Fields of Testing and Analytes" which accompanies this Certificate.

This Certificate is granted in accordance with provisions of Section 1010, et seq. (New Section 100825) of the Health and Safety Code.

Certificate No.: **2390**

Expiration Date: **11/30/2001**

Issued on: **11/12/1999**
at Berkeley, California,
subject to forfeiture or revocation.

George C. Kulasingam, Ph.D.
Manager
Environmental Laboratory Accreditation Program

COPY

# CALIFORNIA DEPARTMENT OF HEALTH SERVICES
## ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
**List of Approved Fields of Testing and Analytes**

**FRONTIER GEOSCIENCES, INC.**
414 PONTIUS AVE N
SEATTLE, WA
PHONE No. (206) 622-6960

Certificate No. 2390
Expiration Date 11/30/2001

17 **Toxic Chemical Elements in Wastewater**

17.16 Mercury

COPY

As of 11/12/1999, this list supersedes all previous lists for this certificate number.

# ATTACHMENT 5

# LABORATORY AUDIT REPORTS

# ONSITE ENVIRONMENTAL LABORATORIES

**MEMORANDUM**

TO: Brian Weith, Earth Tech

FROM: Chris Davis, Project Chemist for the Benicia Tourtelot Project

DATE: January 5, 2000

RE: Earth Tech Response Comments to the audit and follow-up audit by Julia Halsne, ACOE as attached

---

TO: Chris Davis, Earth Tech

FROM: Chris Amstutz, Quality Assurance Officer

DATE: December 22, 1999 (Updated March 15, 2000 with References to Appendices [in italics])

RE: Onsite Laboratories Response Comments to the audit and follow-up audit by Julia Halsne, ACOE as attached

---

CESPK-ED-EF 17 December 1999

SUBJECT: Follow up comments for audit conducted 16 December 1999 – Laboratory Audit of Onsite Environmental Laboratories, Inc. (Onsite) Mobile Laboratory, Benicia Tourtelot Property, Remedial Investigation, Tourtelot Cleanup Project, Earth Tech, original audit conducted 08 December 1999. Prepared by Julia Halsne (LDC) USACE project chemist.

ATTENDEES: Chris Davis, Project Chemist, Earth Tech
Peter Balas, Program Manager, Onsite
Chris Amstutz, QA Manager, Onsite
Michael Yurosky, Volatile Organic Compounds (VOC) and Log-In Analyst
Chris Cao, High Performance Liquid Chromatography (HPLC) Analyst
Isabel Rivera, Explosives Extractions

Each finding is ranked according to the level of corrective action required, as follows:

**M = major deficiency**
A major deficiency is a significant finding that should be resolved immediately.

**m = minor deficiency**
A minor deficiency is an important finding with less urgency than a major deficiency; however, resolution of these findings should be seriously considered.

**r = recommendation**
A recommendation is a suggestion for improvement of current practices that the laboratory should consider adding to the scheduled changes in procedures.

**o = observation**
An observation is an issue identified that Earth Tech should be aware of, as it may impact the project. Earth Tech should discuss these issues with the laboratory, but they do not require corrective action by the laboratory at this time.

**USACE Technical Manager Notes (John Esparza) - C = concur** (concur with comment, response necessary); **D = deny** (no response necessary); **C&R = check and resolve** (concur with comment – look into it further and resolve at a later time).

| No. | Section | Comments | Follow-Up USACE Comments with Onsite Response Comments | Earth Tech Comments |
|---|---|---|---|---|
| 1 | Overall | The laboratories (3 trailers) had arrived on site one week prior to the audit. There were boxes and papers throughout the area and some of the laboratories were not well organized. It was difficult to find paperwork and documentation due to the lack of organization. Each laboratory should be evaluated for adequate facilities for storage and workspace. Old boxes and paperwork should be removed and stored elsewhere. Current documentation should be made readily available in an organized manner. **(m) (C)** | Resolved<br><br>**Onsite Comment**: The laboratories, paperwork, and work areas have been organized and the work areas maximized to facilitate the timely completion of the work. | The issue has been resolved.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 2 | Overall | The laboratory Quality Assurance (QA) Manual provided during the audit was not current (1996). There was one copy of the applicable method standard operating procedures (SOPs) available for all staff. Due to the recent revisions of the SOPs, the staff was not very familiar with the current SOPs. Each staff member should have a copy of the current QA Manual, applicable SOPs, and USEPA reference methods. The staff must be trained regarding the QA Manual, applicable SOPs, project-specific QAPP, and reference | The QA Manual on site was dated 1998. The QA Manual should be updated annually.<br><br>**Onsite Comment**: The QA Manual available for review was from 1998. The procedures and requirements performed by Onsite vary substantially from site to site. Onsite provided input into the site specific QAPP for this project and has revised numerous SOPs. Onsite has been meeting or exceeding the requirements for this project as outlined in the site specific QAPP. In | All laboratory analysts are highly trained, experienced, and familiar with the requirements of the US EPA Methods and the project QAPP, as well as for US ACOE projects. The staff were further trained in updating SOPs, which are current and available.<br><br>There is no indication in the audit report that analyses were performed in any way not in compliance with QAPP and method requirements. All data will be thoroughly reviewed for compliance with QAPP and method requirements in the data validation process. If any non-compliance is identified, |

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| | | methods. **(M) (C – provide documentation that personnel reviewed project specific QAPP)** | addition, the QA Manual has been updated and copy placed in each of the laboratories. *See Appendix A for copy of QA Manual.* | the results will be reviewed for potential impact on data usability and qualified accordingly.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 3 | Overall | The method detection limit (MDL) studies were not consistent for each analysis and were incomplete at the time of the audit for all analyses. A SOP should be developed which outlines the procedures and requirements for MDLs studies. Each method performed should have a spreadsheet documenting the MDL results and the subsequent PQL to be used for the project. **(M) (C – resolve immediately)** | All MDLs are complete. The only nonconformance was with Explosives analysis. See comments below for specific issues.<br><br>**Onsite Comment:** Onsite has a SOP for conducting MDL studies and the studies that we have conducted are consistent with that SOP. Spreadsheets are available that list the MDL and the PQLs. *See Appendix B and C.* | All MDL studies were complete for each method and each matrix before samples of that matrix were analyzed. All MDL studies were performed according to SW-846 requirements for the instruments set up specifically for this project, and were compliant with the requirements of the QAPP, with the exception noted (refer to comment 56, MDL for SW8330 not less than ½ PQL).<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 4 | Overall | The instrument maintenance, balance calibration, and refrigerator logbooks were not shipped with the instruments for this project. New logbooks were initiated in most areas (see specific findings); however, the type of information recorded, criteria for acceptance and long term historical information was lost. It is recommended that a SOP be developed and implemented to handle this situation and ensure documentation of the information. **(m) (C)** | Resolved<br><br>**Onsite Comment**: All logbooks were available for review and have been updated to indicate the specific acceptance criteria for this project. Onsite typically routinely keeps all original data with the specific project from which it was generated. It is not uncommon for new logbooks to be created for balances, refrigerators, and other equipment on a project by project basis so the project data can be collectively filed and retrieved. | The issue was resolved. Acceptable documentation exists for all equipment used for this project. Such documentation will be included in the final project data packages, as appropriate, where it will be reviewed in the data validation process.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| | | | Fixed based labs do not have this issue since they are stationary and the equipment is the same from project to project. *See Appendix D, H, J, K, L, M, N, and O for copies of logbooks.* | |
| 5 | Overall | The procedures for peer and secondary reviews were inconsistent throughout the analytical departments. All peer reviews and secondary reviews should be documented with a checklist which specifies the criteria reviewed, the initials of the reviewer, and the date of review. **(M) (C)** | All data is marked as preliminary. Secondary review will be performed and documented by the QA Manager, Chris Amstutz.<br><br>**Onsite Comment**: All final data will undergo final review by the project QA Officer . The analysts are familiar with the project requirements and corrective action procedures. Project specific checklists are available and will be utilized during the final review process. *See Appendix E for summary of Checklist Items.* | The audit took place before secondary review could take place. Peer and secondary review was never in question. Such review is performed after initial data are generated and in time for corrective actions if appropriate. Complete review of all data must occur before finalization of the data product.<br><br>The laboratory has implemented appropriate review procedures as specified in the QAPP. This process insures that all data will meet the requirements of the QAPP, and that the laboratory implements corrective actions and non-conformance reports prior to release of any final data. All data will be further reviewed for compliance in the data validation process. The multiple review process insures that all reported data are usable or appropriately qualified, and legally defensible.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 6 | Overall | The procedures for documenting the analysis run logs were inconsistent between analysts. Analytical run logs should contain the following information: instrument identification, | This procedure is still inconsistent between analytical departments. All information will be maintained in binders and paginated. Upon completion of this project Onsite will | No compromise of data integrity is indicated for the run logs. The format of storage for the run logs has been updated to meet the audit comments. The format of all run logs for this project meet method and QAPP requirements, |

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| | | analysis method, date and time of analysis of project samples and the associated laboratory QA/QC samples (initial calibration, continuing calibration, method blank, matrix spikes, etc.). This information should be kept in a bound notebook. The instrument computer print outs can be taped into notebooks to fulfill this requirement. All standard identifications must be clearly documented for traceability to the reference materials. **(M) (C)** | place the printouts in documents as described in comment.<br><br>**Onsite Comment:** The run logs for this project are available in paginated project binders. In addition, the run copies of all run logs will be stored in project files. The run logs list every sample analyzed along with the QA/QC samples such as continuing calibrations, method blanks, laboratory control spikes, and matrix spikes that were analyzed with the field samples. The required data is available and only needed to be packaged to meet this requirement. *Run logs are provided in each hard copy deliverable package and also in Appendix O.* | allowing data validation verification of instrument ID, date, time, sample ID, dilution, and all other parameters required to ensure legal defensibility of the data. If any non-compliance is identified in the data validation process, the results will be reviewed for potential impact on data usability and qualified accordingly.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 7 | Overall | The Corrective Action form used by the laboratory was inadequate to document nonconformances, impact of the nonconformance, corrective action implemented, and resolution of the nonconformance. The Corrective Action form should contain the following, as applicable – department or analysis area initiating the form, a detailed description of the nonconformance, name of project, preparation batch, analysis batch, originator's signature, supervisor's signature, date, detailed description of | The Corrective Action form has been revised. The laboratory will need to set up a tracking system to monitor initiation and resolution of Correction Action Reports.<br><br>**Onsite Comment**: Onsite has always utilized a Corrective Action form to document non-conformance issues. The form when completed becomes part of the project file. The form has been updated for this project to | All incidences requiring corrective action are documented and discussed with the Earth Tech project chemist. Appropriate corrective actions have been taken including re-extraction and/or reanalysis of samples, as appropriate. All data will be thoroughly reviewed for compliance and acceptability in the data validation process, especially with regard to the need for corrective actions. All corrective action forms will be examined to verify compliant follow-up.<br><br>The format and tracking of the corrective |

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| | | the corrective action implemented or planned, responsible person's signature, date, if client notification is necessary, detailed description of the corrective action verification or resolution, QA staff signature, and date of closure. All Corrective Action forms should be given a distinct unique number or identification and be tracked to systematic problems and documentation of completion. **(M) (C)** | include the requested information. In addition to the updated form, a numbering system has been devised to track the forms. *See Appendix F for Corrective Action Log.* | action forms do no affect the quality or usability of the data. Upon review, some data may require qualification, which is normal for any field sampling effort. Note that not all corrective actions result in unqualified data, even when performed according to the requirements of the QAPP. |
| 8 | Overall | The laboratory did not have a QA Manager/Officer identified for this project. The current QA Manager was on travel out of the country. The QA Manager or his/her designee must be identified for the project. The QA Manager must be sufficiently independent of cost and schedule considerations. The QA Manager should perform routine internal audits to ensure compliance with the QA Manual and project-specific requirements. **(M) (C)** | Resolved.<br><br>**Onsite Comment**: The Quality Assurance Manager originally specified for this project was unavailable at the time of project initiation. Chris Amstutz of Onsite has assumed the role of QAM. Chris Amstutz has over 19 years of laboratory experience and has been involved with numerous government and commercial clients in both a Laboratory Manager role and Quality Assurance role. He has extensive experience with explosives and acted as the QAM for work Onsite recently completed at the Joliet Army Ammunition Plant where over 2500 samples were analyzed for | Due to the rapid start-up of the project, the Quality Assurance Manager, Chris Amstutz, was still at Joliet at the beginning of the project. However, all analysts were highly experienced at setting up and running the mobile laboratory for US ACOE projects, and managerial oversight was provided by Peter Balas, a company principal, also highly experienced at setting up mobile laboratory operations. Upon arrival, Chris Amstutz reviewed all aspects of the operation, and implemented all actions necessary to assure complete compliance with the project QAPP and audit findings.<br><br>The resolution to this comment helps to assure an acceptable data product. Chris Amstutz is working closely with the Earth Tech project chemist to ensure that all final data packages meet all requirements for legally defensible data. |

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| | | | explosives. | |
| 9 | Overall | The procedures and documentation for the electronic data tapes and disks was not consistently performed. A SOP for these procedures should be developed and implemented for all analytical areas. **(M) (C)** | In process<br><br>**Onsite Comment:**<br><br>A SOP has been developed and implemented in all analytical areas. *See Appendix B for all SOPs.* | Electronic data for all applicable methods is stored on the instrument specific computer hard drive and backed up to tape or Zip disks. These data are then archived and stored in a secure location, and will be available for inspection or audit upon request. This complies with EPA guidelines for the maintenance of GC and GC/MS electronic file archives.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 10 | Overall | The standards were labeled with concentration and type of standard. All standards should be labeled with the standard identification, concentration, date of preparation, and the identify of the person preparing the standard. **(m) (C)** | Expiration date should also be included on standard labels.<br><br>**Onsite Comment**: Onsite routinely labels standards in order to accurately track the source and concentration of the standards. Logbooks are routinely used to document the standards preparation. Onsite has updated the labels to include the expiration date. No out-of-date standards have been used for the project. | Additional information has been added to the labels as requested by the auditor. All standards used for this project have been labeled in a manner such that traceability to the original standard with a certificate of analysis is possible. All such documentation has been made available at the site, will be included in the final data product, and will be reviewed in the data validation process.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 11 | Overall | PQL check standards were analyzed once after initial calibration. PQL check standards should be at a concentration at or near the PQL. These check standards should be analyzed daily to confirm sensitivity | PQL checks are not performed at this time.<br><br>**Onsite Comment**: Daily PQL checks are not specified in the project QAPP or in the respective methods. | Daily PQL checks are not a specified requirement in the project QAPP or in the US EPA Methods. The auditor reviewed this QAPP before project start-up and all comments were incorporated, but no request for daily PQL checks was made. PQL checks |

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| | | of the instrument. **(M) (C)** | However daily PQL checks have been used for Method 8330 for this project. *See Appendix G for the results of the daily checks that were performed.* | are analyzed for every method after initial calibration. Daily PQL checks have been analyzed for the explosives analyses by SW8330 throughout the project, which exceeds specified requirements.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 12 | Overall | Manual integration was handled differently in each analytical area. The procedures should be as follows for manual integration – the unaltered chromatographs should be attached to the altered chromatographs, the initials of the analyst, date, and reason for the manual integration must be documented on the raw data. **(M) (C)** | This procedure needs to be implemented.<br><br>**Onsite Comment:** All analysts have been instructed to include a copy of the original data and reprocessed data (manual integration) in all data packages as they have for previous projects. The chromatograms will be dated, initialed, and documented with the reason for manual integration. Each manual integration will be reviewed in the peer review process. *Manual integration documentation can be found in the data packages where applicable.* | All manual integrations will be reviewed according to the format specified in the auditor's comments during the secondary review process. This is the routine timeframe for manual integration review. All manual integrations will be printed, reviewed, initialed and dated, and included in the project files. These printouts will be included in all EPA Level IV data packages and reviewed in the data validation process. These procedures meet EPA guidelines for the review and maintenance of GC and GC/MS manual integration documentation.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 13 | Log In | The Sample Log In SOP used was out of date and inconsistent with the field laboratory procedures currently being performed by the staff. The SOP should be updated to accurately reflect the current procedures. **(M) (C)** | Resolved<br><br>**Onsite Comment:** The SOP for Sample Log-In has been updated to include the specific requirements of this project. *See Appendix B.* | The issue was resolved and sample log-ins prior to resolution have been reviewed. There is no indication that the initial issue will affect the quality or usability of the data. |
| | | Due to the volume of samples | Resolved. | All samples were kept on ice throughout the |

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| 14 | Log In | received, some samples were stored in coolers upon receipt. The temperature of the samples in cooler was not measured or recorded. The cooler receipt form required in the project QAPP was not being used by the laboratory. The temperature of the samples upon receipt should be measured and recorded on the cooler receipt form for each cooler. All samples should be placed in cold storage refrigerators. **(M) (C)** | **Onsite Comment**: The samples stored in coolers were kept on ice and the ice replaced on a daily basis. The Cooler Receipt form has been completed for all samples received for the project. Additional refrigeration has been obtained for sample storage and to ensure that samples are segregated from the sample extracts. *See hard copy packages for completed Cooler Receipt Forms.* | period in question. Samples were extracted or transferred to more permanent refrigeration without reaching temperatures at any point that would affect sample results or require data qualification according to EPA guidelines.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 15 | Log In | All bottles, sample containers, coolers, and chain-of-custody (COC) forms are supplied by Earth Tech. The coolers and COCs do not have unique numbers or identifications. To ensure traceability all coolers and COCs should have unique numbers or identifications. **(m) (C)** | Resolved<br><br>**Onsite Comment: T**his is typically the responsibility of the sample collection team. Chain-of-custody (COC) numbers are now assigned by Onsite to each COC when samples are received and the COC number logged in the sample-receiving logbook. The coolers have also been labeled and the Cooler number recorded on the COC. Earth Tech is in the process of numbering all COCs. *See data packages for assigned COC numbers.* | Coolers are delivered directly from the field sampling team to the onsite laboratory. Therefore, numbering and tracking of coolers was considered unnecessary. Earth Tech is numbering all COCs for the project.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 16 | Log In | There is no evidence of secondary review of all documents and logbooks by someone other than the person generating the documents. It is recommended that the forms are updated to include signature and date | All data is marked as preliminary. Secondary review will be performed and documented by the QA Manager, Chris Amstutz. | All documents will receive secondary review, including signature and date for each page of logs.<br><br>Please refer to the response for comment #5. |

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| | | lines to document peer review. **(m) (C)** | **Onsite Comment:** All logbooks will undergo secondary review by the project QA Officer. Where possible the logbooks have been updated include signature and date lines. At a minimum each page will be initialed and dated. *See Appendix D.* | No effect on the quality or usability of the data is expected, pending data validation. |
| 17 | Log In | The thermometer used for recording temperature was not calibrated with an NIST thermometer. All thermometers used in the laboratory must be calibrated with a NIST thermometer. **(M) (C)** | The staff needs to provide links with the thermometers in the refrigerators to complete traceability. The NIST thermometer should be certified annually. The NIST thermometer was calibration in 1998.The NIST thermometer needs to be re-certified.<br><br>**Onsite Comment:** The calibration of all thermometers has been checked with a NIST thermometer and recorded in a bound notebook. Each thermometer has been assigned a unique number and the number recorded in the corresponding logbook. The calibration of the NIST thermometer has been re-certified and the thermometer calibration was unchanged. *See Appendix H for a copy of the recertification certificate and thermometer calibration logbook.* | Traceable linkage to recertified NIST thermometers is complete and documented.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 18 | Log In | The records available did not clearly confirm the notification to the client when samples were received broken, warm, with insufficient volume, or other nonconformances. All | This procedure still needs to be implemented. Cooler receipt forms will be used to document this issue. | Samples are received directly from the sampling team, usually within 4 hours of collection, so temperature non-compliance is not an issue. Broken containers or other non-conformances were communicated directly to |

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| | | nonconformances identified during log in should be recorded on the cooler receipt forms. **(m) (C)** | **Onsite Comment:** Earth Tech has been appraised of all sample receipt problems through communication with the field team leader throughout the duration of the project. Cooler receipt forms are being utilized and any and all nonconformances are documented on the form. *See hard copy packages for completed cooler receipt forms.* | the field team leader so collection of additional sample could be performed if necessary. There were no lost sample analyses due to non-conformances. Appropriate forms have been implemented, and will be included for all samples in the final data product.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 19 | VOCs | The VOCs SOP was current (December 1999); however, the SOP did not address the Arcon autosampler, preservation and spiking requirements. The laboratory manager (Peter Balas) and the analyst were not intimately familiar with the EPA method requirements. The SOP should be revised to contain information regarding the instrumentation in use. All soil samples must be analyzed or chemically (acid or methanol) preserved within 48 hours from time of collection. The holding time is 14 days after collection if the sample is preserved. Analytical surrogate and matrix spiking must be introduced into the soil prior to sample extraction, by water or solvent. The SOP should be revised to include these requirements. **(M) (C)** | This SOP is still under revision. It is important to note that the laboratory is proposing to analyze all samples within 48 hour holding time without preservation. This includes re-analyses for failed QC results.<br><br>**Onsite Comment:** The SOP has been updated. The Arcon sampler was requested by the client to be utilized for this project after Onsite had mobilized to the site and the SOP has been updated accordingly. All samples have been analyzed within 48 hours including reanalyses where applicable. *See Appendix B.* | The SOP is available for review. All samples were analyzed within 48 hours according to the requirements of the method and project QAPP.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| | | MDLs were not available for review. | Resolved | MDL studies were complete and |

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| 20 | VOCs | MDLs must be completed and accurately documented prior to analysis of any samples. **(M) (C)** | **Onsite Comment:** MDLs have been conducted prior to sample analysis and were available for review during the follow-up visit. *See Appendix C.* | documentation is available.<br><br>Please refer to the response for comment #3.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 21 | VOCs | The analyst had difficulty tracing the standards back to the certificates of analysis. There was no established system for filing certificates of analysis or recording receipt of standards used for this analysis. A SOP describing standard filing systems and standard preparation and labeling should be established for all analytical methods. **(m) (C)** | Resolved.<br><br>**Onsite Comment:** Onsite maintains copies of all standard certificates either in the Onsite Laboratories or at the Corporate office. Onsite has in place a SOP for preparation and tracking of standards that has been revised for this project. *See Appendix I for copies of the certificates.* | All standards used for this project are traceable to the original standard and certificate of analysis.<br><br>Please refer to the response for comment #10.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 22 | VOCs | The laboratory runs a practical quantitation limit (PQL) check standard once during initial calibration or at the beginning of a project. A PQL check standard should be analyzed daily to confirm sensitivity is not lost during analysis of samples. **(M) (C)** | This procedure is not currently being implemented.<br><br>**Onsite Comment:** As specified in the QAPP PQL checks have been performed following the initial calibration curve. The QAPP utilized for this project did not specify PQL checks as a daily requirement for this project. | Daily PQL checks are not a specified requirement in the project QAPP or in the US EPA Methods.<br><br>Please refer to the response for comment #11.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 23 | VOCs | The raw data for the calibration curve was not available in hard copy for review for this analysis. All raw data should be printed out and filed in a consistent manner for easy review and | Resolved.<br><br>**Onsite Comment**: The raw data for the calibration curve was available during the follow-up visit. *See hard* | The issue was resolved. No compromise of data integrity is indicated and acceptable documentation is available at the location for each instrument. |

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| | | archival. **(m) (C)** | *copy packages for the calibration curves.* | No effect on the quality or usability of the data is expected, pending data validation. |
| 24 | VOCs | The analyst was not clear whether reporting tentatively identified compounds (TICs) was required for this project. This should be clarified with the Earth Tech prior to reporting final data. **(m) (C)** | Resolved<br><br>**Onsite Comment:** Onsite has consulted with the client on this issue and will be reporting TICs for this project. *See hardcopy packages for TIC data.* | The issue was resolved. TICs will be reported if found.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 25 | VOCs | Certification of the hood flow rate was not documented. The flow of the hoods should be periodically checked and recorded. **(m) (C)** | Flow rates have been verified and documented. Hoods have not been certified by an independent technician service.<br><br>**Onsite Comment:** The hood flow rates have been verified and Onsite is in the process of scheduling an independent verification of the flow rates. | Hood flow rates have been documented by Onsite. Independent verification will be performed and the results made available.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 26 | VOCs | The water used for preparation of standards and blanks was not documented to be organic-free. The QAPP specifies that the water meets specific criteria and be monitored for acceptability and recorded in a logbook. The laboratory should document the lot numbers, criteria for acceptance, and analysis of water used within the area. **(m) (C)** | The laboratory still needs to document the lot of water used and establish criteria for acceptance in SOP.<br><br>**Onsite Comment:** The source of water used in the preparation of blanks and standards has been documented to be organic-free. A logbook has been set up to track and document the blank water utilized and to verify that it is free of organic contaminants. *See Appendix J for* | All source water has been documented as free of contamination.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| | | | *Blank Reagent Logbook.* | |
| 27 | VOCs | The analyst had difficulty tracing the standards back to the certificates of analysis. There was no established system for filing certificates of analysis or recording receipt of standards used for this analysis. A SOP describing standard filing systems and standard preparation and labeling should be established for all analytical methods. **(M) (C)** | Resolved.<br><br>**Onsite Response:** This comment is the same as #21 above and has previously been addressed. | All standards used for this project are traceable to the original standard and certificate of analysis.<br><br>Please refer to the response for comment #10.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 28 | VOCs | It was not documented that the balance used was calibrated and checked within one year by a certified technician. The daily calibration with Class S weights was not recorded. This information should be documented for each balance used within the laboratory. **(m) (C)** | The balance was certified 12/14/99, after the balance had been used. However, the level of correction with less than 0.03 grams and the criteria for weighing in 0.5 accuracy. The laboratory needs to document acceptance criteria for daily calibration.<br><br>**Onsite Comment:** Onsite routinely performs a daily calibration check of all balances. The daily checks have been within the required limits. Balances are typically calibrated annually by a certified technician. All balances have been re-certified during the course of this project. The certification process provided documentation that indicated the balances were within calibration. Re-certification documentation on the balances is available for review. *See* | The balances were recertified and well within specified criteria.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| | | | *Appendix N for balance logbook and certification record.* | |
| 29 | VOCs | There was no evidence of secondary review of all documents and logbooks by someone other than the person generating the documents. It is recommended that the forms are updated to include signature and date lines to document peer review. **(m) (C)** | All data is marked as preliminary. Secondary review will be performed and documented by the QA Manager, Chris Amstutz.<br><br>**Onsite Comment**: All logbooks will undergo secondary review by the project QA Officer. Where possible the logbooks have been updated include signature and date lines. At a minimum each page will be initialed and dated. *See Appendix D.* | All documents will receive secondary review, including signature and date for each page of logs.<br><br>Please refer to the response for comment #5.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 30 | VOCs | The analytical run log instrument printouts were placed in a three-ring binder. The standards and initial calibration were not clearly cross-referenced. This information should be kept in a bound notebook. The instrument computer print outs can be taped into notebooks to fulfill this requirement. All standard identifications must be clearly documented for tracing. **(m) (C)** | All information will be maintained in binders and paginated. Upon completion of this project Onsite will place the printouts in documents as described in comment.<br><br>**Onsite Comment:** The analytical run log sequences have limited space for surrogate standard identification. Each data printout has a unique file number that enables the user to accurately trace back to the standards used and provide a cross-reference to the initial calibration files utilized for data reduction. *See hard copy packages for run logs and Appendix O.* | The issues in the original comment were resolved by the time of the follow-up audit. All standards for all calibrations for this project are traceable. The format of storage for the run logs has been updated to meet the audit comments.<br><br>Please refer to the response for comment #6.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| | | The manufacturer's operating | Resolved | All manufacturers operating manuals were |

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| 31 | VOCs | manuals were not readily available for all instruments. These manuals should be stored and shipped with the instruments or additional copies should be available in the laboratory. **(m) (C)** | **Onsite Comment:** All manufacturers operating manuals were in an adjacent mobile laboratory and have since been moved to the GC/MS laboratory. | available. No improper use or servicing of any instrument took place at any time.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 32 | VOCs | Sample log in and VOCs analysis are performed in the same trailer. There was limited workspace for both operations and storage of the samples and coolers. It is recommended that the operation be evaluated to provide adequate space for all required operations. **(r) (C&R)** | An extra sample refrigerator was brought on site to handle sample storage.<br><br>**Onsite Comment:** Onsite has added two more refrigerators to handle the sample storage necessary for this project. | The issue was resolved.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 33 | VOCs | The maintenance schedules were not specified in the SOP or documented in the maintenance logbook for all instruments. The requirements for instrument preventative maintenance and schedules should be documented in the SOP and recorded in the maintenance logbooks for all instruments. **(m) (C)** | This procedure requires corrective action. All logbooks should be bound and the system set-up (instrument identification, detectors, and auto-samplers) should be specified in the logbook.<br><br>**Onsite Comment:** Maintenance schedules for instrumentation are typically not mentioned in analytical method SOPs. Onsite typically defaults to the manufacturers recommendations for preventative maintenance as outlined in the instrument manuals. The maintenance logbooks have been revised accordingly. *See Appendix K for the maintenance logbooks.* | There is no indication that instruments were not set-up or maintained adequately. All systems have been properly maintained according to manufacturers recommendations. All analysts are highly experienced and knowledgeable in the maintenance and servicing of the instrumentation. The maintenance logbooks have been revised to meet the format suggested by the auditor.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| 34 | VOCs | The laboratory does perform corrective action for precision failure (relative percent difference – RPDs) for matrix spikes. Re-analysis should be performed for RPD failures to confirm matrix effects versus laboratory error. **(m) (C)** | Resolved<br><br>**Onsite Comment**: This is a QAPP requirement and has been incorporated into our procedures for this project. Soil concentrations can be highly variable. Typically an LCS/LCSD is a better indication of analytical precision on a batch basis since they are free from matrix interference that may be present | This issue has been resolved. The data were reviewed and re-extractions and/or reanalyses were performed if appropriate. The data will be reviewed for compliance in the data validation process. Results associated with RPDs greater than control limits specified in the QAPP, if found, will be qualified according to the QAPP.<br><br>The effect on the quality of the data is not expected to be significant. |
| 35 | Sample Prep | Certification of the hood flow rate was not documented. The flow of the hoods should be periodically checked and recorded. **(m) (C – see comment # 25 above)** | Flow rates have been verified and documented. Hoods have not been certified by an independent technician service.<br>**Onsite Comment**: The hood flow rates have been verified and Onsite is in the process of scheduling an independent verification of the flow rates. | Hood flow rates have been documented by Onsite. Independent verification will be performed and the results made available.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 36 | Sample Prep | The solvent used for preparation of field and quality control samples was not documented to be organic-free. The laboratory should document the lot numbers and analysis of water used within the area. **(m) (C)** | This issue requires corrective action as stated in original comment.<br><br>**Onsite Comment:** The source of water used in the preparation of blanks and standards has been documented to be free of the analytes of interest. A logbook has been set up to track and document the blank water utilized and to verify that it is acceptable for use. *See Appendix J.* | All source water has been documented as free of contamination.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| | | The analytical reagents did not | Resolved | The issue has been resolved. |

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| 37 | Sample Prep | consistently have the date the received recorded. All reagents and standards should have the date received documented. **(m) (C)** | **Onsite Comment**: A reagent receipt logbook has been set up for this project and reagents received documented. All reagents are also dated and initialed upon receipt. *See Appendix L for reagent log book.* | No effect on the quality or usability of the data is expected, pending data validation. |
| 38 | Sample Prep | The standard preparation and tracking was not maintained in the mobile laboratory. All standards should be tracked in a document logbook. **(C – consider comment as Major)** | This still needs to be addressed.<br><br>**Onsite Comment**: All standards are tracked in a logbook and the logbook was available for review during the follow-up visit. *See Appendix M for the standards logbooks.* | All standards used for this project are traceable to the original standard and certificate of analysis. Standards can be tracked in a logbook which is available at the site. The documentation will be included in the final data product and will be reviewed in the data validation process.<br><br>Please refer to the response for comment #10.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 39 | Sample Prep | The analyst had difficulty tracing the standards back to the certificates of analysis. There was no established system for filing certificates of analysis or recording receipt of standards used for this analysis. A SOP describing standard filing systems and standard preparation and labeling should be established for all analytical methods. **(M) (C)** | This issue still needs to be addressed.<br><br>**Onsite Comment:** Onsite maintains copies of all standard certificates either in the Onsite Laboratories or at the Corporate office. Copies of the certificates have been added to the project files. Onsite has in place a SOP for preparation and tracking of standards that has been revised for this project. *See Appendix B and I.* | All standards used for this project are traceable to the original standard and certificate of analysis.<br><br>Please refer to the response for comment #10.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| 40 | Sample Prep | The preparation logbook had more than one batch per page. It is recommended that each batch be documented separately on one page. **(r) (C&R)** | This procedure has not been implemented. There was evidence of open batching for 13-14 December 1999. All batches must contain appropriate QC samples (method blank, LCS, and MS/MSD). This is unacceptable. **(M)**<br><br>**Onsite Comment:** Extraction logbooks have been set-up for 8310, 8015, and 8330 analyses. The logbook provides one page per extraction batch. All batches are being reviewed to verify that adequate QC has been added. If inadequate QC is noted samples will be re-extracted and /or reanalyzed, or a NCR will be initiated. *See hardcopy packages for copies of applicable extraction logbooks and Appendix D.* | The logbook has been revised to include one batch per page. All batches have been reviewed by the QA Officer. Re-extractions and reanalyses were performed on samples in the non-compliant batch for December 13-14, 1999, as appropriate, with compliant QC. All sample results were the same as in the original analyses.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 41 | Sample Prep | There was no evidence of secondary review of all documents and logbooks by someone other than the person generating the documents. It is recommended that the forms are updated to include signature and date lines to document peer review. **(m) (C)** | All data is marked as preliminary. Secondary review will be performed and documented by the QA Manager, Chris Amstutz.<br><br>**Onsite Comment**: All logbooks will undergo secondary review by the project QA Officer. Where possible the logbooks have been updated include signature and date lines. At a minimum each page will be initialed | All documents will receive secondary review, including signature and date for each page of logs.<br><br>Please refer to the response for comment #5.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| | | | and dated. *See Appendix D.* | |
| 42 | Sample Prep | The thermometer used for recording temperature was not calibrated with an NIST thermometer. All thermometers used in the laboratory must be calibrated with a NIST thermometer. **(m) (C)** | The staff needs to provide links with the thermometers in the refrigerators to complete traceability. The NIST thermometer should be certified annually. The NIST thermometer was calibration in 1998.The NIST thermometer needs to be re-certified.<br><br>**Onsite Comment**: The calibration of all thermometers has been checked with a NIST thermometer and recorded in a bound notebook. Each thermometer has been assigned a unique number and the number recorded in the corresponding logbook. The calibration of the NIST thermometer has been re-certified and the thermometer calibration was unchanged. *See Appendix H.* | Traceable linkage to recertified NIST thermometers is complete and documented.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 43 | Sample Prep | The maintenance logbooks were not shipped with the refrigerators or sonicator used in this area. New logbooks were not initiated for all instruments and apparatus in the area. It is recommended that a SOP be developed and implemented to handle this situation and ensure documentation of the information. **(m) (C)** | Resolved for all instruments except for the TurbVap.<br><br>**Onsite Comment**: Onsite does not maintain a maintenance log for refrigerators. If a refrigerator malfunctions it is immediately replaced. A logbook has been set up for the TurboVap sonicator used in this area. To date the surrogate recoveries for samples processed have been within acceptable limits. *See* | This issue has been resolved. For the TurboVap sonicator, a maintenance log-book has been initiated. The sonicator did not require maintenance during the course of this project, and all surrogate recoveries for samples extracted using the TurboVap have been within acceptable limits, indicating acceptable performance.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| | | | *Appendix K for logbook.* | |
| 44 | Sample Prep | The laboratory concentrates down to the exact volume used for analysis. The extracts are placed on the instruments in crimp top vials for analysis. The extracts could potentially concentrate or biodegrade over night as the resonance time on the autosampler could range from 2 hours to 10 hours. The laboratory analysts use surrogate performance to monitor the extract degradation. The analysts would re-extract and re-analyze based on QC failure. This could potentially lead to delays in reporting of final results and increased labor for corrective action due to surrogate or matrix spike failure. **(o) (C&R – no immediate response necessary)** | No action required.<br><br>**Onsite Comment**: The procedures that Onsite utilizes are well within the constraints of the method and the instrumentation used. The comment would seem to suggest that we not utilize the autosampler provided with the instrument or that we inject one extract at a time after letting it warm to room temperature. Both are impractical. | No action required.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 45 | TPH | The SOP referenced EPA Method 8015M. The SOP for TPH analysis needs to be updated to reflect the current requirements for EPA Method 8015B. **(M) (C)** | This SOP is still under revision.<br><br>**Onsite Comment**: The SOP has been updated to reflect the current requirements of EPA Method 8015B, which are similar to those of the SOP for CA LUFT 8015. *See Appendix B.* | The analyses were performed as required in the QAPP and in the method. The SOP has been updated.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 46 | TPH | The SOP did not stipulate the calibration curve standards used for this analysis. This was inconsistent with the other SOPs reviewed. This information should be included in the | This SOP is still under revision.<br><br>**Onsite Comment:** The calibration curve standards were provided in Section 8.1.1 of the SOP and have | The analyses were performed as required in the QAPP and in the method using acceptable levels of standards. The SOP has been updated. |

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| | | SOP. **(m) (C)** | been updated. *See Appendix B.* | No effect on the quality or usability of the data is expected, pending data validation. |
| 47 | TPH | The laboratory did not have adequate space for dish washing. Dish washing for the extractions were performed outside the trailer on the sidewalk. No protective plastic was placed under the buckets used for collection of spilled dishwater. If the weather worsens, this may not be adequate. **(o) (C&R)** | Resolved<br><br>**Onsite Comment**: Onsite collects all wash waters into storage containers. | This issue has been resolved.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 48 | TPH | Certification of the hood flow rate was not documented. The flow of the hoods should be periodically checked and recorded. **(m) (C)** | Flow rates have been verified and documented. Hoods have not been certified by an independent technician service.<br><br>**Onsite Comment**: The hood flow rates have been verified and Onsite is in the process of scheduling an independent verification of the flow rates. | Hood flow rates have been documented by Onsite. Independent verification will be performed and the results made available.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 49 | TPH | The thermometer used for recording temperature was not calibrated with an NIST thermometer. All thermometers used in the laboratory must be calibrated with a NIST thermometer. **(m) (C)** | The staff needs to provide links with the thermometers in the refrigerators to complete traceability. The NIST thermometer should be certified annually. The NIST thermometer was calibration in 1998. The NIST thermometer needs to be re-certified<br><br>**Onsite Comment**: The calibration of all thermometers has been checked with a NIST thermometer and | Traceable linkage to recertified NIST thermometers is complete and documented.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| | | | recorded in a bound notebook. Each thermometer has been assigned a unique number and the number recorded in the corresponding logbook. The calibration of the NIST thermometer has been re-certified and the thermometer calibration was unchanged. *See Appendix H.* | |
| 50 | TPH | The manufacturer's operating manuals were not readily available for all instruments. These manuals should be stored and shipped with the instruments or additional copies should be available in the laboratory. **(m) (C)** | Resolved<br><br>**Onsite Comment**: The manuals are now located adjacent to the instrument. | All manufacturers operating manuals are available. No improper use or servicing of any instrument took place at any time.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 51 | TPH | The analyst had difficulty tracing the standards back to the certificates of analysis. There was no established system for filing certificates of analysis or recording receipt of standards used for this analysis. A SOP describing standard filing systems and standard preparation and labeling should be established for all analytical methods. **(M) (C – immediate response required)** | This issue still needs to be resolved for this department.<br><br>**Onsite Comment**: Onsite maintains copies of all standard certificates either in the Onsite Laboratories or at the Corporate office. Onsite has in place a SOP for preparation and tracking of standards that has been revised for this project. *See Appendix I for copies of the certificates.* | All standards used for this project are traceable to the original standard and certificate of analysis.<br><br>Please refer to the response for comment #10.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 52 | TPH | The laboratory staff was unclear regarding the quantitation ranges and fuel standard for motor oil range organic TPH. Discussions took place between the USACE representative, the Earth Tech Chemist, and Onsite | Resolved. Onsite will provide the quantitation ranges.<br><br>**Onsite Comment**: The quantitation ranges for fuel standards has been clarified and the ranges will be added | The issue has been resolved. Appropriate quantitation ranges are used for each fuel.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| | | staff. It was determined that the laboratory would provide Earth Tech with the carbon ranges appropriate for the correct standards and stipulate these on the raw and final data packages. **(C – consider this a Major comment)** | to the data packages and final reports. | |
| 53 | TPH | There was no evidence of secondary review of all documents and logbooks by someone other than the person generating the documents. It is recommended that the forms are updated to include signature and date lines to document peer review. **(m) (C)** | All data is marked as preliminary. Secondary review will be performed and documented by the QA Manager, Chris Amstutz.<br><br>**Onsite Comment**: All documents and logbooks will undergo secondary review by the project QA Officer. The analysts are familiar with the project requirements and corrective action procedures. Project specific checklists are available and will be utilized during the final review process. Where possible the logbooks have been updated include signature and date lines. At a minimum each page will be initialed and dated. *See Appendix D.* | All documents will receive secondary review, including signature and date for each page of logs.<br><br>Please refer to the response for comment #5.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 54 | HPLC | The SOP for PAHs analysis was not available at the time of the audit. All SOPs should be readily available to the analysts performing the work on site. **(M) (C)** | Resolved<br><br>**Onsite Comment**: The SOP for PAH was available for review during the follow-up visit. *See Appendix B.* | The issue has been resolved.<br><br>Please refer to the response for comment #2.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| 55 | HPLC | The analyst had difficulty tracing the standards in the area back to the certificates of analysis. There was not an established system for filing certificates of analysis or recording receipt of standards used for this analysis. A SOP describing standard filing systems and standard preparation and labeling should be established for all analytical methods. **(M) (C)** | Resolved<br><br>**Onsite comment:** Onsite maintains copies of all standard certificates either in the Onsite Laboratories or at the Corporate office. Onsite has in place an SOP for preparation and tracking of standards that have been revised for this project. *See Appendix B and I.* | All standards used for this project are traceable to the original standard and certificate of analysis.<br><br>Please refer to the response for comment #10.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 56 | HPLC | The MDLs were not completely documented for all analyses. The analyst stated that retention times had not been established at the time of the audit; however, preliminary sample results were being reported. The information for MDLs and retention times must be completely documented and approved prior to analysis of samples. **(M) (C)** | The MDLs for amino-DNTs, tetryl, and 4-NT did not meet the PQL requirements in the QAPP. The analyst is reporting all data as preliminary. However, this issue needs to be resolved immediately.<br><br>**Onsite Comment**: The MDLs have been resolved and the data forwarded to Chris Davis of Earth Tech. All MDLs meet or exceed the levels specified in the QAPP with the exception of amino-DNT. The amino-DNTs co-elute and therefore the MDL is elevated. The MDLs are not less than one-half the PQL. However, daily PQL standards are analyzed to document sensitivity at the PQL. Onsite has performed an additional study that documents MDLs of one-half the PQL or less. *See Appendix C and G.* | Although MDL and retention time (RT) window studies were completed for each method and each matrix before samples of that matrix were analyzed, the MDL and RT were not fully evaluated immediately for each method. Analytical results provided before evaluation of the studies were reported as preliminary. The studies have been evaluated, and results are reported accordingly.<br><br>The SW8330 MDL studies were performed according to SW-846 method requirements. MDLs were compliant with the requirements of the QAPP, with the following exception:<br>All MDLs for SW8330 target analytes were at or below the PQL, but not less than ½ PQL. The MDLs for the amino-DNTs, which co-elute, have been summed and are therefore at 2X the PQL of the individual amino-DNTs.<br><br>The usability of the SW8330 data is not |

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| | | | | expected to be affected as the MDLs are valid and the resulting data are usable. Daily standards at the PQL have been analyzed for all SW8330 analyses for this project, and sensitivity and accuracy (±25%) have been documented. All PE sample results for explosives have met project accuracy goals.<br><br>In addition, the laboratory performed a second MDL study for the same instrumentation using lower concentrations of each analyte at the end of the project to document sensitivity at the PQL. The PQL specified in the QAPP for the SW8330 target analytes in soils is 0.4 mg/kg. The initial study was performed using standards at 1.6 mg/kg and the final study was performed using standards at 1.0 mg/kg. The results for the final MDL study indicate MDLs of 0.1 mg/kg for most analytes, 0.2 mg/kg for two analytes and 0.3 mg/kg for the summed amino-DNTs. These MDLs meet the QAPP requirement that MDLs be less than or equal to ½ the PQL. Although the second set of MDLs cannot be reported for the analyses performed prior to the follow-up MDL study, it demonstrates the ability of the laboratory to meet the PQL for the data set.<br><br>Data for all analyses are reported down to the MDL for all analytes for all methods, which will still apply to method SW8330 using the initial MDLs). The PQL may need to be raised to 2X the MDL for each SW8330 analyte, or |

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| | | | | results less than 2X the MDL may require the same general qualification as all data reported between the MDL and PQL, which are qualified as estimated (J).<br><br>The PQLs for the SW8330 target analytes are 1-4 orders of magnitude below the respective PRGs referenced as the action levels for decision making for this project (the TNT PRG in soil is 40X the PQL), therefore, the data still meet the DQOs for this project.<br><br>Thus, the reporting of SW8330 results with PQLs not greater than 2X the MDLs is not expected to significantly affect the legal defensibility or usability of the data. The data are expected to fulfill project DQOs.<br><br>A complete evaluation of the MDLs with respect to the DQOs will be made in the QCSR which will accompany the final data validation reports. |
| 57 | HPLC | There is no secondary review of the documents and logbooks by someone other than the person generating the documents. It is recommended that secondary review is implemented and the forms are updated to include signature and date lines to document peer review. **(M) (C)** | All data is marked as preliminary. Secondary review will be performed and documented by the QA Manager, Chris Amstutz.<br><br>**Onsite Comment**: All documents and logbooks will undergo secondary review by the project QA Officer. The analysts are familiar with the project requirements and corrective | All documents will receive secondary review, including signature and date for each page of logs.<br><br>Please refer to the response for comment #5.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

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| | | | action procedures. Project specific checklists are available and will be utilized during the final review process. Where possible the logbooks have been updated and include a signature and date lines. At a minimum each page will be initialed and dated. *See Appendix D.* | |
| 58 | HPLC | The manufacturer's operating manuals were not readily available for all instruments. These manuals should be stored and shipped with the instruments or additional copies should be available in the laboratory. **(m) (C)** | Resolved<br><br>**Onsite comment**: The operating manuals are now located adjacent to the instrumentation | All manufacturers operating manuals are available. No improper use or servicing of any instrument took place at any time.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 59 | HPLC | Due to space limitations, after analysis the sample extracts were stored in the hood. It is required that all extracts be stored properly. **(M) (C)** | Extracts and samples are stored together. Due to the potential for cross contamination, it recommended that extracts and samples be stored separately.<br><br>**Onsite comment**: Additional refrigeration has been obtained and the samples and extracts are stored separately. | The issue has been resolved. The potential for cross-contamination exists, but is not likely. In general, results have included a mix of non-detects and hits for SW8330 analyses, including significant hits, and all non-detects for SW8310 analyses, so the data indicate that cross-contamination from HPLC extracts should not be an issue. Note that if cross-contamination were to occur, the effect would be possible false positives or high bias resulting in over-estimation of contamination, so any effect on decision making would tend to be conservative.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| | | The analyst indicated that he was | The analyst is using a different | The use of the surrogate 1,2-dinitrobenzene |

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| 60 | HPLC | using QC limits from the previous project for evaluation of the data for this project. This is unacceptable. All analysts should be intimately familiar with the QAPP for this project and use the QC limits specified in the QAPP. **(M) (C)** | surrogate with tighter QC limits (70-130%) than the QAPP (65-135%). This deviation should be documented.<br><br>**Onsite Comment**: The surrogate used is consistent with the method and Onsite has used this surrogate at numerous ACOE sites that have explosives. The final reports will utilize the limits specified in the QAPP. | instead of 2,4-dinitrofluorobenzene was added to the QAPP 11/29/99, and the auditor was appraised of this change as the ACOE reviewer of the QAPP. Use of this compound as a surrogate does not affect the quality or defensibility of the data as this surrogate is in many ways more representative of the target analytes, is compliant with the method, and is considered acceptable to US EPA and US ACOE for this method.<br><br>The use of tighter control limits is acceptable according to standard US EPA and US ACOE protocols. When a laboratory has control limits not identical to those specified in a project specific QAPP, protocol requires use of the more stringent control limits. Project control limits have been implemented, are being used for corrective action, and will be reported with the final data.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 61 | HPLC | The analyst was not clear on what constituted an event that would require the initiation of a correction action form. All the analysts should undergo training in the corrective action procedures and requirements. **(M) (C)** | Resolved<br><br>**Onsite comments**: The Corrective action procedures have been reviewed with all the laboratory analysts | The issue has been resolved.<br><br>Please refer to the response for comment #7.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |
| 62 | HPLC | The laboratory manager and analyst were not clear about the criteria for | The freezer was marked with the criteria of –2 to –10 degrees Celsius. | The control limits have been updated. The unit is a refrigerator/freezer combination unit. |

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| | | the appropriate temperature storage for the standards for this analysis. The laboratory manager and analysts should be trained in the appropriate storage requirements for the standards. A reference table should be posted in the analytical areas for easy reference. **(m) (C)** | The criteria for the freezer should be –20 to –10 degrees Celsius.<br><br>**Onsite comment**: The control limits have been updated in the appropriate logbook. *See Appendix D for refrigerator logbooks.* | Prior to the audit, temperatures for the freezer were not logged. After the audit, the temperatures for the freezer were consistently logged at –10 to -16°C with no adjustment to the unit, indicating temperatures in the freezer were compliant and that standards were not stored in non-compliant temperatures.<br><br>No effect on the quality or usability of the data is expected, pending data validation. |

Onsite has made significant strides in improvement of their operation since the previous audit. However, it important to note that the SOPs and the explosives MDL issues should be resolved immediately.

If you have any questions please contact me by phone at (925) 335-0306 or by e-mail at [halsne2@earthlink.net].

Julia R. Halsne
Project Chemist

cc: John Esparza, Roger Henderson, Dave Elskamp, Maria Susbilla, Brad Call, Clinton Huckins (USACE)

MEMORANDUM

TO: Brian Weith, Earth Tech

FROM: Chris Davis, Project Chemist for the Benicia Tourtelot Project

CC: Peter Balas, Program Manager, Onsite

DATE: January 5, 2000

RE: Onsite Response Comments to the Earth Tech Audit – Benicia Project 12/99

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TO: Chris Davis, Earth Tech

FROM: Chris Amstutz, Quality Assurance Officer

CC: Peter Balas, Program Manager, Onsite

DATE: December 28, 1999 (Updated March 15, 2000 with References to Appendices [in italics])

RE: Response Comments to the Earth Tech Audit Report – Benicia Project 12/99

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The following responses are provided to the Earth Tech audit of the Onsite Environmental Laboratory facilities mobilized for the Benicia project. The audit findings are provided in their entirety along with ONSITE's responses.

December 15, 1999

Onsite Environmental Laboratories, Inc.
5500 Boscell Common
Fremont, CA 94538

Attn: Peter Balas, Program Manager

Subject: AUDIT REPORT

Audit Date: December 6 and 7, 1999

Audit Location: Onsite Environmental Laboratories, Inc.- Benicia, CA

Auditor: Janet Tsukamoto

Audit Scope: Mobile laboratory audit in compliance with the Earth Tech Draft Final Non-Ordnance and Explosives RI/FS QAPP for the Tourtelot Cleanup Project

Personnel Contacted: Peter Balas, Chris Cao, Doug Honzay, John Mough, Isabel Rivera, Michael Yurovsky

Audit Summary: An audit of the Onsite mobile laboratories was performed. The audit concentrated on analytical methods performed for the Tourtelot Cleanup Project, i.e., organics preparation, Total Extractable Petroleum Hydrocarbons by GC/FID, Volatile Organic Compounds by GC/MS, Polynuclear Aromatic Hydrocarbons, and Nitroaromatics and Nitroamines by HPLC, as well as sample receiving, and included interviews with laboratory personnel.

General Comments

The laboratory personnel interviewed seemed familiar and proficient with the analytical procedures and protocols. However, laboratory SOPs and the laboratory documentation (standard preparation and maintenance logbooks, MDL and RT window studies), refrigerator monitoring and calibration records (thermometer, balance and weights calibration certification) was either incomplete or unavailable during the audit. One copy of the Project QAPP was shared between the three trailers, and all personnel were not familiar with the requirements. At the time of the audit, the only sample analyses performed were for Nitroaromatics and Nitroamines by HPLC.

SOPs need to be up-to-date, all laboratory documentation should be better organized, and calibration records should be available on site. On the whole, the laboratory was not well organized.

**Onsite Response:** The referenced information has all been compiled and organized and is now available for review. The Onsite QAM has been updated *(Appendix A)* and copies of the QAM and project QAPP placed in each trailer. All required log books and maintenance logs are in place *(Appendix D and K)*. The balances and thermometers have been certified during this project *(Appendix H and N)*.

The SOPs have all been updated to reflect the requirements of this project *(Appendix B)*. The labs have been organized to perform the work as efficiently as possible. Calibration records have been printed and are available for each instrument *(see data packages)*.

**Earth Tech Response:** Response acceptable. The Earth Tech audit took place very early in the sampling event so corrective actions could be made in time for the analysis of most samples. All of the Onsite Laboratory analysts are highly trained, experienced, and familiar with the requirements of the US EPA Methods and for US ACOE projects. The staff were familiarized with the project QAPP, and SOPs and other documentation were updated and are current and available. Calibration records were stored

electronically for each instrument and have been printed and are available at each analytical station.

There is no indication in the audit report that analyses were performed in any way not in compliance with QAPP and method requirements. All data will be thoroughly reviewed for compliance with QAPP and method requirements in the data validation process. If any non-compliance is identified, the results will be reviewed for potential impact on data usability and qualified accordingly.

No effect on the quality or usability of the data is expected, pending data validation.

The following sections constitute the rest of the findings, concerns and recommendations grouped by area or subject.

Sample Receiving and Control (John Mough)

The following items and systems were reviewed: sample receipt; log-in; storage; tracking and anomalies; refrigerator monitoring and sample security.

Sample receiving and log-in are documented in a bound logbook. Temperature and pH are not verified upon receipt. Samples are received as soon as they are brought in from the field and are not cooled down [i.e, samples have not had time to cool to specified temperatures] at the time of receipt; preservatives are not required for the analytical methods employed for the project. The COCs are checked against the sample labels.

**Onsite Response:** At the time of the audit, samples were either placed in a refrigerator or kept in the coolers on ice. Additional refrigerators were brought to the site, so all samples were maintained at acceptable temperatures *(Appendix D)*. Sample receipt forms were also implemented *(Appendix D)*.

**Earth Tech Response:** Response acceptable. Most samples were brought in from the field so quickly that the samples did not have time to reach 2-6°C at time of sample receipt, but were kept on ice so sample receipt temperature is not of significance. All samples were kept cool by the laboratory throughout the period in question. Samples were transported and stored without reaching temperatures at any point that would affect sample results or require data qualification according to EPA guidelines. No effect on the quality or usability of the data is expected, pending data validation.

Findings:

1. The SOP for sample receiving was not available in the sample-receiving trailer.

**Onsite Response:** The SOP is now available in the sample-receiving trailer *(see Appendix B)*.

**Earth Tech Response:** Resolved. No effect on the quality or usability of the data is expected, pending data validation.

2. A discrepancy was found between the COC and the sample label on one sample in the 12/4/99 shipment. The discrepancy was resolved after verifying with the Earth Tech field personnel but the incidence was not documented. Anomalies must be properly documented. It is recommended that discussions also be noted in the comment section on the COC form.

   **Onsite Response:** Onsite has been utilizing the Sample Receipt Checklist to document problems with COC forms *(see Appendix D)*. In addition, Onsite is in daily communication with Earth Tech personnel regarding any issues with samples received.

   **Earth Tech Response:** The discrepancy was discovered and corrected indicating acceptable diligence in sample receiving. Proper documentation has been implemented. No effect on the quality or usability of the data is expected, pending data validation.

3. The final received time was not filled out on the COC of cooler #12 for the 12/4/99 sampling. All receiving information must be completely filled out on the COC.

   **Onsite Response:** Comment noted. All pertinent information will be recorded on the COC form *(see Appendix D)*.

   **Earth Tech Response:** Resolved. No effect on the quality or usability of the data is expected, pending data validation.

4. Sample log-in procedure is performed by only one person and there was no procedure in place to double check the labeling of sample containers in the lab to preclude mislabeling, i.e., mixing up containers.

   **Onsite Response:** The second level check is performed at the analyst level. The analysts are all provided a copy of the COC and they verify the assigned lab IDs with the Client Ids *(see Appendix B and D)*.

   **Earth Tech Response:** Response acceptable. No effect on the quality or usability of the data is expected, pending data validation.

5. Temperature monitoring of refrigerator 3A has not been documented. The last temperature recording was made on 6/25/98.

   **Onsite Response:** Refrigerator 3A was not functioning and was not used for sample storage for this project. All refrigerators and freezers used for sample and standards storage are monitored for temperature on a daily basis using a dedicated calibrated thermometer *(see Appendix D)*.

   **Earth Tech Response:** Response acceptable. The refrigerator in question was not used for sample storage for this project. No effect on the quality or usability of the data is expected, pending data validation.

6. The refrigerator thermometer calibration record was not available in the mobile lab. The record is kept at the Onsite Corporate office in Fremont.

**Onsite Response:** Thermometer calibration is now provided at the site and is available for review *(Appendix H)*.

**Earth Tech Response:** Resolved. No effect on the quality or usability of the data is expected, pending data validation.

7. The refrigerator thermometers are not tagged with the calibration due date.

**Onsite Response:** The thermometers were checked for calibration during this project and will be calibrated annually thereafter. The NIST certified thermometer was recalibrated during this project and all thermometers checked against it. There were not appreciable differences from expected values with the thermometers used for this project with the exception of one thermometer, which was discarded. The differences did not cause any temperature readings taken prior to recalibration to be non-compliant *(see Appendix H)*.

**Earth Tech Response:** Response acceptable. All temperatures logged for the project were cross checked against a properly calibrated NIST certified thermometer and found to be compliant. No effect on the quality or usability of the data is expected, pending data validation.

Organic Extractions/Sample Preparation (Michael Yurovsky and Doug Honzay)

The following items and systems were reviewed for Separatory Funnel Liquid-Liquid Extraction (3510), Sonication (3550) and Silica Gel Cleanup (3630): SOPs; standard traceability; standard logbook; calibration of analytical balance; extraction bench worksheets; laboratory refrigerators with thermometers and temperature log.

The analytical balance (Serial No. C0146491) used in the organic extraction trailer was last calibrated on 6/27/99.

**Onsite Response:** All balances have been calibrated during the course of this project. The recalibrations were in good agreement with the previous results *(see Appendix N)*.

**Earth Tech Response:** Response acceptable. No effect on the quality or usability of the data is expected, pending data validation.

Findings:

1. The laboratory SOPs for the Separatory Funnel Liquid-Liquid Extraction and Silica Gel Cleanup were not available in the organic extraction trailer. The updated EPA Method is not referenced in the SOP. (i.e., 3550 is referenced instead of 3550B)

**Onsite Response:** The SOPs are now available for review *(see Appendix B)*. Silicia gel cleanup was utilized for sample clean-up and extraction according to SW3550B was used for samples from this project.

**Earth Tech Response:** Response acceptable. The SOPs were updated and the correct methods were used on the samples for this project. No effect on the quality or usability of the data is expected, pending data validation.

2. Temperature was monitored and recorded (all @ 10°C) for the sample storage refrigerator #2 from 12/4/99 to 12/6/99. This refrigerator was labeled as "not in use" on 12/6/99. The temperature monitoring was not documented for the standard freezer #1.

   **Onsite Response:** The freezer is now temperature monitored on a daily basis *(see Appendix D)*. Samples were not stored in refrigerator #2 for this project. A thermometer in freezer #1 was checked daily, but not logged prior to the audit. This freezer was used to store TEPH fuel standards, for which no temperature requirements are specified.

   **Earth Tech Response:** Response acceptable. The refrigerator in question was not used for sample storage for this project. The freezer remained at acceptable temperatures for its use at all times. No effect on the quality or usability of the data is expected, pending data validation.

3. Two sets of class "S" weights are used. However, documentation of calibration was only available for one set of the weights. This weight set was last calibrated on 8/1/96. Weight sets are to be calibrated at least every three years.

   **Onsite Response:** The weights have been replaced with calibrated weights. The balances utilized for this project were recertified during this project. The recertification showed that the balances were functioning within accuracy guidelines before recertification was performed indicating that the weights used were also acceptable *(see Appendix N)*.

   **Earth Tech Response:** Response acceptable. All measurements referencing the weights can be documented with traceability to calibrated weights. No effect on the quality or usability of the data is expected, pending data validation.

4. The addition of a surrogate standard and the surrogate standard ID were not identified and recorded in the extraction logbook.

   **Onsite Response:** The addition and ID of the surrogate is now provided in the extraction logbook for all extractions. Extraction records have been amended to include the ID, concentration, and quantities of surrogate added to the samples prior to extraction *(see Appendix D)*.

   **Earth Tech Response:** Response acceptable. All surrogate additions are documented in the logbook. No effect on the quality or usability of the data is expected, pending data validation.

5. The extraction method name and the method number were not listed in the extraction logbook.

**Onsite Response:** The extraction name and method number has been added to the extraction logbook for all extractions related to this project *(see Appendix D)*.

**Earth Tech Response:** Response acceptable. No effect on the quality or usability of the data is expected, pending data validation.

6. The tuning record for the sonicators and the thermometer calibration records were not available in the extraction lab trailer.

   **Onsite Response:** The records are now available for review *(see Appendix K and H)*.

   **Earth Tech Response:** Response acceptable. Note that all surrogate recoveries for samples extracted using sonication have been within acceptable limits, indicating acceptable performance. Traceable linkage to recertified NIST thermometers is complete and documented. No effect on the quality or usability of the data is expected, pending data validation.

TEPH by GC/FID (Michael Yurovsky)

The following items and systems were reviewed for this method: SOP; calibration requirements; frequency and criteria of calibration checks; corrective action procedures; maintenance and analysis logbooks; standard traceability and labeling; the condition of laboratory refrigerators including thermometers and temperature logbooks.

Findings:

1. There was no current, approved SOP available in the laboratory. The SOP in use is under revision and contains a lot of manual corrections and is not a final, approved copy.

   **Onsite Response:** The required SOP has been updated and a copy available for review in the appropriate laboratory area *(see Appendix B)*.

   **Earth Tech Response:** Response acceptable. The SOP is available for review. The Onsite Laboratory analyst for this method is highly trained, experienced, and familiar with the requirements for the method. There is no indication that analyses were performed in any way not in compliance with QAPP and method requirements. All data will be thoroughly reviewed for compliance with QAPP and method requirements in the data validation process. If any non-compliance is identified, the results will be reviewed for potential impact on data usability and qualified accordingly. No effect on the quality or usability of the data is expected, pending data validation.

2. The instrument maintenance logbook could not be located during the audit.

   **Onsite Response:** The instrument maintenance logbook is now available *(see Appendix K)*.

   **Earth Tech Response:** Response acceptable. There is no indication that instruments were not set-up or maintained adequately. All systems have been properly maintained

according to manufacturers recommendations. All analysts are highly experienced and knowledgeable in the maintenance and servicing of the instrumentation. The maintenance logbook is available for review. No effect on the quality or usability of the data is expected, pending data validation.

3. Several motor oil calibration standards are only labeled as M.O. with the individual concentration. Therefore, it is not possible to trace these standards back to the standard logbook. All standards should be labeled with the standard ID, concentration, as well as the date of preparation, expiration and the preparer ID.

   **Onsite Response:** Comment noted. Onsite has reviewed the available information to reconstruct the documentation necessary to provide traceability to the appropriate source material in the standards preparation logbook *(see Appendix M)*.

   **Earth Tech Response:** Response acceptable. The standards were prepared for the project and the preparer was able to link the standards to the appropriate documentation. The standards are now adequately labeled. No effect on the quality or usability of the data is expected, pending data validation.

4. The standard preparation logbook does not contain instructions for preparation of standards. However, instructions for standard preparation are included in the SOP.

   **Onsite Response:** Standards preparation is provided in each of the method SOPs. The standards preparation logs document the concentration of the parent solution, amount used, dilution volume, and final concentration of the working standards *(see Appendix B and M)*.

   **Earth Tech Response:** Response acceptable. Standard preparation logs include all necessary standard identification (chemical name, vendor, lot number, and/or laboratory standard identification) and stoichiometric information (mass, volume, dilutions). All standards used for this project are traceable to the original standard and certificate of analysis. No effect on the quality or usability of the data is expected, pending data validation.

VOC by GCMS (Michael Yurovsky)

The following items and systems were reviewed in the GC/MS volatile section for method 8260B: SOP; maintenance log; analysis log; standard refrigerator; levels of calibration standards, calibration frequency, calibration acceptance criteria, traceability, and standard labeling.

The VOC analysis by GCMS Method 8260B is performed in the same trailer with sample receiving.

Findings:

1. There was no current, approved SOP available in the laboratory. The SOP for method 8260B is not finalized, and the SOP for method 5035 still needs further review.

**Onsite Response:** The SOP has been finalized and is available in the laboratory *(see Appendix B)*.

**Earth Tech Response:** Response acceptable. The SOP is available for review. The Onsite Laboratory analyst for this method is highly trained, experienced, and familiar with the requirements for Method SW8260B. All samples were analyzed within 48 hours according to the requirements of Method SW5035 and the project QAPP. There is no indication that analyses were performed in any way not in compliance with QAPP and method requirements. All data will be thoroughly reviewed for compliance with QAPP and method requirements in the data validation process. If any non-compliance is identified, the results will be reviewed for potential impact on data usability and qualified accordingly. No effect on the quality or usability of the data is expected, pending data validation.

2. A VOC standards logbook was not available. The preparation, expiration date and preparer ID are not listed on the standard labels. All standards should be labeled with the standard ID, concentration, as well as the date of preparation, expiration and the preparer ID.

   **Onsite Response:** The VOCs standards logbook is available in the VOC laboratory. The labels on all standards will be updated to include the requested information *(see Appendix M)*.

   **Earth Tech Response:** Response acceptable. The VOCs standards logbook is available. Additional information has been added to the labels. All standards logbooks and standards used for this project have adequate documentation and labeling such that traceability to the original standard with a certificate of analysis is possible. All such documentation has been made available at the site, will be included in the final data product, and will be reviewed in the data validation process. No effect on the quality or usability of the data is expected, pending data validation.

3. Temperature monitoring of the freezer 3B has not been documented. The last temperature recording was made on 6/26/98.

   **Onsite Response:** Freezer 3B was not utilized for this project.

   **Earth Tech Response:** Response acceptable. This unit was broken and never used at this site. No effect on the quality or usability of the data is expected, pending data validation.

4. The addition of surrogates and the surrogate IDs are not identified in the GCMS instrument print out which serves as the analysis runlog. Method 8260 is specified on the instrument printout, but the updated method "8260B" is not.

   **Onsite Response:** The surrogate IDs and Method 8260B are now specified on the instrument printout *(see Appendix O)*.

   **Earth Tech Response:** Response acceptable. The surrogate IDs have been implemented in the printout on the run log. Little or no data for this method was

generated before the audit. Any data before correction can be verified in the data validation phase by verifying reported results and raw data. EPA Method SW8260B has been used throughout, and the instrument printout has been corrected. No effect on the quality or usability of the data is expected, pending data validation.

PAHs by HPLC (Chris Cao and John Mough)

The following items and systems were reviewed in the HPLC section for method 8310: SOP; analysis log; standard refrigerator; levels of calibration standards, calibration frequency, calibration acceptance criteria, traceability, and standard labeling.

Positive hits detected by the primary UV detector in samples are confirmed by the secondary fluorescence detector for naphthalene, acenaphthylene, acenaphthene, fluorene, and indeno (1,2,3-c,d) pyrene. For the other PAHs, confirmation of the compounds is made by comparison of peak measurement at both wavelength 254 nm and 223 nm on the fluorescence detector.

Findings:

1. The SOP used in the laboratory was not an approved (signed) version.

   **Onsite Response:** The SOP has been finalized and an approved copy posted in the HPLC laboratory *(see Appendix B)*.

   **Earth Tech Response:** Response acceptable. The SOP is available for review. The Onsite Laboratory analyst for this method is highly trained, experienced, and familiar with the requirements for Method SW8310. There is no indication that analyses were performed in any way not in compliance with QAPP and method requirements. All data will be thoroughly reviewed for compliance with QAPP and method requirements in the data validation process. If any non-compliance is identified, the results will be reviewed for potential impact on data usability and qualified accordingly. No effect on the quality or usability of the data is expected, pending data validation.

2. The instrument maintenance logbooks were not available for the three HPLC instruments, one of which is a rental.

   **Onsite Response:** Instrument maintenance logbooks are now available for all instruments including the rental unit *(see Appendix K)*.

   **Earth Tech Response:** Response acceptable. There is no indication that instruments were not set-up or maintained adequately. All systems have been properly maintained according to manufacturers recommendations. All analysts are highly experienced and knowledgeable in the maintenance and servicing of the instrumentation. The maintenance logbooks are available for review. No effect on the quality or usability of the data is expected, pending data validation.

3. Standards are prepared at JPB Laboratories, Sacramento, which is affiliated with Onsite Environmental Laboratories. The standard logbook is kept at JPB lab and is not available in the trailer.

   **Onsite Response:** Onsite has centralized all the records for this project in order to provide complete traceability of standards, and the records are available on-site *(see Appendix M)*.

   **Earth Tech Response:** Response acceptable. All standards used for this project have adequate documentation and labeling such that traceability to the original standard with a certificate of analysis is possible. All such documentation has been made available at the site, will be included in the final data product, and will be reviewed in the data validation process. No effect on the quality or usability of the data is expected, pending data validation.

4. Retention time window checks have not been performed for method 8310.

   **Onsite Response:** All RT window checks have now been performed *(see data packages)*.

   **Earth Tech Response:** Response acceptable. Retention time (RT) window studies were completed for each method and each matrix before samples of that matrix were analyzed, although for some samples preliminary results may have been reported before RT studies were fully evaluated. Analytical results provided before evaluation of the studies were reported as preliminary. The studies have been evaluated, and results are reported accordingly. No effect on the quality or usability of the data is expected, pending data validation.

5. An MDL study was performed, but the results have not been calculated yet.

   **Onsite Response:** The MDL study has been completed and all results calculated prior to sample analysis *(see Appendix C)*.

   **Earth Tech Response:** Response acceptable. MDL studies were completed for each method and each matrix before samples of that matrix were analyzed. No effect on the quality or usability of the data is expected, pending data validation.

Nitroaromatics and Nitroamines by HPLC (Chris Cao)

The following items and systems were reviewed in the HPLC section for method 8330: SOP; analysis log; standard refrigerator; levels of calibration standards, calibration frequency, calibration acceptance criteria, traceability, and standard labeling.

The Diode Array Detector allowing collection of multiple wavelength responses for target compounds is utilized. Compounds detected at 254 nm are also monitored at other wavelengths (240, 250 and/or 210 nm) for comparison. A compound is confirmed when the measurement at all three wavelengths agree within ± 20 %D.

Findings:

1. The SOP for method 8330 using Rapid Extraction Method for soil samples does not include the sample extraction procedure for water samples.

   **Onsite Response:** The SOP has been updated to include the extraction procedure for waters *(see Appendix B)*.

   **Earth Tech Response:** Response acceptable. No effect on the quality or usability of the data is expected, pending data validation.

2. The instrument maintenance logbooks were not available for the three HPLC instrument, one of which is a rental.

   **Onsite Response:** Maintenance logbooks are available for all instruments including the rental unit *(see Appendix K)*.

   **Earth Tech Response:** Response acceptable. No effect on the quality or usability of the data is expected, pending data validation.

3. Standards are prepared at JPB Laboratories, Sacramento, which is affiliated with Onsite Environmental Laboratories. The standard logbook is kept at JPB lab and is not available in the trailer. The expiration date and the preparer ID were not noted on standard containers for the surrogate and the explosive standard that was prepared on 12/2/99. All standards should be labeled with the standard ID, concentration, as well as the date of expiration and the preparer ID.

   **Onsite Response:** Onsite has centralized all the records for this project in order to provide complete traceability of standards, and the records are available on-site. Standard labels have been updated to include the following information: Standard name, log ID, concentration, units, analyst, and date prepared *(see Appendix M)*.

   **Earth Tech Response:** Response acceptable. All standards used for this project have adequate documentation and labeling such that traceability to the original standard with a certificate of analysis is possible. Although expiration date is not on the label, it is known from the preparation date. No expired standards were used during this project. All such documentation has been made available at the site, will be included in the final data product, and will be reviewed in the data validation process. No effect on the quality or usability of the data is expected, pending data validation.

4. Certificate of analysis was found in a stack on the lab bench and was not filed in an organized manner.

   **Onsite Response:** The certificates have been organized in a three ring binder for all standards in all departments *(see Appendix M)*.

   **Earth Tech Response:** Response acceptable. No effect on the quality or usability of the data is expected, pending data validation.

5. Retention time window checks have not been performed for method 8330.

**Onsite Response:** The RT window checks have been completed *(see data packages)*.

**Earth Tech Response:** Response acceptable. Retention time (RT) window studies were completed for each method and each matrix before samples of that matrix were analyzed, although for some samples preliminary results may have been reported before RT studies were fully evaluated. Analytical results provided before evaluation of the studies were reported as preliminary. The studies have been evaluated, and results are reported accordingly. No effect on the quality or usability of the data is expected, pending data validation.

6. An MDL study was performed, but the results have not been calculated yet.

**Onsite Response:** The MDL study results have been calculated and forwarded to Chris Davis of Earth Tech *(see Appendix C)*.

**Earth Tech Response:** Although MDL and retention time (RT) window studies were completed for each method and each matrix before samples of that matrix were analyzed, the MDL and RT were not fully evaluated immediately for each method. Analytical results provided before evaluation of the studies were reported as preliminary. The studies have been evaluated, and results are reported accordingly.

The SW8330 MDL studies were performed according to SW-846 method requirements. MDLs were compliant with the requirements of the QAPP, with the following exception:
All MDLs for SW8330 target analytes were at or below the PQL, but not less than ½ PQL. The MDLs for the amino-DNTs, which co-elute, have been summed and are therefore at 2X the PQL of the individual amino-DNTs.

The usability of the SW8330 data is not expected to be affected as the MDLs are valid and the resulting data are usable. Daily standards at the PQL have been analyzed for all SW8330 analyses for this project, and sensitivity and accuracy (±25%) have been documented. All PE sample results for explosives have met project accuracy goals.

In addition, the laboratory performed a second MDL study for the same instrumentation using lower concentrations of each analyte at the end of the project to document sensitivity at the PQL. The PQL specified in the QAPP for the SW8330 target analytes in soils is 0.4 mg/kg. The initial study was performed using standards at 1.6 mg/kg and the final study was performed using standards at 1.0 mg/kg. The results for the final MDL study indicate MDLs of 0.1 mg/kg for most analytes, 0.2 mg/kg for two analytes and 0.3 mg/kg for the summed amino-DNTs. These MDLs meet the QAPP requirement that MDLs be less than or equal to ½ the PQL. Although the second set of MDLs cannot be reported for the analyses performed prior to the follow-up MDL study, it demonstrates the ability of the laboratory to meet the PQL.

Data for all analyses are reported down to the MDL for all analytes for all methods, which will still apply to method SW8330 using the initial MDLs). The PQL may need to be raised to 2X the MDL for each SW8330 analyte, or results less than 2X the MDL

may require the same general qualification as all data reported between the MDL and PQL, which are qualified as estimated (J).

The PQLs for the SW8330 target analytes are 1-4 orders of magnitude below the respective PRGs referenced as the action levels for decision making for this project (the TNT PRG in soil is 40X the PQL), therefore, the data still meet the DQOs for this project.

Thus, the reporting of SW8330 results with PQLs not greater than 2X the MDLs is not expected to significantly affect the legal defensibility or usability of the data. The data are expected to fulfill project DQOs.

A complete evaluation of the MDLs with respect to the DQOs will be made in the QCSR which will accompany the final data validation reports.

Data Review/Data Management

A Level 1 review is performed by the analyst on 100% of the data. A Level 2 review is crossed checked by a peer. However, no checklist is used to document the data review.

Finding:

1. There is no QA personnel assigned to review the data packages for this project.

**Onsite Response:** Chris Amstutz of Onsite will perform the QA review of all data generated for this project utilizing a checklist created for this project *(see Appendix E)*.

**Earth Tech Response:** The audit took place before secondary review could take place. Such review is performed after initial data are generated and in time for corrective actions if appropriate. Complete review of all data must occur before finalization of the data product.

The laboratory has implemented appropriate review procedures as specified in the QAPP. This process insures that all data will meet the requirements of the QAPP, and that the laboratory implements corrective actions and non-conformance reports prior to release of any final data. All data will be further reviewed for compliance in the data validation process. The multiple review process insures that all reported data are usable or appropriately qualified, and legally defensible. No effect on the quality or usability of the data is expected, pending data validation.

Reagent Water

Distilled or drinking water is used for the TEPH analysis, boiled distilled water is used for the VOC analysis, and HPLC grade water is used for the HPLC analysis.

Finding:

1. The reagent water used for the VOC analysis was checked by Method 8260B. The analysis results of the reagent water check is stored in the computer, but a hard copy of the analysis results of the reagent water was not available.

   **Onsite Response:** The results of the reagent water check are available in the laboratory *(see Appendix L)*.

   **Earth Tech Response:** Response acceptable. No effect on the quality or usability of the data is expected, pending data validation.

Standard Operating Procedures

Finding:

1. A current, approved SOP was not available for all methods performed for the project. There was not a SOP log that includes all the SOPs for the entire laboratory area and analytical methods.

   **Onsite Response:** The SOPs utilized for this project have been updated and organized in a three ring binder under the control of the Project QAO. The SOPs all available for all methods performed for this project. A number of the SOPs required revisions due to the unique requirements of this project *(see Appendix B)*.

   **Earth Tech Response:** Response acceptable. All laboratory analysts for this project are highly trained, experienced, and familiar with the requirements of the US EPA Methods and the project QAPP, as well as for US ACOE projects. The staff were further trained in updating SOPs, which are current and available.

   There is no indication in the audit report that analyses were performed in any way not in compliance with QAPP and method requirements. All data will be thoroughly reviewed for compliance with QAPP and method requirements in the data validation process. If any non-compliance is identified, the results will be reviewed for potential impact on data usability and qualified accordingly.

   No effect on the quality or usability of the data is expected, pending data validation.

2. One typo was found in the SOP for extraction Method 3510 (Method 3550 was listed instead).

   **Onsite Response:** The typo has been corrected *(see Appendix B)*.

   **Earth Tech Response:** Response acceptable. There is no indication in the audit report that extractions were performed in any way not in compliance with QAPP and method requirements. No effect on the quality or usability of the data is expected, pending data validation.

# QUANTERRA ENVIRONMENTAL SERVICES
# (SEVERN TRENT LABORATORIES)

December 27, 1999

Quanterra West Sacramento
880 Riverside Parkway
West Sacramento, CA 95605-1501

Attention: Pam Schemmer

| | |
|---|---|
| Subject: | AUDIT REPORT |
| Audit Date: | December 15, 1999 |
| Audit Location: | Quanterra, West Sacramento, CA |
| Audit Scope: | Laboratory audit with particular emphasis on Benicia Tourtelot QAPP requirements. |
| Auditor: | Debbie Masonheimer, Earth Tech, San Jose |
| Personnel Contacted: | Pamela Schemmer, Josie Tellars, Mark Frey, John Barnett, David Alltucker |
| Audit Summary: | A walkthrough audit of the laboratory was performed. The audit concentrated on areas where the West Sacramento facility is performing analyses for Benicia Tourtelot project (general chemistry and metals), as well as sample receiving, and included interviews with laboratory personnel. In addition, the QA program was discussed with QA personnel including reporting non-conformances and anomalies. Following the audit, a debriefing with the appropriate area leaders was held. |

Sample Receiving:

Personnel: Marcela Garcia

The following items and systems were reviewed: sample receipt, log-in, storage, tracking and anomalies; sample labeling; and refrigerator temperature monitoring.

Sample storage refrigerator temperatures are monitored and recorded daily except Sundays and holidays.

Sample receiving and log-in are generally handled well. The log-in personnel use checklists to assure that the following items are checked upon sample receipt: presence of chain-of-custody seals; intact sample containers; sample temperature; pH of preserved samples; and initial chain of custody (COC) review. The temperature blank or representative sample is checked against a calibrated IR thermometer. Sample labels are checked against COC records for consistency. Following login, unique laboratory sample numbers are affixed by another person to provide a secondary review of both labels and COCs.

Procedures are in place for monitoring holding times, and notifying the appropriate laboratory section of short hold time samples or rush samples. Samples are stored in segregated refrigerators depending on the analysis group. Each refrigerator has a constant temperature recorder which operates 24 hours a day, seven days a week; manual readings are also recorded daily, excluding Sunday and holidays.

Standard operating procedures (SOPs) are available for reference. All SOPs in this area were appropriately signed and dated. If samples are received which do not meet the specific project criteria (i.e., improper or no preservatives, broken containers, sample temperatures beyond criteria), the PM is informed and then the client is contacted for handling instructions. Any pH adjustment is made after notifying the client and will require a non-conformance report to be generated.

Project samples were received during the audit of this area and all appropriate sample receiving protocols were followed.

There were no findings or concerns in this area.

General Chemistry:

Personnel: Josie Tellars, Mark Frey

The project is concerned with the following wet chemistry analyses for soil and water: anions by ion chromatography, EPA method 300.0; total organic carbon (TOC), EPA methods 415.1 and 9060; pH, EPA method 9045C; and total dissolved solids (TDS), EPA

method 160.1. Personnel were able to describe QAPP-appropriate analytical batches for each method.

Standard operating procedures were reviewed for each method, and were current, controlled, and appropriately signed, which indicates corrective actions have been taken since the previous AFCEE audit in July 1999. Balance calibrations are checked daily prior to use. Standards were labeled correctly and were traceable to original stock solutions. Certificates of analyses were available for purchased standards, although most standards in this are made from dry chemicals. A second source standard is available for ion chromatography analysis.

Maintenance logbooks were up-to-date including notations for return to control, and were signed by both the analyst and the supervisor on a regular basis.

Individual sample runlog books were complete and up-to-date, with few exceptions (i.e., the ion chromatograph's run had not been fully transferred from the previous day's hardcopy printout). Initial and continuing calibrations are preformed as required by the individual methods.

There were no findings or concerns in this area.

Metals (preparation and analysis):

Personnel: John Barnett, David Alltucker,

The following methods were covered during the audit: ICP and ICP-Trace, EPA method 6010B; mercury, EPA methods 7471 or 7470; and appropriate digestion procedures. The metal ICP and ICP-Trace digestions are now being performed utilizing block digestors rather than hot plates. This change was noted in the appropriate SOPs.

Personnel were able to describe QAPP-appropriate preparation and analytical batches. Due to the low level spiking amounts required in the QAPP, the project's samples are digested separate from samples from other projects. Most of the soil samples for the project required equal-weight compositing prior to analysis. Balance calibrations are verified daily prior to use.

Standards were correctly labeled and traceable to stock solutions; certificates of analyses were available. Calibrations are performed according to the individual methods. Initial calibration verification standards are from a second source supplier. ICP interference checks are run daily following calibration prior to sample analysis for those elements listed in EPA method 6010B; the acceptance criteria for other elements is less than their respective reporting limit.

Pipettes were appropriately labeled with calibration dates and calibrations were documented.

Finding:

1. According to the QAPP, mercury analyses were to be analyzed by duplicate injections. The personnel were to see if their instrumentation and software would allow this to be performed on a routine basis.

**Earth Tech Response: Earth Tech Response:** For mercury by methods SW7470A/7471A, Table 3.2-5 of the QAPP specifies that instrument precision be documented by %RSD<10% for 2 integrations of calibration standards; and if the duplicate integrations are not provided, alternate documentation of instrument precision must be provided. Quanterra has historically requested and received from ACOE Sacramento District a variance to the 2 integration requirement. If the variance is accepted by the ACOE Sacramento District, the issue is resolved. If the variance to the QAPP is not granted and the duplicate integrations cannot be provided by Quanterra, additional documentation of instrument precision will be requested, such as LCS and/or CCV control charts. This issue does not represent a serious data quality issue, and no qualification of data will be required unless other QC data indicate precision problems.

100 West Broadway, Suite 3000, Long Beach, California [illegible]

July 27, 1999

Quanterra Environmental Services
880 Riverside Parkway
West Sacramento, CA 95605-1501

Attn. Eric Redman/Pamela Schemmer

Subject: AUDIT REPORT

Audit Date: July 12 and 13, 1999

Audit Location: Quanterra, West Sacramento, CA

Audit Scope: Laboratory audit with particular emphasis on AFCEE requirements, and compliance with SW846 and other EPA methods protocol

Auditors: Elisabeth Fruth (Earth Tech, Long Beach), Debbie Masonheimer (Earth Tech, San Jose), and Jim Cirillo (Earth Tech, Long Beach)

Observers: Patricia Rowans (AFCEE), R. William Kessler (AFCEE)

Personnel Contacted: Pamela Schemmer, Gail Celaschi, Ray Lucero, Karla Buechler, Dennis Gall, Michael Filigenzi, Donald Taylor, Emily Uebelhoer, Marcia Reed, Karen Mason, Pam Niya, Josie Jones, David Alltucker, Alan Wong, John Barnett, Wayne Halozan, Kyle Stephens, Mike Flournoy, Pat Rainey, Andre Algazi, Eric Redman

Audit Summary: A walkthrough audit of all areas of the laboratory was performed. The audit concentrated on areas where the West Sacramento facility has performed or will perform analyses for Edwards AFB and Norton AFB projects, as well as sample receiving, and included interviews with laboratory personnel. In addition, the performance evaluation studies for the past two years were reviewed, and the QA program was discussed with QA personnel. A debriefing was held where the audit findings, concerns and recommendations from the laboratory audit were summarized. In addition to some personnel listed above, the debriefing was attended by Roger Frieze.

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The following sections constitute the findings, concerns and recommendations grouped by area or subject.

**Sample Receiving and Control** *(Ray Lucero)*

The following items and systems were reviewed: sample receipt, log-in, storage, tracking and anomalies; sample labeling; and refrigerator temperature monitoring.

Sample receiving and log-in are generally handled well. Temperature and pH are checked upon receipt. If the temperature or pH is not per requirements the PM is informed. Any pH adjustment is made after notifying the client. Procedures are in place for monitoring holding times, and notifying the appropriate laboratory section of short hold time samples or rush samples. Sample storage refrigerator temperatures are monitored and recorded daily except Sundays and holidays.

Findings:

1. The acceptable freezer temperature range is –20 degrees Celsius to –10 degrees Celsius. However, a temperature of –8 degrees Celsius was recorded for freezer F2 on 7/10/99. There was no entry on the temperature log indicating any corrective action in response to the anomalous temperature even though there is space available for such an entry at the bottom of the log. Furthermore, sample receiving personnel was not aware of the correct protocol, i.e., was not familiar with the instructions in the SOP.

Concerns:

1. Several sample container mix-ups have occurred at the West Sacramento facility. For instance, for the Site 4 Landfill in January (samples 4-MW02-WG26 and 4-MW01-WG26 were switched) and in April (4-MW06-WG224 and 4-MW04-WG27, inorganics, were switched). For Norton, lot 303515, the trip blank and sample MLW-2-3 were switched. Furthermore, for sample G9D150172-006 it is suspected that a vial for method 504.1 was switched. Per sample receiving personnel, it has happened that analysts will return bottles to sample receiving if the field ID and the lab ID on the bottle do not match written information. Although the system of setting up the bottles grouped by field ID should work, no double checking is performed. Earth Tech is truly concerned with the repeated incidence of known sample mix-ups, and even more concerned with possible undetected mix-ups.

2. There have been several incidents where sample containers shipped from West

Sacramento to Edwards AFB have arrived broken. Trip blanks shipped in coolers have also arrived broken. Apparently, the trip blanks are not cushioned, i.e., wrapped individually or put in a foam block, but are lying loose in a plastic bag. Since the plastic bag is not secured, the bottles can crash together and break. This situation is both hazardous and may result in too few vials available for analyses.

3. Temperature logs do not show whether a correction factor needs to be applied to the temperature readings, nor whether the temperature reading is corrected or not. According to Pam Schemmer, a goal of the QA department is to remove from service any thermometer that requires a correction factor, thereby eliminating the need to record them. While this policy is reasonable, until it is implemented, temperature logs should contain spaces to list correction factors, and indicate whether the recorded temperature is corrected or not.

**General Chemistry** *(Josie Jones)*

The following information was reviewed for the wet chemistry methods 9060 and 415.1 (TOC); 351.2 (TKN); 365.3 (Total P); 9012 (CN); 160.1 (TDS); 300.0, 340.2, and 4500-F/C (anions); 310.1 and 2320 (alkalinity); 350.1 (ammonia-N), Walkley-Black; 1664 and 413.1 (oil&grease) and 376.2 (sulfide): calibration requirements, frequency and criteria of calibration checks, corrective action procedures, standards traceability, and a review of maintenance logbooks and SOP's.

Findings:

1. The SOP for the 9060 method for soil is not consistent with current laboratory protocol. Quadruplicate analyses are performed in accordance with AFCEE requirements, but are not addressed in the SOP. This is a repeat finding from the previous year's audit.

2. The SOP for method 365.3 (LM-CAL-1020) was not available. No approved SOP for the method was available. According to the analyst, the SOP was in progress. This is a repeat finding from the previous year's audit.

3. No SOP for method 351.2 was available. According to the analyst an SOP was in progress, but no copy was provided. This is a repeat finding from the previous year's audit.

4. The SOP for Walkley-Black was dated 8/4/92 and on Enseco letterhead.

5. Supervisor review and initials had not been performed since mid-May on any

log book or maintenance log reviewed.

6. The SOP for method 340.2 was not a controlled copy.

Recommendations:

1. Some of the SOPs in the General Chemistry section are older than July 1994, and are therefore printed on Enseco letterhead. It is highly recommended that all SOPs be reviewed and updated. If updating is not necessary, at least reprint them on Quanterra letterhead with a review date indicated.

**Metals Preparation and STLC/TCLP extractions** *(David Alltucker)*

The following information was reviewed for metals preparation by methods 3005, 3015 and 3050, and leachate preparation: SOP's, standards traceability, maintenance logbooks, and prep logs.

Findings:

1. Pipettes in the metals and metals preparation areas list the date calibrated, but do not list calibration due dates.

2. Expiration dates are not listed on spiking solution or extraction fluid containers.

3. STLC and TCLP extraction procedures were in revision according to the analyst and not available for review.

**Metals** (*David Alltucker, John Barnett, Allan Wong*)

The following information was reviewed for mercury (7470A, 7471A, 245.1) and Inductively Coupled Plasma (ICP) metals (6010B, 200.7): calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance logbooks, SOP's, retention time and MDL studies.

Organic lead is not performed by the laboratory. Graphite furnace atomic absorption and ICP-MS methodologies were not audited.

Findings:

1. Review signatures were missing on SOP CORP-MT-0005 for mercury analysis of water sample. The SOP is also past its review date of 12-18-97.

2. The Mercury instrument maintenance schedule was manufacturer software-driven, and the information of what was performed was manually transferred to a maintenance log . Return to control was noted, but the log showed no indication of supervisor review.

3. Daily working standards for mercury analysis were not tracked.

4. A certificate of analysis (Lot number IC-0704, Ultra ICM-441, solution 979-ICP-067) could not be located.

5. Multi-element solutions do not list individual analyte concentrations on the label.

6. An out-of-date calcium standard stock solution was found. The standard was taken out of service during the audit.

**Extraction/Sample Preparation-organics** *(Emily Uebelhoer, Karen Mason)*

Documentation for extraction methods 3510C, 3520C, 3550C, 3540C as well as extraction for method 1664 were reviewed.

Findings:

1. Certificate of analysis for EDB/DBCP (lot number LA76854) was not properly filed with other certificates. It was located during the audit in the box of stock standards in the refrigerator.

2. The standards QC Package for an EDB standards was not in the Standard Prep file cabinet. However, a GC#69 sequence file from 2/8/99 was reviewed and showed the analysis of the standard in question.

3. The standard logbooks have an entry available to indicate whether or not a standard, after analysis, was acceptable for use. However, many pages in standard logbook #'s 887 and 954 did not indicate acceptability or lack thereof. Moreover, several standards observed in the GC semivolatiles lab were not labeled as being acceptable for use. According to Emily Uebelhoer

and Gail Celaschi, in order to improve the system of indicating standard acceptability, the current procedure would be reviewed and updated.

4. Monthly review of the run and maintenance logbooks is not up to date.

**GC/MS, semivolatiles (8270C)** *(Donald Taylor)*

8270C is currently performed and the following information was reviewed: calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance logbooks, SOP's, retention time and MDL studies.

Findings:

1. The Controlled Documents Index lists method 8270A and the pertinent SOP references method 8270C. The index should list 8270C.

2. Run logbooks indicated method 8270, but did not list the version performed.

3. The 'QC' on the standard labels are blank. According to the analyst, this area is to be filled in following acceptance of the standards following comparison to a second source.

4. According to method 8270C, the internal standards (ISs) in the CCV are to be evaluated against the ISs in the ICAL mid-point standard. However, according to Donald Taylor, this procedure has not been followed consistently, in part due to current limitations between the mass spec upload software and the LIMS. Until IS evaluation is automated, this procedure must be done manually for every CCV.

**Gas Chromatography, Extractables** *(Emily Uebelhoer, Marcia Reed, Pam Niya)*

The following information was reviewed for organochlorine pesticides (8081A), PCBs (8082), 8151A, 504.1 and LUFT TEPH (8015): calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance logbooks, SOP's, retention time and MDL studies.

Findings:

1. The implementation date on SOP SAC-GC-0007 (LUFT- Extractables) was

blank, although it was revised in September 1998.

2. The 'QC' on the standard labels are blank. According to the analyst, this area is to be filled in following acceptance of the standards following comparison to a second source.

Recommendations:

1. Chromatograms for EDB/DBCP (method 504.1) standards indicate interferences from one or more compounds. Some of these non-target peaks were also present in the reviewed method blank. It is recommended that an EDB/DBCP standard be made up in methanol and analyzed by GC/MS to possibly determine these interferences.

2. On samples analyzed by both 504.1 and 8260, 504 results should be compared to those of the 8260 analysis.

**GC/MS, volatiles (8260B)** *(Wayne Halozan)*

8260B is currently performed and the following information was reviewed: calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance logbooks, SOP's, retention time and MDL studies. Method 5035 is available.

Findings:

1. Updated method, 8260B, was not identified in the runlog. Only 8260 was noted. This is a repeat finding.

2. Refrigerator blanks are routinely placed in the sample refrigerator by the QA department but are not always analyzed per the analyst. The blanks have a much lower priority than clients' samples. Following analysis, the data is sent back to QA.

**GC, Volatiles** (*Kyle Stephens*)

The following information was reviewed for volatile compounds (8021B) and LUFT purgeables (8015): calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance logbooks, SOP's, retention time and MDL studies. EDB/DBCP (504) is performed

by the semivolatile GC group.

There were no findings in this section.

Recommendations:

1. A peer review stamp was used in the maintenance logbooks. It is recommended that subsequent stamps be applied on the opposing blank page, from top to bottom, for greater legibility.

2. According to Kyle Stephens, headspace that would prevent the use of a VOA vial was determined to be any bubble in excess of 4 mm in size. However, Mr. Stephens was not sure if the pertinent SOP(s) addressed this issue. If not already done, discussion of unacceptable bubble size in VOA vials should be part of all pertinent SOPs.

**GC/MS, Dioxins and Dioxins Preparation - low resolution (8280 and 8280A) and high resolution (8290)** *(Pat Rainey, Mike Flournoy, Andre Algazi)*

Both low and high resolution dioxins preparation area were visited. The following items and systems were reviewed: SOPs, preparation procedures, standard traceability and labeling, and standard logbooks. Laboratory SOPs, maintenance logbooks, and analysis logbooks were reviewed.

Findings:

1. The maintenance logbooks showed no indication of supervisor review.

2. Run logs were chronologically placed in a three-ring binder instead of in a bound notebook. According to the analyst, these were periodically transferred to a bound notebook similar to those in other departments; however, the transfer had not taken place for over six months. The run log showed no indication of supervisor review.

**HPLC Extraction/Sample Analysis – 8330 and 8330M (nitroguanidine); 8321 CWM** *(Dennis Gall, Mike Filigenzi, Karla Buechler)*

The following items and systems were reviewed: calibration of analytical balances; extraction bench worksheets; and spiking procedures; calibration requirements; frequency and criteria of calibration checks; corrective action procedures; standards

traceability; maintenance and analysis logbooks; and the condition of laboratory refrigerators including thermometers and temperature logbooks.

Findings:

1. The SOP for method 8321-CWM (Chemical Warfare Materials) was not available. According to the analyst the SOP is in draft form.

2. The standard logbooks have an entry available to indicate whether or not a standard, after analysis, was acceptable for use. However, the "QC'd by" in the standard logbook was not filled out. Acceptability of use had been received by e-mail, but was not documented in the logbook.

3. The temperature of the refrigerator had not been read the day of the audit. The area was visited in the afternoon.

4. The header on the current page in the balance log was incomplete – no model or balance ID was entered.

5. MDL studies were overdue.

6. A low level standard is run at the end of the sequence in order to confirm that the instrument would still detect contamination down to the reporting limit. No acceptance criteria were stipulated for this standard in the SOP.

**GC/MS, semivolatiles (CWM)** *(Donald Taylor)*

The following items and systems were reviewed: sample storage; maintenance logbooks and analysis logbooks; standard preparation, traceability, and labeling; and calibration criteria and frequency.

Finding:

1. The SOP was a draft, dated 3/31/99. The method is referenced as 8270 – however, since there are so many modifications it would be advisable to just call it Semivolatile GCMS – CWM.

**Quality Assurance Management**

*Method Detection Limit Studies*. MDL studies are performed annually for each

method. Many MDL studies for the reviewed methods were overdue. A discussion regarding modifications to the annual verification requirement was initiated between AFCEE, Earth Tech and the laboratory. This issue will be resolved at a later date.

*Standard Operating Procedures*. Standard Operating Procedures (SOP) are available for most of the methods reviewed. Quanterra Corporate SOPs and facility specific SOPs are available.

Findings:

1. SOPs were either not available or were in need of updating for some general chemistry methods – see discussion in this section. Draft SOPs were used in some areas to perform the analyses.

2. The required review of SOPs are overdue. These reviews are not documented on the SOP, and it is therefore difficult to determine when and if they are actually done.

*Document Control*. SOPs are issued as controlled copies. Revisions are issued to the original holders of the documents. Logs are kept electronically.

*Performance Evaluation Samples*. PE sample results from the WP and WS studies for the past two years were reviewed. As of January 1999, EPA no longer provides these samples. ERA (Environmental Resource Associates) is the new provider. The results are very good.

*Audits*. Corporate audits are performed annually. Audits by facility QA are performed periodically, as are data audits. Implementation of corrective action is verified during audits, and a database file has been set up to track previous findings so that an evaluation can be made regarding frequency of findings in each area, and repetition of similar findings.

Concerns:

1. Quanterra has been subcontracting perchlorate analyses for Earth Tech to CLS laboratories. However, no audit of the perchlorate method has been performed at CLS. An audit for method 8260 only had been performed some time ago.

*Nonconformance/Corrective Action*. Electronic anomaly reports are generated, and are automatically included as part of the project file. Resolution of non-routine anomalies must be accepted by QA before closure. Anomalies are tracked, and a database file has been set up to track previous anomalies so that an evaluation can be

made regarding frequency of anomalies in each area, and repetition of similar anomalies.

*Thermometer, Balance and Weight Set Calibrations*. Thermometers used in the lab are calibrated annually against an NIST traceable thermometer. This calibration is performed in-house. Calibration of balances is performed by an outside vendor annually in the laboratory. Weight sets are sent to an outside vendor for calibration every three years.

Findings:

1. The records for thermometer calibration were in disarray. However, it appears that the NIST traceable calibration thermometer 1E3319 was last calibrated on 8/2/1995 and was used for the refrigerator calibrations on 8/26/98. It also appears that the metals preparation thermometers were last calibrated in-house in 1997. The IR thermometer 015609 (presumably used in sample receiving) was last calibrated on 2/11/98. Thermometers are not tagged with calibration date or calibration due date. Therefore it is of utmost importance that the records in the QA department, which are the only source of information regarding calibrations, be kept organized and up to date.

2. Calibration records for two thermometers, lab #s 4552 and 4040, assigned to Air Tox showed a 1 degree difference from the NIST thermometer; however, no correction factor was listed. If these thermometers are indeed used, there must be a correction factor assigned and noted on the temperature log.

3. One balance, QA38, was found to be out of tolerance when calibrated. It was taken out of service the day of calibration. How was the data handled that was generated by this balance prior to the calibration date?

4. There is no current log for tracking the weight sets, i.e., listing locations.

5. The 200 mg weight for set QA-033 was listed as out of tolerance with no adjustment. Is this weight still in the set? Calibration records indicate that a different weight value should be used for this weight. What is being done?

**Personnel Training**

A training program is in place, documenting proficiency for analytical methods, and resumes listing education and previous experience are available for all technical personnel. Files were well organized in specific sections. Improvements are

continually made to facilitate tracking and electronic entries.

Concerns:

1. Most resumes reviewed were out of date

2. There were no records of training on sample receiving protocol except handling of hazardous waste for Ray Lucero, and during the interview with Mr. Lucero he did not appear to be familiar with all protocol required of sample receiving personnel.

**Data Review**

Data review is performed by the analyst (Level 1), by supervisor (Level 2) and by the project manager. Checklists are used.

There have been numerous problems with late reports, electronic data format and EDDs. Some of these problems were caused by the rapid transition from Santa Ana, and also the conversion to a new LIMS system. These problems are being corrected.

**General Comments**

The staff, including analysts and supervisors, are knowledable and proficient in performing the analyses. However, SOPs *must* be written to reflect current practices in the laboratory, and approved SOPs *must* be available in the appropriate laboratory areas.

During the audit process, it was indicated that SOP's were routinely reviewed by the QA department. If found to be acceptable, the SOP review date was listed on a form, and the SOP was not updated; however the SOP's in the laboratory areas do not list this review. A current listing of SOP's with their respective QA review dates needs to be provided. It is also recommended that evidence of this review be added to the SOP within the laboratory areas.

Several of the corrective actions proposed in the response to the Earth Tech audit of 8/98 have not been implemented.

I want to express my appreciation to all laboratory personnel for their interest, courteous assistance and time spent during this audit.

Please respond to the findings and concerns enumerated in the audit report by August 13, 1999.

Very truly yours,

Earth Tech

Elisabeth Fruth

Elisabeth Fruth
QA Manager

cc. Bonnie McNeill

Quanterra
Environmental Services

***Pamela A. Schemmer***
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August 20, 1999

Elisabeth Fruth
Earth Tech
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***Subject: Audit Response***

Dear Elisabeth:

Enclosed is our response to audit findings from July 12 and 13, 1999 for Edwards AFB and Norton AFB. If you have any questions in regards to this response, please feel free to call Pamela Schemmer at (916) 374-4441.

Sincerely,

Pamela Schemmer

Pamela A. Schemmer
Quality Assurance Manager

Eric W. Redman

Eric Redman
Laboratory Manager

enclosure

**Sample Receiving and Control**

Findings:

1. The acceptable freezer temperature range is –20 degrees Celsius to –10 degrees Celsius. However, a temperature of –8 degrees Celsius was recorded for freezer F2 on 7/10/99. There was no entry on the temperature log indicating any corrective action in response to the anomalous temperature even though there is space available for such an entry at the bottom of the log. Furthermore, sample receiving personnel was not aware of the correct protocol, i.e., was not familiar with the instructions in the SOP.

Response: Sample control personnel have been informed of the incident, and the appropriate corrective action. This corrective action is detailed in Quanterra SOP-QA-0005, and includes documentation of incident and adjustments to temperature control, and contacting PMs, QA, and clients if any samples are affected. The sample receiving personnel have been instructed to review the SOP, and the SOP will be discussed in a follow-up QA meeting with the pertinent staff.

Concerns:

1. Several sample container mix-ups have occurred at the West Sacramento facility. For instance, for the Site 4 Landfill in January (samples 4-MW02-WG26 and 4-MW01-WG26 were switched) and in April (4-MW06-WG224 and 4-MW04-WG27, inorganics, were switched). For Norton, lot 303515, the trip blank and sample MLW-2-3 were switched. Furthermore, for sample G9D150172-006 it is suspected that a vial for method 504.1 was switched. Per sample receiving personnel, it has happened that analysts will return bottles to sample receiving if the field ID and the lab ID on the bottle do not match written information. Although the system of setting up the bottles grouped by field ID should work, no double checking is performed. Earth Tech is truly concerned with the repeated incidence of known sample mix-ups, and even more concerned with possible undetected mix-ups.

Response: The normal procedure is to lay out all the samples according to the chain of custody with labels, and have this verified by another analyst prior to labels being applied. We believe the discrepancy with Earth Tech has arisen due to the Earth Tech requirement that samples are numbered according to field Ids, and not the chain of custody. This issue has been addressed with the sample receiving personnel, and will

be addressed in a follow-up QA meeting with the pertinent staff. as well as QA spot audits.

2. There have been several incidents where sample containers shipped from West Sacramento to Edwards AFB have arrived broken. Trip blanks shipped in coolers have also arrived broken. Apparently, the trip blanks are not cushioned. i.e.. wrapped individually or put in a foam block. but are lying loose in a plastic bag. Since the plastic bag is not secured. the bottles can crash together and break. This situation is both hazardous and may result in too few vials available for analyses.

Response: The laboratory was not previously aware of this issue. All vials are shipped either in the manufacturer's boxes, or are padded with bubble-wrap. The laboratory would appreciate more timely feedback to this issue so we may correct any difficulties without incurring any additional shipping costs.

3. Temperature logs do not show whether a correction factor needs to be applied to the temperature readings, nor whether the temperature reading is corrected or not. According to Pam Schemmer. a goal of the QA department is to remove from service any thermometer that requires a correction factor, thereby eliminating the need to record them. While this policy is reasonable, until it is implemented, temperature logs should contain spaces to list correction factors, and indicate whether the recorded temperature is corrected or not.

Response: The thermometers that exceeded the calibration range during the last annual calibration were not used in any refrigerators containing samples. These two thermometers (located in non-sample storage refrigerators in air toxics) will be checked at the next annual calibration, and notes will be added to the prior year temperature logs indicating the correction factor that should have been applied. Any thermometers outside of calibration at the next check will be removed from service. Alternatively, any requiring a correction factor will be clearly labeled, with instructions on how to record the correction factor. Since no samples have been affected, no further corrective action is needed.

**General Chemistry**

Findings:

1. The SOP for the 9060 method for soil is not consistent with current laboratory

protocol. Quadruplicate analyses are performed in accordance with AFCEE requirements, but are not addressed in the SOP. This is a repeat finding from the previous year's audit.

Response: This SOP is currently in technical review, and includes the quadruplicate analysis criteria. The SOP is to be finalized by September 20, 1999.

2. The SOP for method 365.3 (LM-CAL-1020) was not available. No approved SOP for the method was available. According to the analyst, the SOP was in progress. This is a repeat finding from the previous year's audit.

Response: This SOP, LM-CAL-1039, still needs to updated from Enseco letterhead. This entire process is being prioritized, and is anticipated to be completed by the end of October 1999.

3. No SOP for method 351.2 was available. According to the analyst an SOP was in progress, but no copy was provided. This is a repeat finding from the previous year's audit.

Response: This SOP is scheduled to be finalized by 10/31/99.

4. The SOP for Walkley-Black was dated 8/4/92 and on Enseco letterhead.

Response: The process of converting SOPs from Enseco letterhead is anticipated to be completed by the end of October 1999.

5. Supervisor review and initials had not been performed since mid-May on any log book or maintenance log reviewed.

Response: Logbooks containing raw data are reviewed daily during the data review process. For all other logbooks, the monthly review process will be re-implemented.

6. The SOP for method 340.2 was not a controlled copy.

Response: All QA documentation showed the SOP was issued as a controlled document. The missing control number was an oversight, and has been corrected and verified.

Recommendations:

1. Some of the SOPs in the General Chemistry section are older than July 1994, and are therefore printed on Enseco letterhead. It is highly recommended that all SOPs be reviewed and updated. If updating is not necessary, at least reprint them on Quanterra letterhead with a review date indicated.

Response: The process of converting SOPs from Enseco letterhead is being prioritized, and is anticipated to be completed by the end of October 1999.

**Metals Preparation and STLC/TCLP extractions**

Findings:

1. Pipettes in the metals and metals preparation areas list the date calibrated, but do not list calibration due dates.

Response: Since the SOP clearly indicates that pipettes are calibrated once a month, the last calibration date is sufficient for determining due date. This system has proven effective, and no corrective action will be taken.

2. Expiration dates are not listed on spiking solution or extraction fluid containers.

Response: All spiking solutions have expiration dates. Mercury daily standards are the only exception, and these standards have the date prepared, and a label that states the standard must be prepared daily. Expiration dates will be added to STLC and TCLP extraction solutions.

3. STLC and TCLP extraction procedures were in revision according to the analyst and not available for review.

Response: Only the STLC SOP had been recently removed for reviewed, however both SOPs were available at the time of the audit (TCLP, SOP CORP-IP-0004, effective date 9/14/99, reviewed 4/8/99, and STLC, SOP SAC-IP-0003, effective date 7/9/99). The analyst will be reminded of this, and will be asked to become re-familiarized with the SOPs in the area.

**Metals**

Findings:

1. Review signatures were missing on SOP CORP-MT-0005 for mercury analysis of water sample. The SOP is also past its review date of 12-18-97.

Response: Only the laboratory director's signature was missing from a lab specific change form from 1996. The management signature was on the original, as well as the review page from 1998. The SOP was reviewed in 1998, and the SOP is scheduled for revision by Quanterra Corporate QA.

2. The Mercury instrument maintenance schedule was manufacturer software-driven, and the information of what was performed was manually transferred to a maintenance log . Return to control was noted, but the log showed no indication of supervisor review.

Response: This is a new logbook, and will be reviewed with all the other logbooks during the next monthly review.

3. Daily working standards for mercury analysis were not tracked.

Response: The preparation of daily mercury standards are documented in the prep sheet since they undergo digestion with the sample batch. This sheet details all pertinent information for the standard preparation, including date prepared, analyst, stock standard ID, amount and expiration date. All standard information may be tracked from the prep log. All daily standards will be labeled with a statement that they are prepared daily, and the date prepared.

4. A certificate of analysis (Lot number IC-0704, Ultra ICM-441, solution 979-ICP-067) could not be located.

Response: A copy of this certificate of analysis will be requested from the vendor.

5. Multi-element solutions do not list individual analyte concentrations on the label.

Response: Standard logbooks are readily available with the stock standards with the elements listed out for quick cross-reference.

6. An out-of-date calcium standard stock solution was found. The standard was taken out of service during the audit.

Response: The analysts have been reminded to be vigilant at looking for expired standards.

**Extraction/Sample Preparation-organics**

Findings:

1. Certificate of analysis for EDB/DBCP (lot number LA76854) was not properly filed with other certificates. It was located during the audit in the box of stock standards in the refrigerator.

Response: The certificate of analysis has been returned to its proper place. The staff has been reminded of the paperwork trail and its importance.

4. The standards QC Package for an EDB standard was not in the Standard Prep file cabinet. However, a GC#69 sequence file from 2/8/99 was reviewed and showed the analysis of the standard in question.

Response: The paperwork has been regenerated and will be filed with the QC package.

5. The standard logbooks have an entry available to indicate whether or not a standard, after analysis, was acceptable for use. However, many pages in standard logbook #'s 887 and 954 did not indicate acceptability or lack thereof. Moreover, several standards observed in the GC semivolatiles lab were not labeled as being acceptable for use. According to Emily Uebelhoer and Gail Celaschi, in order to improve the system of indicating standard acceptability, the current procedure would be reviewed and updated.

Response: We have reviewed the process of documentation when accepting/rejecting QC of standards. All QC dates will appear on the standard label and the logbook will be completed after analysis.

4. Monthly review of the run and maintenance logbooks is not up to date.

Response: We have updated the review of run and maintenance logbooks in both the prep and instrument areas as of August 1, 1999.

**GC/MS, semivolatiles (8270C)**

Findings:

1. The Controlled Documents Index lists method 8270A and the pertinent SOP references method 8270C. The index should list 8270C.

Response: This typographical error in the SOP database has been corrected.

2. Run logbooks indicated method 8270, but did not list the version performed.

Response: The logbooks will be corrected when the next logbooks are generated. All raw data indicates the current method version.

3. The QC on the standard labels are blank. According to the analyst, this area is to be filled in following acceptance of the standards following comparison to a second source.

Response: We have reviewed the process of documentation when accepting/rejecting QC of standards. All QC dates will appear on the standard label and the logbook will be completed after analysis.

4. According to method 8270C, the internal standards (ISs) in the CCV are to be evaluated against the ISs in the ICAL mid-point standard. However, according to Donald Taylor, this procedure has not been followed consistently, in part due to current limitations between the mass spec upload software and the

LIMS. Until IS evaluation is automated, this procedure must be done manually for every CCV.

Response: The automated software, called Data Checker, has been implemented. Data Checker is checked after each run, and will indicate any out of control QC, including checking the IS's in the CCV evaluated against the ICAL.

**Gas Chromatography, Extractables**

Findings:

1. The implementation date on SOP SAC-GC-0007 (LUFT- Extractables) was blank, although it was revised in September 1998.

Response: The implementation date of 10/15/98 will be added to the controlled copy of the SOP to complete the documentation.

2. The QC on the standard labels are blank. According to the analyst, this area is to be filled in following acceptance of the standards following comparison to a second source.

Response: We have reviewed the process of documentation when accepting/rejecting QC of standards. All QC dates will appear on the standard label and the logbook will be completed after analysis.

Recommendations:

1. Chromatograms for EDB/DBCP (method 504.1) standards indicate interferences from one or more compounds. Some of these non-target peaks were also present in the reviewed method blank. It is recommended that an EDB/DBCP standard be made up in methanol and analyzed by GC/MS to possibly determine these interferences.

Response: The interference peaks are at too low of concentration to be detected by GC/MS. These peaks do not create an interference to either quantitation or qualification of EDB/DBCP.

2. On samples analyzed by both 504.1 and 8260, 504 results should be compared to those of the 8260 analysis.

Response: This "sanity" check of similar analyses will be conducted at the project management review stage.

**GC/MS, volatiles (8260B)**

Findings:

1. Updated method, 8260B, was not identified in the runlog. Only 8260 was noted. This is a repeat finding.

Response: The cover page of the run log indicates the method and version used. Possible re-programming of the electronically generated logbook will be investigated. If re-programming is not possible, the method will be added to the logbook page as new logbooks are generated.

2. Refrigerator blanks are routinely placed in the sample refrigerator by the QA department but are not always analyzed per the analyst. The blanks have a much lower priority than clients samples. Following analysis, the data is sent back to QA.

Response: Refrigerator blanks appear on the VOA work list with the normal sample load. The analysts are assigned samples to analyze, including refrigerator blanks. Samples with the shortest holding time and rush work is given priority is instrument space is limited. Refrigerator blanks do not have a holding time, but are tracked instead by a minimal residence time of 30 days in the refrigerator being monitored, as per Quanterra SOP SAC-QA-0002. Refrigerator blanks are always analyzed, and data is maintained in the QA department.

**GC, Volatiles**

There were no findings in this section.

Recommendations:

1. A peer review stamp was used in the maintenance logbooks. It is recommended that subsequent stamps be applied on the opposing blank page, from top to bottom, for greater legibility.

Response: Applying the peer review stamp from top to bottom is a good idea and will be utilized in the future. As the opposing pages of the logbook are not numbered, they are not considered valid pages, and will not be peer reviewed.

2. According to Kyle Stephens, headspace that would prevent the use of a VOA vial was determined to be any bubble in excess of 4 mm in size. However, Mr. Stephens was not sure if the pertinent SOP(s) addressed this issue. If not already done, discussion of unacceptable bubble size in VOA vials should be part of all pertinent SOPs.

Response: Samples with bubbles of any size are anomolized. The SOP's will be amended to reflect this procedure by 10/31/99.

**GC/MS, Dioxins and Dioxins Preparation - low resolution (8280 and 8280A) and high resolution (8290)**

Findings:

1. The maintenance logbooks showed no indication of supervisor review.

Response: Logbooks in this area are reviewed by peer review. All logbooks have a review system, either at the beginning of the logbook, or in the margins.

2. Run logs were chronologically placed in a three-ring binder instead of in a bound notebook. According to the analyst, these were periodically transferred to a bound notebook similar to those in other departments; however, the transfer had not taken place for over six months. The run log showed no indication of supervisor review.

Response: The transfer of pages from this logbook is based on number of pages (approximately 500) and not a time period. These pages are peer reviewed consistently.

**HPLC Extraction/Sample Analysis – 8330 and 8330M (nitroguanidine); 8321 CWM**

Findings:

1. The SOP for method 8321-CWM (Chemical Warfare Materials) was not available. According to the analyst the SOP is in draft form.

Response: This SOP is scheduled to be finalized by September 10, 1999.

2. The standard logbooks have an entry available to indicate whether or not a standard, after analysis, was acceptable for use. However, the "QC'd by" in the standard logbook was not filled out. Acceptability of use had been received by e-mail, but was not documented in the logbook.

Response: This system is being evaluated, and an electronic tracking system will be developed.

3. The temperature of the refrigerator had not been read the day of the audit. The area was visited in the afternoon.

Response: As was noted during the audit, the analyst had just started his shift. The refrigerators are consistently monitored by the split-shift analyst, or other arrangements are made to have the temperature recorded daily. The logbook showed no other gaps, and the temperature was checked that day, as scheduled.

4. The header on the current page in the balance log was incomplete – no model or balance ID was entered.

Response: The header of the balance logbook has been completed.

5. MDL studies were overdue.

Response: The MDL for the 8321 CWM method has been scheduled for September 1, 1999.

6. A low level standard is run at the end of the sequence in order to confirm that

the instrument would still detect contamination down to the reporting limit. No acceptance criteria were stipulated for this standard in the SOP.

Response: This low level standard is not a requirement of the method or Quanterra policy, but is a client specific requirement. The criteria is established in the client QAPP. The standard is evaluated for signal to noise ratio. The ratio should be 10:1 S/N, and if it is less than that criteria, the analyst will evaluate the system for possible sources of error. The S/N ratio must be greater than 5:1, or corrective action and re-analysis is performed.

**GC/MS, semivolatiles (CWM)**

Finding:

1. The SOP was a draft, dated 3/31/99. The method is referenced as 8270 – however, since there are so many modifications it would be advisable to just call it Semivolatile GCMS – CWM.

Response: Method 8270 is not referenced this in this SOP. The SOP title is "Preparation and Analysis of Organosulfur Compounds (OS) and Tear Gas Degradates (TGD) in Water and Soil by Gas Chromatography with a Mass Spectrometer (GC/MS).

**Quality Assurance Management**

Findings:

1. SOPs were either not available or were in need of updating for some general chemistry methods – see discussion in this section. Draft SOPs were used in some areas to perform the analyses.

Response: The QA department has been making a concerted effort to update all Enseco SOPs, finalize all draft SOPs, create SOPs where lacking, and finally to review all existing SOPs according to the two-year schedule. These activities are listed according to the current prioritizing of events. While this process is on-going, we have made definite advances in the last few months.

2. The required review of SOPs are overdue. These reviews are not documented on the SOP, and it is therefore difficult to determine when and if they are actually done.

Response: See previous response for review scheduling. A better tracking system will be implemented to track any completed reviews. This will include an update to the electronic database, and issuing a review sheet to accompany all controlled copies of SOP (currently, the review form is maintained in the QA department).

Concerns:

1. Quanterra has been subcontracting perchlorate analyses for Earth Tech to CLS laboratories. However, no audit of the perchlorate method has been performed at CLS. An audit for method 8260 only had been performed some time ago.

Response: The audit of CLS will be arranged as times are available before the end of the year.

Findings:

1. The records for thermometer calibration were in disarray. However, it appears that the NIST traceable calibration thermometer 1E3319 was last calibrated on 8/2/1995 and was used for the refrigerator calibrations on 8/26/98. It also appears that the metals preparation thermometers were last calibrated in-house in 1997. The IR thermometer 015609 (presumably used in sample receiving) was last calibrated on 2/11/98. Thermometers are not tagged with calibration date or calibration due date. Therefore it is of utmost importance that the records in the QA department, which are the only source of information regarding calibrations, be kept organized and up to date.

Response: All prior thermometers calibrations have been organized and bound. A new bound logbook has been created for all thermometer calibrations going forward. This system will allow better tracking of thermometer calibration. Thermometers will be tagged whenever possible with the most recent calibration date. IR thermometer 015609 is labeled IR-1, and had been most recently calibrated on 4/29/99.

2. Calibration records for two thermometers, lab #s 4552 and 4040, assigned to Air Toxics showed a 1 degree difference from the NIST thermometer; however, no correction factor was listed. If these thermometers are indeed used, there must be

a correction factor assigned and noted on the temperature log.

Response: The thermometers that exceeded the calibration range during the last annual calibration were not used in any refrigerators containing samples. These two thermometers (located in air toxics) will be checked at the next annual calibration, and notes will be added to the prior year temperature logs indicating the correction factor that should have been applied. Any thermometers outside of calibration at the next check will be removed from service. Alternatively, any requiring a correction factor will be clearly labeled, with instructions on how to record the correction factor. Since no samples have been affected, no further corrective action is needed.

3. One balance, QA38, was found to be out of tolerance when calibrated. It was taken out of service the day of calibration. How was the data handled that was generated by this balance prior to the calibration date?

Response: The balance had met daily calibration requirements, so no corrective action was taken.

4. There is no current log for tracking the weight sets, i.e., listing locations.

Response: An inventory of the weight sets, including location, was available in the weight calibration file for 1998. This inventory will be updated as needed and included in the 1999 weight calibration file.

5. The 200 mg weight for set QA-033 was listed as out of tolerance with no adjustment. Is this weight still in the set? Calibration records indicate that a different weight value should be used for this weight. What is being done?

Response: The 200 mg weight is not routinely used from that weight set. Although the weight was out of tolerance for a class S weight (± 0.025 mg), it was within the specifications for balance calibration use of class Q ( 0.12mg), and is traceable to a class S set. Since that weight is not used, no corrective action will be taken beyond the labeling that is already on the weight set.

**Personnel Training**

Concerns:

1. Most resumes reviewed were out of date

Response: Most resumes have been updated electronically, but printouts have not been placed in the training files. Links to the resumes will be established with the new training records database.

2. There were no records of training on sample receiving protocol except handling of hazardous waste for Ray Lucero, and during the interview with Mr. Lucero he did not appear to be familiar with all protocol required of sample receiving personnel.

Response: The sample receiving personnel training records will be updated to include all training, including the QA corrective action meeting to be scheduled based on these audit findings. Documentation will include acknowledgement that the analyst has read the SOP and agree to abide by the SOP.

Quanterra
Environmental Services

***Pamela A. Schemmer***
*Quality Assurance Manager*

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August 30, 1999

Elisabeth Fruth
Earth Tech
100 West Broadway
Suite 5000
Long Beach, CA 90802-4443

***Subject: Audit Response***

Dear Elisabeth:

Enclosed is a clarification to he previous audit response. If you have any questions in regards to this response, please feel free to call Pamela Schemmer at (916) 374-4441.

Sincerely,

Pamela A. Schemmer
Quality Assurance Manager

Eric Redman
Laboratory Manager

enclosure

1. Several sample container mix-ups have occurred at the West Sacramento facility. For instance, for the Site 4 Landfill in January (samples 4-MW02-WG26 and 4-MW01-WG26 were switched) and in April (4-MW06-WG224 and 4-MW04-WG27, inorganics, were switched). For Norton, lot 303515, the trip blank and sample MLW-2-3 were switched. Furthermore, for sample G9D150172-006 it is suspected that a vial for method 504.1 was switched. Per sample receiving personnel, it has happened that analysts will return bottles to sample receiving if the field ID and the lab ID on the bottle do not match written information. Although the system of setting up the bottles grouped by field ID should work, no double checking is performed. Earth Tech is truly concerned with the repeated incidence of known sample mix-ups, and even more concerned with possible undetected mix-ups.

Response: The normal procedure is to lay out all the samples according to the chain of custody with labels, and have this verified by another analyst prior to labels being applied. We believe the discrepancy with Earth Tech has arisen due to the Earth Tech requirement that samples are numbered according to field Ids, and not the chain of custody. This issue has been addressed with the sample receiving personnel, and will be addressed in a follow-up QA meeting with the pertinent staff, as well as QA spot audits.

Clarification: With each shipment from Earth Tech, we receive a Field Sample Identifier / Code Reference Sheet along with a Chain of Custody. This additional sheet lists all of the samples, depth and collection time, but not the bottle types or amount. Frequently, these two documents list the samples in the same order, but not always. It is our understanding that Earth Tech prefers the report to be in order of the Field Sampler Identifier instead of the Chain of Custody when there is a difference in order between the two. For example, the first sample on the list would be –001, the second would be –002, and so on. We are able to log samples into our system in this order easily. However, our labeling system has been to label according to the COC, as this is our normal log-in order. In this case, if there is a difference in order, the bottles may be mislabeled. Sample receiving staff has been reminded to use a sheet generated by the system to match labels to bottles instead of relying on the COC, which does not have the LIMS number. While this theory has not been verified to be the cause of mislabeling, it is a possibility due to the unique procedure of the Field Sample Identifier. Awareness of this potential problem will hopefully assure more careful checking of sample labels.

100 West Broadway, Suite 5000, Long Beach, California [illegible]

August 30, 1999

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880 Riverside Parkway
West Sacramento, CA 95605

Attn.: Eric Redman/Pamela Schemmer

I have received the audit response dated August 20, 1999, and the requested clarification to audit response dated August 30, 1999. The responses to the audit deficiencies are acceptable and the audit is closed.

However, it is apparent that there is a misunderstanding regarding the use of the Field Sample Identifier / Code Reference Sheet (FSI/CRS). This data sheet was developed in concert with the Quanterra Santa Ana laboratory as an aid in logging in the samples per the IRPIMS and Earth Tech Standard EDD formats. Therefore, this sheet includes the actual database field names, as well as the "sample type" field, which is not included on the COC. There was never any instruction or requirement that the samples had to be logged in in the order they were listed on the FSI/CRS. The use this form, which is merely provided as a log-in aid, is optional, but we feel that using this form has improved the accuracy of the electronic deliverables. Please clarify this issue with your sample receiving personnel. This has never been an issue at any other laboratory that has used this form.

Telephone
562.951.2000
Facsimile
562.951.2100

Very truly yours,

Earth Tech

Elisabeth Fruth

Elisabeth Fruth
QA Manager

100 West Broadway, Suite 5000, Long Beach, California 90802-4443

September 8, 2000

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Telephone
562.951.2000
Facsimile
562.951.2100

Attn.: Eric Redman/Pamela Schemmer

I have received the follow-up audit response dated September 1, 2000. The responses to the audit deficiencies are acceptable and the audit is closed.

Very truly yours,

Earth Tech

Elisabeth Fruth

Elisabeth Fruth
QA Manager

Quanterra
Environmental Services

*STL Sacramento*
*880 Riverside Parkway*
*West Sacramento, CA 95605*

*(916) 374-4441 Direct*
*(916) 372-1059 Fax*

September 1, 2000

Elisabeth Fruth
Earth Tech
100 West Broadway
Suite 5000
Long Beach, CA 90802-4443

***Subject: Audit Response***

Dear Elisabeth:

Enclosed is our follow up response to audit findings from June 20, 21, 2000 for Edwards AFB and Norton AFB. If you have any questions in regards to this response, please feel free to call Pamela Schemmer at (916) 374-4441.

Sincerely,

Pamela Schemmer

Pamela A. Schemmer
Quality Assurance Manager

enclosure

## General Findings

1. Supervisor review of maintenance logbooks was not performed on a monthly basis as specified by the laboratory. This problem was consistent throughout the laboratory.

- **A reminder of the policy will be sent out to all area leaders. All logbooks should be reviewed on a monthly basis, with the following exceptions. Logbooks that complete less than one page per month may be reviewed on a page by page basis. Logbooks that are copied and reviewed as part of a data package will have notes for peer review on a 3 month basis in the logbook itself, as each page is reviewed daily on a photocopy.**
- **See attached email memo sent to all area leaders.**

2. The general policy throughout the laboratory is for analysts to prep and report a DCS when a MS/MSD is not prepared. Because of this policy we frequently receive in our final reports LCSD data when it is not required. This is a client specific requirement that has not been communicated to the analysts.

- **These requirements will be conveyed to the laboratory via the QAS system, now in effect.**
- **The QAS system was implemented 8/1/00.**

3. The QC acceptance date for the standards is not consistently documented on the standard labels and in the standard logbooks. Although this field of information is not required by Earth Tech, last year's audit response indicated that it would be filled out, and if it is provided it should be filled out.

- **QC acceptance of standards will now only be tracked on the Standard QC form, which is filed with the QC data.**
- **The new system, which will continue to use the existing labels, was implemented on 8/1/00.**

5. Several of the corrective actions proposed in the response to the Earth Tech audit of 7/99 have not been implemented.

- **See responses below.**
- **All deficiencies noted during the audit have been addressed in this response. Please refer to these responses for revised timelines.**

## General Chemistry

Findings:

1. Recent laboratory reports have not included information on laboratory duplicates as required by Earth Tech.

- **This has only occurred for percent moisture determinations, which is performed in our Organic Preparation area. The laboratory has developed a system to assure duplicates are performed as requested. This system has been in place and operating effectively since June.**
- **This has been verified by the QA department on 9/1/00. If there are any concerns that exist concerning this issue, please notify the laboratory.**

2. Certificates of analyses were not available for all method standards that originated from salts. Air Force work requires certificates of analysis for all methods.

- **Certificate of analysis will be obtained for stock chemicals. Also, manufactured stock solutions will be purchased with certificate of analyses.**
- **This will be completed by November 30, 2000.**

5. The alkalinity logbook did not include the criteria for standardization of pH and electrical conductivity.

- **The logbooks will be revised to include this criteria.**
- **Logbook revision to be completed by November 30, 2000.**

8. The instrument ID is not specified on the cover page for the total cyanide distillation instrument logbook (#1573) and the LACHAT autoanalyzer logbook (#1525).

- **All logbooks in the area have been checked and all instrument IDs have been placed on the cover.**
- **This was verified by QA on 9/1/00. Copies of the logbook covers will be included with this response.**

9. A 2 mL pipette (WC-144530) did not have a calibration record tag. According to the analyst, this pipette was taken out of service. However, this was not documented in the pipette calibration logbook, and the pipette was still left in the laboratory with another pipette that is currently being used.

- **All out of service pipettes have been removed from the area, and will be better documented in the future when they are taken out of service.**
- **All out of service pipettes will be removed from the laboratory. Since the calibration logbooks are not meant to be maintenance or tracking logs, pipettes that will not calibrate will be replaced. This practice was verified by QA on 9/1/00.**

10. The SOP for alkalinity (SAC-WC-0028) references method 310.1, not 2320 which is specified by Earth Tech. The implementation date is missing on the cover page for this SOP.

- **The implementation date has been added to all SOPs in the area. The Alkalinity SOP will be revised to include the reference to Standard Methods 2320 by October 31, 2000.**
- **The implementation dates have been added to all SOPs in the General**

**Chemistry area. This was verified by QA on 9/1/00. Copies of the SOP cover pages have been included with this response.**

13. The implementation date is missing on the cover page for the SOP for ammonia-N by method 350.1 (SAC-WC-0105) and sulfide by method 376.2 (SAC-WC-0014).

- **The implementation date has been added to all SOPs in the area.**
- **The implementation dates have been added to all SOPs in the General Chemistry area. This was verified by QA on 9/1/00. Copies of the SOP cover pages have been included with this response.**

Recommendations:

2. Out of control incidents were not clearly documented on the oven temperature chart. The temperature, 80 degrees C, of the drying oven #4 was out of the acceptable range (73-77 degrees C) on 6/15/00. No documentation or explanation was recorded on the oven temperature chart. The analyst noted "re-adjust" in the comment section. It is recommended that the corrective action taken and return to control be documented.

- **Better documentation procedures have been implemented to indicate out of control temperatures, corrective actions taken, when the equipment is brought back into control, and the initials of the analyst and date of corrective action.**
- **Copies of the oven temperature log have been included with this response.**

**Metals Preparation and Analysis and STLC/TCLP extractions**

Findings:

5. Expiration date was not included on the containers for three standards: trace ICP soil MDL 922-066, soil MDL spike 1237-ICP-015, and ICP soil mix solution 1237-ICP-023.

- **Expiration dates will be added to all bottles.**
- **Implemented the date of the audit. Verified by QA 9/1/00.**

**Extraction/Sample Preparation-organics**

Findings:

4. The concentration of the source standard (pesticides MS S053100B) is not specified in the standard logbook (#1533) on 5/31/00 (page 4).

- **This standard page will be amended.**
- **This was corrected on 7/24/00. A copy of this logbook page is included with this response.**

**Gas Chromatography, Extractables**

Findings:

4. The standard refrigerator contained herbicide standards that were labeled as 8150, but the actual method in use is 8151A. Labeling needs to be accurate.

- **The analyst will be asked to update the labels and the storage box. Since Method 8150 has been replaced entirely with Method 8151A, no confusion would arise from this mislabeling.**
- **Effective 7/15/00. Verified by QA 9/1/00.**

6. The run logbook for herbicides had not been reviewed. This is a problem throughout the laboratory. There needs to be a policy regarding how often logbooks need to be reviewed, and this policy needs to be implemented.

- **The area leaders will be reminded to review all logbooks on a monthly basis.**
- **See General Finding #1.**

**GC/MS, Dioxins Preparation - low resolution (8280A) and high resolution (8290)**

Findings:

3. The standard refrigerator (R58) located in the low-resolution prep lab had not month or year indicated on the checklist, and it had not been checked since the fifteenth of the month. Refrigerators need to be checked daily and all header information should be completed.

- **Corrective action was taken immediately and personnel were reminded that temperature checks need to be done daily.**
- **QA verified 9/1/00.**

4. Header information (balance ID, model #) was not recorded consistently on each page for logbook #619.

- **Corrective action was taken immediately and personnel were reminded that all information must be recorded in the logbook.**
- **QA verified 9/1/00.**

5. The oven located in the low-resolution prep lab (VWR1305) was monitored inconsistently during the month of June.

- **Corrective action was taken immediately and personnel were reminded that temperature checks need to be done daily.**
- **QA verified 9/1/00.**

## Schemmer, Pamela

**From:** Schemmer, Pamela
**Sent:** Friday, September 01, 2000 3:26 PM
**To:** Sacramento Area Leaders
**Cc:** Krsulic, Nancy; Stafford, Lisa; Votaw, Barry
**Subject:** Logbook review

It is the beginning of the month, and a good time to think about logbook review. Our policy is that all logbooks should be reviewed by the area leader (or peer reviewed) monthly. The following exceptions apply:

Logbooks that complete less than one page per month may be reviewed on a page by page basis. Reviews should occur on a quarterly basis at a minimum. Just mark how much of the page was completed at time of review. This may include balance calibration logs, or instrument maintenance logs.

Logbooks that are copied for data packages, and the copy is reviewed as part of the second level review of the data package: Review of the logbook page should be indicated during second level review, either by the reviewer initial and dating the copy, or as part of the second level review checklist. The original logbook should be marked (on the front review page), on a quarterly basis, that each page was reviewed as part of the daily data review process.

Thanks for your cooperation. If you have any questions about a particular logbook review schedule, please notify me.

*Pamela Schemmer*
*QA Manager*
*STL Sacramento*
*916-374-4441*
*pschemmer@stl-inc.com*

Instrument I.D. # NA

STL

Committed To *Your* Success

# Title: Total Cyanide Distillation ~~Instrument~~ Log

9/1/00

Logbook # 1573

Start 5·1·00 End

d:\qsacca01\sacqa\logbook.dot

Instrument I.D. # AA1

Quanterra

Environmental Services

# LACHAT AUTOANALYZER LOGBOOK

Logbook # 1524

Start 1-7-2000 End 7-11-00

Instrument I.D. # AA1

SEVERN
TRENT
SERVICES

# Title: LACHAT AUTOANALYZER LOGBOOK
## QA-173

Logbook # 1630

Start 7/12/00 End

d:\qsacca01\sacqa\logbook.dot

Controlled Copy
Copy No. 1

Implementation Date: 7/9/99

Quanterra Environmental Services

SOP No.SAC-WC-002
Revision No.1.0
Revision Date: 7/2/99
Page: 1 of 15

# OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

## TITLE: DETERMINATION OF ALKALINITY, CONDUCTIVITY AND PH

## (SUPERSEDES: LM-CAL-1056 )

Prepared by: Kuldip Hundal

Reviewed by: Technical Specialist

Approved by: Pamela Schemmer
Quality Assurance Manager, Pamela Schemmer

Approved by: Pamela Schemmer
Environmental Health and Safety Coordinator

Approved by:
Laboratory Manager, Eric Redman

Proprietary Information Statement:

This document has been prepared by and remains the sole property of Quanterra Incorporated. It is submitted to a client or government agency solely for its use in evaluating Quanterra's qualifications in connection with the particular project, certification, or approval for which it was prepared and is to be held proprietary to Quanterra.

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Implementation Date: 7/9/99

Quanterra Environmental Services

SOP No. SAC-WC-0105
Revision No. 2.1
Revision Date: 11/18/97


# OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

## TITLE: AMMONIA AS N (LACHAT AUTOANALYZER)

## (SUPERSEDES: LM-CAL-1105, REVISION 2.0, MARCH 6, 1990)

Prepared by: Hamid Foolad

Reviewed by: _____
Technical Specialist

Approved by: _____
Quality Assurance Manager, Pamela Schemmer

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Manager, Eric Redman

Proprietary Information Statement:

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Controlled Copy
Copy No. 1

Implementation Date: 7/9/99

Quanterra Environmental Services

SOP No. SAC-WC-001
Revision No. 1.0
Revision Date: 9/02/98
Page: 1 of 13

# OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

## TITLE: SULFIDE ANALYSIS BY EPA 376.2 - MODIFIED

## (SUPERSEDES: LM-CAL-1042)

Prepared by: Kuldip Hundal

Reviewed by: Technical Specialist

Approved by: Pamela Schemmer
Quality Assurance Manager, Pamela Schemmer

Approved by: Pamela Schemmer
Environmental Health and Safety Coordinator

Approved by:
Laboratory Manager, Eric Redman

Proprietary Information Statement:

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# QUANTERRA INCORPORATED
**West Sacramento**
## Temperature Log

Thermometer ID 227-520 Location Gen Chem

Unit # OVEN 2 Month/Year 08 - 2000

**Temperature must be within 103 - 105 °C.**

If temperature readings fall outside this range, inform the QA Department. If the date indicated falls on a weekend, write "WKND" in the space.

| Date | Temperature | Initials | Date | Temperature | Initials |
|---|---|---|---|---|---|
| 1 | 104 | TN | 17 | 103 | TN |
| 2 8/8/00 LW | 100 | adjusted no samples | 18 | 110* | TN No samples. |
| 3 | 105 | LW | 19 | WKEND | |
| 4 | 105 | LW | 20 | | |
| 5 | wkend | | 21 | 105 | LW |
| 6 | | | 22 | 105 | TN |
| 7 | 105 | LW | 23 | 105 | TN |
| 8 | 110* | TN No sample. TN 8-8-00 | 24 | 108* | TN No samples. TN 8-24- |
| 9 | 105 | LW | 25 | sink adjust. | No samples. TN 8-25-0 |
| 10 | 105 | TN | 26 | WKEND | |
| 11 | WKEND | | 27 | | |
| 12 | | | 28 | 103 | TN |
| 13 | TN | 108 No sample. TN 8-13-00 | 29 | 103 | TN |
| 14 | 103 | LW | 30 | 103 | TN |
| 15 | 104 | LW | 31 | 105 | TN |
| 16 | 104 | LW | | | |

Comments:

Please be sure to anomalize any excursions or omissions on this log sheet.

QA-0129 7/94 RMH

# QUANTERRA INCORPORATED
West Sacramento
## Temperature Log

Thermometer ID 4059 Location Gerchem

Unit # 6VEN 4 Month/Year 08-2000

**Temperature must be within 73 - 77 °C.**

If temperature readings fall outside this range, inform the QA Department. If the date indicated falls on a weekend, write "WKND" in the space.

| Date | Temperature | Initials | Date | Temperature | Initials |
|---|---|---|---|---|---|
| 1 | 75 | TN | 17 | 76 | TN |
| 2 | 78 adjusted | NO SAMPLED 8/4/00 LW | 18 | 75 | TN |
| 3 | 76 | TN | 19 | WKEND | |
| 4 | 75 | TN | 20 | | |
| 5 | WKEND | | 21 | 75 | TN |
| 6 | | | 22 | 75 | TN |
| 7 | 75 | TN | 23 | 75 | TN |
| 8 | 76 | TN | 24 | 75 | TN |
| 9 | 75 | TN | 25 | 75 | TN |
| 10 | 75 | TN | 26 | 75 | TN |
| 11 | 77 | TN | 27 | 75 | TN |
| 12 | WKEND | | 28 | 75 | TN |
| 13 | | | 29 | 75 | TN |
| 14 | 74 | TN | 30 | 75 | TN |
| 15 | 75 | TN | 31 | 74 | TN |
| 16 | 76 | TN | | | |

Comments:

Please be sure to anomalize any excursions or omissions on this log sheet.

QA-0129 7/94 RMH

# Quanterra Environmental Services, West Sacramento
## GC SEMI-VOLATILES
### STANDARDS PREPARATION
### QUALITY CONTROL LOGSHEET

TE 5-31-00 STD ID 5053100 B

:PARED BY [signature] STD'S NAME 3-20 pm Mix spike

_VENT Acetone LOT # 1X451 W46H03 LOCATION Org prep

| IPOUND/SOLUTION | VENDOR | LOT # | CONC. (ug/mL) | VOL. USED (mL) | FINAL VOL. (mL) | FINAL CONC. (ug/mL) |
|---|---|---|---|---|---|---|
| LTRA SCIENTIFIC 250 Smith St., North Kingstown, RI 02852 401-294-9400 | | | | 2 ml | 200 ml | 0.5 - 1.0 µg/ml |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| LDRIN | | | 50.2 | 2.0 | 200 | 0.5 |
| RBHC | | | 50.0 | | | 0.5 |
| DIELDRIN | | | 100.2 | | | 1.0 |
| 4,4'-DDT | | | 100.2 | | | 1.0 |
| NDRIN | | | 100.2 | | | 1.0 |
| PTACHLOR | | | 50.0 | | | ~~0.05~~ 0.5 |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |
| | | | | | | |

LAB USE ONLY

: No. Exp. Date

EWED BY: [signature] DATE: 6/12/00 EXP. DATE: 11-30-00

ACCEPTABLE FOR USE? YES ☑ NO ☐

ENTS:

• %DIFFERENCE = $\frac{RRFr - RRFs}{RRFr} \times 100$

where RRFr = relative response factor for the reference standard
where RRFs = relative response factor for the new standard

•• Standards/Mixes may be made in varying amounts - please document changes!

Page No. 04

QA-0158 RMH 7/95

100 West Broadway, Suite 5000, Long Beach, California 90802-4443

August 8, 2000

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

Attn. Pamela Schemmer

Subject: Audit Response

Telephone
562.951.2000
Facsimile
562.951.2100

Dear Pamela,

We have reviewed your responses to our audit findings from June 20/21, 2000. While most responses appear adequate in content, no verification of implementation was provided.

There are numerous responses that contain the statement "will be" without any completion date, e.g., general findings #1, #2 and #3, gen chem findings #2 and #5, metals #5, extraction #4, GC #4 and #6. Some of these were simple enough that they should already be done, and a copy of the correction or the memo, e-mail or what ever could have been provided with the audit response.

Numerous responses indicate that the corrective action has been performed, e.g., gen chem findings #1, #8, #9, #10 and #13, and recommendation #2, dioxins #3, #4 and #5. However, no verification was provided.

Audit findings in this audit and previous audits show that implementation of corrective action has been deficient at the West Sacramento facility in the past, it is very important that timely implementation and verification of corrective action be performed. Corrective action with a listed time line will be checked during next year's audit. Verification of other items, where possible, should be provided with your response to this letter.

Very truly yours,

Earth Tech

Elisabeth Fruth

Elisabeth Fruth
QA Manager

Quanterra
Environmental Services

*STL Sacramento*
*880 Riverside Parkway*
*West Sacramento, CA 95605*

*(916) 374-4441 Direct*
*(916) 372-1059 Fax*

July 24, 2000

Elisabeth Fruth
Earth Tech
100 West Broadway
Suite 5000
Long Beach, CA 90802-4443

***Subject: Audit Response***

Dear Elisabeth:

Enclosed is our response to audit findings from June 20, 21, 2000 for Edwards AFB and Norton AFB. If you have any questions in regards to this response, please feel free to call Pamela Schemmer at (916) 374-4441.

Sincerely,

Pamela Schemmer

Pamela A. Schemmer
Quality Assurance Manager

Eric W. Redman
Laboratory Manager

enclosure

**General Findings**

1. Supervisor review of maintenance logbooks was not performed on a monthly basis as specified by the laboratory. This problem was consistent throughout the laboratory.

- **A reminder of the policy will be sent out to all area leaders. All logbooks should be reviewed on a monthly basis, with the following exceptions. Logbooks that complete less than one page per month may be reviewed on a page by page basis. Logbooks that are copied and reviewed as part of a data package will have notes for peer review on a 3 month basis in the logbook itself, as each page is reviewed daily on a photocopy.**

2. The general policy throughout the laboratory is for analysts to prep and report a DCS when a MS/MSD is not prepared. Because of this policy we frequently receive in our final reports LCSD data when it is not required. This is a client specific requirement that has not been communicated to the analysts.

- **These requirements will be conveyed to the laboratory via the QAS system, now in effect.**

3. The QC acceptance date for the standards is not consistently documented on the standard labels and in the standard logbooks. Although this field of information is not required by Earth Tech, last year's audit response indicated that it would be filled out, and if it is provided it should be filled out.

- **QC acceptance of standards will now only be tracked on the Standard QC form, which is filed with the QC data.**

4. Requests for amended reports are not being processed in a timely manner.

- **This is being handled according the schedule agreed upon by STL and EarthTech from the meeting on July 7, 2000.**

5. Several of the corrective actions proposed in the response to the Earth Tech audit of 7/99 have not been implemented.

- **See responses below.**

**Sample Receiving and Control**

There are no findings for this area.

**General Chemistry**

Findings:

1. Recent laboratory reports have not included information on laboratory duplicates as required by Earth Tech.

- **This has only occurred for percent moisture determinations, which is**

**performed in our Organic Preparation area. The laboratory has developed a system to assure duplicates are performed as requested. This system has been in place and operating effectively since June.**

2. Certificates of analyses were not available for all method standards that originated from salts. Air Force work requires certificates of analysis for all methods.

- **Certificate of analysis will be obtained for stock chemicals. Also, manufactured stock solutions will be purchased with certificate of analyses.**

3. The CCV working standard (924-WC-80-6) label for method 300.0 did not contain the concentration units of the analytes. This information was added at the time of the audit.

- **Corrective action completed during audit.**

4. The electrical conductivity meter logbook did not contain the criteria for the daily calibration of the instrument.

- **The logbook will be revised to include acceptance criteria, and will be replaced by September 7, 2000.**

5. The alkalinity logbook did not include the criteria for standardization of pH and electrical conductivity.

- **The logbooks will be revised to include this criteria.**

6. Uncontrolled Enseco and Quanterra (some SOPs were not stamped "uncontrolled") were found in all maintenance logbooks and balance calibration logbooks in this area. The uncontrolled SOPs were marked "obsolete" during the audit.

- **The practice of putting uncontrolled SOPs at the front of new logbooks was eliminated in January 2000. No new logbooks have uncontrolled SOPs at the beginning. Logbooks that have these SOPs are being marked "Obsolete", with the date marked, until all logbooks are replaced.**

7. Maintenance logbooks (#256 for Dionex 2000 and #303 for IC DX-100) were last reviewed on 11/30/99, and the balance logbooks had not been reviewed since 11/29/99. It is not clear what the policy is regarding the frequency of logbook review. However, there should be a policy in place that establishes consistency with regard to this issue.

- **A better effort will be made to review logbooks on a monthly basis.**

8. The instrument ID is not specified on the cover page for the total cyanide distillation instrument logbook (#1573) and the LACHAT autoanalyzer logbook (#1525).

- **All logbooks in the area have been checked and all instrument IDs have been placed on the cover.**

9. A 2 mL pipette (WC-144530) did not have a calibration record tag. According to the analyst, this pipette was taken out of service. However, this was not documented in the pipette calibration logbook, and the pipette was still left in

the laboratory with another pipette that is currently being used.

- **All out of service pipettes have been removed from the area, and will be better documented in the future when they are taken out of service.**

10. The SOP for alkalinity (SAC-WC-0028) references method 310.1, not 2320 which is specified by Earth Tech. The implementation date is missing on the cover page for this SOP.

- **The implementation date has been added to all SOPs in the area. The Alkalinity SOP will be revised to include the reference to Standard Methods 2320 by October 31, 2000.**

11. The SOP for pH references method 150.1 for water samples.

- **The SOP will be amended to include the reference to SW-846 Method 9040B by October 31, 2000.**

12. The SOP for fluoride references method 340.2, not SM4500F.

- **The SOP will be amended to include the reference to SM4500F by November 30, 2000.**

13. The implementation date is missing on the cover page for the SOP for ammonia-N by method 350.1 (SAC-WC-0105) and sulfide by method 376.2 (SAC-WC-0014).

- **The implementation date has been added to all SOPs in the area.**

Recommendations:

1. A number of certificates of analysis have not been properly filed in the notebook. Instead they have been stored in the sleeve of the notebook. These certificates should be filed.

- **The Certificates of analysis have been properly filed in the appropriate place within the Certification of Standards Binder.**

2. Out of control incidents were not clearly documented on the oven temperature chart. The temperature, 80 degrees C, of the drying oven #4 was out of the acceptable range (73-77 degrees C) on 6/15/00. No documentation or explanation was recorded on the oven temperature chart. The analyst noted "re-adjust" in the comment section. It is recommended that the corrective action taken and return to control be documented.

- **Better documentation procedures have been implemented to indicate out of control temperatures, corrective actions taken, when the equipment is brought back into control, and the initials of the analyst and date of corrective action.**

**Metals Preparation and Analysis and STLC/TCLP extractions**

Findings:

1. Uncontrolled Enseco and Quanterra SOPs were found in the metals reagent tracking logbook (#603) and balance calibration logbooks (#103 and #1245). The uncontrolled SOPs were marked "obsolete" during the audit.

- **The practice of putting uncontrolled SOPs at the front of new logbooks was eliminated in January 2000. No new logbooks have uncontrolled SOPs at the beginning. Logbooks that have these SOPs are being marked "Obsolete", with the date marked, until all logbooks are replaced.**

2. Pipettes (MP010 and MP011) which were last calibrated on 4/12/00 according to the logbook could not be located during the audit. It was discovered after discussion with other laboratory personnel that these pipettes had been taken out of service. This was not recorded in the logbook.

- **Pipettes that are taken out of service are marked as "unusable" with tape, so they will not inadvertently be used. The logbook is a record of calibration only. It is the responsibility of the analyst to verify that the pipette has current calibration (as marked on the pipette) prior to use.**

3. The balance ID number, model number and acceptance limits were not consistently filled out in logbook #103 (information was missing on pages 24,29,30 and 31).

- **The balance ID and acceptance criteria will be completed on these pages.**

4. Expired standards (ICP water IDL solution [1237-ICP-035, expired 5/31/00] and ICP water MDL solution [1237-ICP033, expired 5/31/00]) were placed on the laboratory counter with current standards (ICP4 cal blank). Expired standards should be kept separated from the current working standards.

- **We make every attempt to segregate expired standards. However, there are times when we do use expired standards, such as initial instrument method development. During the audit, the laboratory was working on setting up our new ICP, using the expired MDL standards for preliminary work. All standards used for an analytical run are documented in the run and the analyst is responsible for insuring that expired standards are not used.**

5. Expiration date was not included on the containers for three standards: trace ICP soil MDL 922-066, soil MDL spike 1237-ICP-015, and ICP soil mix solution 1237-ICP-023.

- **Expiration dates will be added to all bottles.**

6. Expired standards and reagents were found in the mercury storage area. This area also had dust; this poses a potential contamination problem for metals analysis.

- **We make every attempt to segregate expired standards. However, there are times whey we do use expired standards, such as initial instrument method development. We will work on keeping the hood cleaner. Regular digests are not done in that hood, so there is no problem with contamination other**

**than mercury, and we have not seen any problem with mercury contamination.**

Recommendation:

1. The maintenance logbook (logbook #802) for the ICP was not specific about the type of routine maintenance that occurs. For example, a "PM check" is listed as being performed but no definition of PM check was provided. It is recommended that the logbook be updated to a checklist form similar to the ICPMS maintenance logbooks.

- **This recommendation will be taken into consideration.**

**Extraction/Sample Preparation-organics**

Findings:

1. The SOPs list only descriptions of the methods. EPA method numbers are not specified in the SOP or on the electronic extraction batch sheet. It is recommended that the SOP specify what the EPA methods are.

- **All methods are referenced in section 16 of the SOP.**

2. Uncontrolled Enseco and Quanterra SOPs were found in the balance logbooks (#476 for QA004 and #114 for QA036). The uncontrolled SOPs were marked obsolete during the audit.

- **The practice of putting uncontrolled SOPs at the front of new logbooks was eliminated in January 2000. No new logbooks have uncontrolled SOPs at the beginning. Logbooks that have these SOPs are being marked "Obsolete", with the date marked, until all logbooks are replaced.**

3. The certificates of analysis are not filed consistently. Some certificates are stapled in the extraction logbooks (e.g., #1520) while several certificates for 8270 LCS mix #1 lot LA-866125 were placed in a card board box on the counter top. This problem was an audit finding in the 1999 audit. Certificates of analysis need to be kept filed and organized.

- **Use of the electronic standards logbook will facilitate tracking and filing of certificates of analysis, as each incoming vendor solution is assigned an ID number, which can be transferred to the certificate of analysis. In the meantime, the semi-volatile instrument department is responsible for certificate of analysis. All certificate of analysis have been filed.**

4. The concentration of the source standard (pesticides MS S053100B) is not specified in the standard logbook (#1533) on 5/31/00 (page 4).

- **This standard page will be amended.**

5. The sonicators that are used in the extraction lab do not have a logbook to document tuning, maintenance, and daily checks. There needs to be documentation on a daily basis to show the sonicators are operating properly.

- **A sonicator tuning logbook has been re-implemented. Sonicator tuning is recommended by the manufacturer only when there is a change in the sonicator tip or a new horn is installed. STL Sacramento will implement a policy to tune the sonicators on an annual basis, or if complete disruption of the soil samples is not occurring. All sonicators are visually checked for settings and proper use each time they are used.**

6. The recorded daily temperature (-24 degrees C) for refrigerator/freezer 5-3 exceeded the temperature criteria of –10 to –20 degrees C. This occurred several times beginning in April 2000 and continuing until the time of the audit. The freezer also contained a Dioxin Test Kit that indicated it should be stored at 4-8 degrees C.

- **This freezer only contains expired immunoassay kits, which are no longer used. The freezer is marked for expired standard storage only.**

7. A certificate of analysis for a pesticide standard (lot # PO38900C) in logbook #978 could not be located by Karen Mason. This certificate was later located by Marcia Reed.

- **As noted above and during the audit, the Semi-volatile instrument area, not the sample preparation area, is responsible for certificate of analysis maintenance. This department was represented by Marcia Reed, who efficiently found the certificate of analysis.**

**GC/MS, semivolatiles (8270C)**

Findings:

1. Both analysts were new in the semivolatiles department. They were transferred from the volatiles department approximately two weeks before the audit. These analysts were not aware of the Earth Tech acceptance criteria for the initial calibration, which specifies that the percent RSD for individual compounds cannot be >30%.

- **These requirements will be communicated to the analysts for each project via the new QAS system, currently in effect. All analysts are aware that they are familiar with knowing QAPP requirements prior to analysis of samples. Due to the many different client requirements that we work with, analysts will generally cite our standard procedures during audits.**

Recommendation:

1. No pagination is assigned on the standard preparation sheets in the standard logbook binder for year 2000. It is recommended that this logbook be paginated.

- **The logbook will be paginated, and analysts reminded that pages need to paginated as they are placed in the binder, per our policy.**

**Gas Chromatography, Extractables**

Findings:

2. Two versions of the SW8000 method were found in the SOP binder. Version 4.1 is superseded by version 5.0 and should be removed from the binder.

- **There are two SOPs for Method 8000, CORP-GC-0001, which is in the binder for reference, and the lab version, CORP-GC-0001SAC.**

3. There is no SOP for the 504.1 analysis, but according to lab personnel there is one currently being written.

- **This SOP will be completed by October 6, 2000.**

4. The standard refrigerator contained herbicide standards that were labeled as 8150, but the actual method in use is 8151A. Labeling needs to be accurate.

- **The analyst will be asked to update the labels and the storage box. Since Method 8150 has been replaced entirely with Method 8151A, no confusion would arise from this mislabeling.**

5. The standard for 8015 diesel was not checked as acceptable for use in the standard logbook #1281, page 3. Although this field of information is not required by Earth Tech, if it is provided it should be completed. This is an inconsistency found throughout the laboratory.

- **This information will be tracked only on the Standard QC forms. Logbook pages and labels will be revised accordingly.**

6. The run logbook for herbicides had not been reviewed. This is a problem throughout the laboratory. There needs to be a policy regarding how often logbooks need to be reviewed, and this policy needs to be implemented.

- **The area leaders will be reminded to review all logbooks on a monthly basis.**

Concern:

1. Working standards are stored in vials with no written markings distinguishing them from sample vials. During the course of running a sequence, standards and samples could easily be confused. We recommend these vials be marked.

- **Labels are not applied to the vials to prevent mis-injections. Each analyst has his own practices to prevent misloading. This includes preceding every**

**series of standards with a solvent blank containing no surrogates. Analysts will be advised of the suggestion, and will look for convenient solutions that will not cause mis-injections.**

**GC/MS, volatiles (8260B)**

Findings:

1. Several procedure changes for 8260B (initial calibration standard range, BFB temperature program and quantitation ion changed [from ion 119 to ion 117]) were hand written on yellow post-it notes and placed on several CORP-MS002 SOP pages. The analyst (Yun Pan) has been working in the area for a year. She is familiar with the procedure changes. However, when asked about why these changes were not included in the SOP revision, she replied that she was not sure why the SOP was not changed but that she was going by her peer training. These procedure changes were later verified to be included in the facility specific CORP-MS002-SAC SOP.

- **The notes in the Corporate SOP were to insure all changes requested were incorporated into the SAC version of the SOP. These notes have been removed.**

2. Analysts are not aware of the Earth Tech acceptance criteria for initial calibration (RSD for individual compounds cannot be >30%). The area leader (Wayne H.) claims that he has submitted variance requests for this requirement a year ago to the project manager. Currently they do not monitor this requirement.

- **The variance has been submitted. The non CCC compounds that are exceed 30% will be narrated.**

3. An uncontrolled Enseco SOP was found in the maintenance logbook (for HP4). The uncontrolled SOP was marked obsolete during the audit.

- **The practice of putting uncontrolled SOPs at the front of new logbooks was eliminated in January 2000. No new logbooks have uncontrolled SOPs at the beginning. Logbooks that have these SOPs are being marked "Obsolete", with the date marked, until all logbooks are replaced.**

4. Instructions for standard preparation are not included in the method SOP or in the standard logbook.

- **The standard preparation is discussed in section 10.4.1 and Table 2, 4 and 9.**

5. Corrective action procedures are included in the SOP for IS and BFB outlier, but not for calibration failure.

- **This is discussed in section 10.4.3 and 10.4.3.1.**

**GC, Volatiles**

There were no findings in this section

**GC/MS, Dioxins Preparation - low resolution (8280A) and high resolution (8290)**

Findings:

2. Two standards for high-resolution (#1203-98 and 1203-97) were found by the analyst to have incorrect labels. It appears that they were made at the same time and the labels were switched. This was caught and corrected by Mike Fluornoy.

- **This was corrected during the audit.**

3. The standard refrigerator (R58) located in the low-resolution prep lab had not month or year indicated on the checklist, and it had not been checked since the fifteenth of the month. Refrigerators need to be checked daily and all header information should be completed.

- **Corrective action was taken immediately and personnel were reminded that temperature checks need to be done daily.**

4. Header information (balance ID, model #) was not recorded consistently on each page for logbook #619.

- **Corrective action was taken immediately and personnel were reminded that all information must be recorded in the logbook.**

5. The oven located in the low-resolution prep lab (VWR1305) was monitored inconsistently during the month of June.

- **Corrective action was taken immediately and personnel were reminded that temperature checks need to be done daily.**

**GCMS Dioxins – low resolution (8280A)**

There were no findings in this area.

Concern:

1. The analysis logbook for method 8280A was second reviewed by the same person.

- **Analysis logbooks are always reviewed by another person via a photocopy when the secondary review is done on the data package.**

**GCMS Dioxin – high resolution (8290)**

There were no findings in this area.

**HPLC Extraction/Sample Analysis – 8330 and 8330M (nitroguanidine) [HPLC/UV]; 8321 CWM [HPLC/MS]**

Findings:

1. An uncontrolled Quanterra SOP was found in the balance logbook (#113 for QA002). The uncontrolled SOP was marked obsolete during the audit.

- **The practice of putting uncontrolled SOPs at the front of new logbooks was eliminated in January 2000. No new logbooks have uncontrolled SOPs at the beginning. Logbooks that have these SOPs are being marked "Obsolete", with the date marked, until all logbooks are replaced.**

2. Balance logbook #113 was last reviewed on 10/11/99.

- **The new area leader for the group will make monthly reviews of maintenance, instrument and balance logbooks.**

**GC/MS, semivolatiles (CWM) –extraction and analysis**

There are no findings.

**Quality Assurance Management**

Finding:

1. Many MDL studies for the reviewed methods were overdue. The time for data work-up and approval of the MDL seems overly long.

- **The QA staff is currently short by one person. This person is filling in for another department's leave of absence. After the QA staff is at full force in September, all missing MDLs will be scheduled with the laboratory, and will be able to be reviewed on a more timely basis.**

Finding:

1. The required review of SOPs is overdue in many cases. These reviews are not documented on the SOP or SOP log, and it is therefore difficult to determine when and if they are actually done.

- **Review of SOPs are currently being scheduled. A better system for tracking SOPs that have been reviewed by not revised will be developed.**

**Data Review**

There are still problems with late reports, and slow response to requests for amendments.

- **This being handled as per the meeting between EarthTech and STL on July 7, 2000.**

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July10, 2000

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605-1501

Attn. Eric Redman/Pamela Schemmer

Subject: AUDIT REPORT

Audit Date: June 20 and 21, 2000

Audit Location: STL, West Sacramento, CA

Audit Scope: Laboratory audit with particular emphasis on AFCEE requirements, and compliance with SW846 and other EPA methods protocol

Auditors: Elisabeth Fruth, Janet Tsukamoto, Angela Wranic and Nancy Carpenter (all Earth Tech, Long Beach)

Personnel Contacted: Pamela Schemmer, Nancy Krsulic, Ray Lucero, Cheryl Hytrek, Dennis Gall, Marcia Reed, Karen Mason, Jonathan Reed, David Alltucker, Alan Wong, John Barnett, Wayne Halozan, Yun Pan, Kyle Stephens, Mike Flournoy, Robert Hrabak, Steve Rogers, Kevin Sanchez, Clark Pickell, Mark Frey, Tuan Nguyen, Lesa Waldron, Mike Lucchesi, Christopher Jenkins, Diana Brooks, Bonnie McNeill, Dave Herbert, Doug Winter, Jo Yamazawa, Eric Redman

Audit Summary: A walkthrough audit of all areas of the laboratory was performed. The audit concentrated on areas where the West Sacramento facility has performed or will perform analyses for Edwards AFB projects, as well as sample receiving, and included interviews with laboratory personnel. In addition, the performance evaluation studies for the past two years were reviewed, and the QA program was discussed with QA personnel. A debriefing was held where the audit findings, concerns and recommendations from the laboratory audit were summarized.

## General Findings

1. Supervisor review of maintenance logbooks was not performed on a monthly basis as specified by the laboratory. This problem was consistent throughout the laboratory.
2. The general policy throughout the laboratory is for analysts to prep and report a DCS when a MS/MSD is not prepared. Because of this policy we frequently receive in our final reports LCSD data when it is not required. This is a client specific requirement that has not been communicated to the analysts.
3. The QC acceptance date for the standards is not consistently documented on the standard labels and in the standard logbooks. Although this field of information is not required by Earth Tech, last year's audit response indicated that it would be filled out, and if it is provided it should be filled out.
4. Requests for amended reports are not being processed in a timely manner.
5. Several of the corrective actions proposed in the response to the Earth Tech audit of 7/99 have not been implemented.

The following sections constitute the findings, concerns and recommendations grouped by area or subject.

## Sample Receiving and Control *(Cheryl Hytrek and Ray Lucero)*

The following items and systems were reviewed: sample receipt, log-in, storage, tracking and anomalies; sample labeling; and refrigerator temperature monitoring.

Sample receiving and log-in are generally handled well. Temperature and pH are checked upon receipt. The temperature of the temperature blank (if applicable) and representative samples (averaged) are measured with an IR instrument and recorded on the Lot Receipt Checklist (LRC). The pH of the preserved samples is measured, and any anomalies recorded. If the temperature or pH is not per requirements the PM is informed. Any pH adjustment is made after notifying the client. Discrepancies between the sample containers and the COC are verified and anomaly forms are filled out. Procedures are in place for monitoring holding times, and notifying the appropriate laboratory section of short hold time samples or rush samples. Short hold time samples are noted on the LRC and delivered to the analytical area by sample receiving personnel; this includes VOC samples listed as not being preserved. Sample storage refrigerator temperatures are monitored and recorded daily except Sundays and holidays.

There are no findings for this area.

**General Chemistry** *(Mark Frey, Tuan Nguyen, and Lesa Waldron)*

The following information was reviewed for the wet chemistry methods 9060 and 415.1 (TOC); 351.2 (TKN); 365.3 (Total P); 9012A (CN); 160.1 (TDS); 300.0, 340.2, and 4500-F/C (anions); 310.1 and 2320 (alkalinity); 350.1 (ammonia-N), and 376.2 (sulfide): calibration requirements, frequency and criteria of calibration checks, corrective action procedures, standards traceability, and a review of maintenance logbooks, analysis run logbooks, oven temperature logs, and balance and pipette calibration logbooks, and SOPs.

Comments:

- Walkley-Black (TOC) and 425.1 (MBAS) methods are no longer performed at the laboratory.

- The development of method 314.0 (perchlorate) has been delayed.

Findings:

1. Recent laboratory reports have not included information on laboratory duplicates as required by Earth Tech.
2. Certificate of analyses were not available for all method standards that originated from salts. Air Force work requires certificates of analysis for all methods.
3. The CCV working standard (924-WC-80-6) label for method 300.0 did not contain the concentration units of the analytes. This information was added at the time of the audit.
4. The electrical conductivity meter logbook did not contain the criteria for the daily calibration of the instrument.
5. The alkalinity logbook did not included the criteria for standardization of pH and electrical conductivity.
6. Uncontrolled Enseco and Quanterra (some SOPs were not stamped "uncontrolled") were found in all maintenance logbooks and balance calibration logbooks in this area. The uncontrolled SOPs were marked "obsolete" during the audit.
7. Maintenance logbooks (#256 for Dionex 2000 and #303 for IC DX-100) were last reviewed on 11/30/99, and the balance logbooks had not been reviewed since 11/29/99. It is not clear what the policy is regarding the frequency of logbook review. However, there should be a policy in place that establishes consistency with regard to this issue.
8. The instrument ID is not specified on the cover page for the total cyanide distillation instrument logbook (#1573) and the LACHAT autoanalyzer

logbook (#1525).

9. A 2 mL pipette (WC-144530) did not have a calibration record tag. According to the analyst, this pipette was taken out of service. However, this was not documented in the pipette calibration logbook, and the pipette was still left in the laboratory with another pipette that is currently being used.
10. The SOP for alkalinity (SAC-WC-0028) references method 310.1, not 2320 which is specified by Earth Tech. The implementation date is missing on the cover page for this SOP.
11. The SOP for pH references method 150.1 for water samples.
12. The SOP for fluoride references method 340.2, not SM4500F.
13. The implementation date is missing on the cover page for the SOP for ammonia-N by method 350.1 (SAC-WC-0105) and sulfide by method 376.2 (SAC-WC-0014).

Recommendations:

1. A number of certificates of analysis have not been properly filed in the notebook. Instead they have been stored in the sleeve of the notebook. These certificates should be filed.
2. Out of control incidents were not clearly documented on the oven temperature chart. The temperature, 80 degrees C, of the drying oven #4 was out of the acceptable range (73-77 degrees C) on 6/15/00. No documentation or explanation was recorded on the oven temperature chart. The analyst noted "re-adjust" in the comment section. It is recommended that the corrective action taken and return to control be documented.

**Metals Preparation and Analysis and STLC/TCLP extractions**
(*David Alltucker, John Barnett, Allan Wong*)

The following information was reviewed for metals preparation by methods 3005, 3015 and 3050, and leachate preparation: SOPs, standards traceability, maintenance logbooks, and prep logs. The following information was reviewed for mercury (7470A, 7471A, 245.1) and Inductively Coupled Plasma (ICP) metals (6010B, 200.7) and ICP-MS (6020): calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance logbooks, SOPs, retention time and MDL studies.

Comments:

- ICPMS is a good method to use when low detection limits are required. The limitation of the method is that it is more sensitive to sample matrix interference than an ICP.

- Organic lead analysis is not performed by the laboratory. Graphite furnace atomic absorption methodologies were not audited.

Findings:

1. Uncontrolled Enseco and Quanterra SOPs were found in the metals reagent tracking logbook (#603) and balance calibration logbooks (#103 and #1245). The uncontrolled SOPs were marked "obsolete" during the audit.
2. Pipettes (MP010 and MP011) which were last calibrated on 4/12/00 according to the logbook could not be located during the audit. It was discovered after discussion with other laboratory personnel that these pipettes had been taken out of service. This was not recorded in the logbook.
3. The balance ID number, model number and acceptance limits were not consistently filled out in logbook #103 (information was missing on pages 24,29,30 and 31).
4. Expired standards (ICP water IDL solution [1237-ICP-035, expired 5/31/00] and ICP water MDL solution [1237-ICP033, expired 5/31/00]) were placed on the laboratory counter with current standards (ICP4 cal blank). Expired standards should be kept separated from the current working standards.
5. Expiration date was not included on the containers for three standards: trace ICP soil MDL 922-066, soil MDL spike 1237-ICP-015, and ICP soil mix solution 1237-ICP-023.
6. Expired standards and reagents were found in the mercury storage area. This area also had dust; this poses a potential contamination problem for metals analysis.

Recommendation:

1. The maintenance logbook (logbook #802) for the ICP was not specific about the type of routine maintenance that occurs. For example, a "PM check" is listed as being performed but no definition of PM check was provided. It is recommended that the logbook be updated to a checklist form similar to the ICPMS maintenance logbooks.

**Extraction/Sample Preparation-organics** *(Karen Mason, Jonathan Reed)*

The following items and systems were reviewed for extraction methods 3510C, 3520C, 3550C, 3540C as well as extraction for method 1664: SOPs, extraction bench worksheets, standard logbooks, certificate of analysis for standards, spiking procedures, analytical balance calibration logbooks, condition of laboratory refrigerators including thermometers and temperature logbooks.

Findings:

1. The SOPs list only descriptions of the methods. EPA method numbers are not specified in the SOP or on the electronic extraction batch sheet. It is recommended that the SOP specify what the EPA methods are.
2. Uncontrolled Enseco and Quanterra SOPs were found in the balance logbooks (#476 for QA004 and #114 for QA036). The uncontrolled SOPs were marked obsolete during the audit.
3. The certificates of analysis are not filed consistently. Some certificates are stapled in the extraction logbooks (e.g., #1520) while several certificates for 8270 LCS mix #1 lot LA-866125 were placed in a card board box on the counter top. This problem was an audit finding in the 1999 audit. Certificates of analysis need to be kept filed and organized.
4. The concentration of the source standard (pesticides MS S053100B) is not specified in the standard logbook (#1533) on 5/31/00 (page 4).
5. The sonicators that are used in the extraction lab do not have a logbook to document tuning, maintenance, and daily checks. There needs to be documentation on a daily basis to show the sonicators are operating properly.
6. The recorded daily temperature (-24 degrees C) for refrigerator/freezer 5-3 exceeded the temperature criteria of –10 to –20 degrees C. This occurred several times beginning in April 2000 and continuing until the time of the audit. The freezer also contained a Dioxin Test Kit that indicated it should be stored at 4-8 degrees C.
7. A certificate of analysis for a pesticide standard (lot # PO38900C) in logbook #978 could not be located by Karen Mason. This certificate was later located by Marcia Reed.

**GC/MS, semivolatiles (8270C)** *(Mike Lucchesi, Christopher Jenkins)*

8270C is currently performed and the following information was reviewed: calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, standard logbooks, maintenance logbooks, analysis run logbooks, SOPs, and project QC requirements.

Comment:

- Expired standards are tagged with pink labels for use for internal QC only, and are kept separately in the standard refrigerator. This is a good system, and is recommended for other sections of the laboratory as well.

Findings:

1. Both analysts were new in the semivolatiles department. They were transferred from the volatiles department approximately two weeks before the audit. These analysts were not aware of the Earth Tech acceptance criteria for the initial calibration, which specifies that the percent RSD for individual compounds cannot be >30%.

Recommendation:

1. No pagination is assigned on the standard preparation sheets in the standard logbook binder for year 2000. It is recommended that this logbook be paginated.

**Gas Chromatography, Extractables** *(Marcia Reed)*

The following information was reviewed for organochlorine pesticides (8081A), PCBs (8082), 8151A, 504.1 and TEPH (8015B): calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance and analysis logbooks, SOPs, retention time studies, and condition of the laboratory refrigerators including thermometers and temperature logbooks.

Comment:

- The new computer program for tracking standards appears to be an improvement when fully developed. This program will improve traceability and help eliminate cumbersome logbooks.

Findings:

1. Two versions of the SW8000 method were found in the SOP binder. Version 4.1 is superseded by version 5.0 and should be removed from the binder.
2. There is no SOP for the 504.1 analysis, but according to lab personnel there is one currently being written.
3. The standard refrigerator contained herbicide standards that were labeled as 8150, but the actual method in use is 8151A. Labeling needs to be accurate.
4. The standard for 8015 diesel was not checked as acceptable for use in the standard logbook #1281, page 3. Although this field of information is not required by Earth Tech, if it is provided it should be completed. This is an inconsistency found throughout the laboratory.

5. The run logbook for herbicides had not been reviewed. This is a problem throughout the laboratory. There needs to be a policy regarding how often logbooks need to be reviewed, and this policy needs to be implemented.

Concern:

1. Working standards are stored in vials with no written markings distinguishing them from sample vials. During the course of running a sequence, standards and samples could easily be confused. We recommend these vials be marked.

**GC/MS, volatiles (8260B)** *(Wayne Halozan, Yun Pan)*

8260B is currently performed and the following information was reviewed: calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance logbooks, SOPs, and project QC requirements. Method 5035 is available.

Findings:

1. Several procedure changes for 8260B (initial calibration standard range, BFB temperature program and quantitation ion changed [from ion 119 to ion 117]) were hand written on yellow post-it notes and placed on several CORP-MS002 SOP pages. The analyst (Yun Pan) has been working in the area for a year. She is familiar with the procedure changes. However, when asked about why these changes were not included in the SOP revision, she replied that she was not sure why the SOP was not changed but that she was going by her peer training. These procedure changes were later verified to be included in the facility specific CORP-MS002-SAC SOP.
2. Analysts are not aware of the Earth Tech acceptance criteria for initial calibration (RSD for individual compounds cannot be >30%). The area leader (Wayne H.) claims that he has submitted variance requests for this requirement a year ago to the project manager. Currently they do not monitor this requirement.
3. An uncontrolled Enseco SOP was found in the maintenance logbook (for HP4). The uncontrolled SOP was marked obsolete during the audit.
4. Instructions for standard preparation are not included in the method SOP or in the standard logbook.
5. Corrective action procedures are included in the SOP for IS and BFB outlier, but not for calibration failure.

**GC, Volatiles** (*Kyle Stephens*)

The following information was reviewed for volatile compounds (8021B) and purgeables (gasoline by 8015B): calibration requirements, frequency and criteria of calibration checks, corrective action procedures, and standards traceability, maintenance and analysis logbooks, SOPs, and the condition of laboratory refrigerators including thermometers and temperature logbooks.

There were no findings in this section.

Comment:

- This section of the laboratory was found to be very organized and well documented. All the standard and analysis logbooks were up to date.

**GC/MS, Dioxins Preparation - low resolution (8280A) and high resolution (8290)** *(Mike Flournoy)*

Both low and high resolution dioxins preparation area were visited. The following items and systems were reviewed: SOPs, preparation procedures, standard traceability and labeling, standard logbooks, refrigerator and oven logs.

Findings:

1. Two standards for high-resolution (#1203-98 and 1203-97) were found by the analyst to have incorrect labels. It appears that they were made at the same time and the labels were switched. This was caught and corrected by Mike Fluornoy.
2. The standard refrigerator (R58) located in the low-resolution prep lab had not month or year indicated on the checklist, and it had not been checked since the fifteenth of the month. Refrigerators need to be checked daily and all header information should be completed.
3. Header information (balance ID, model #) was not recorded consistently on each page for logbook #619.
4. The oven located in the low-resolution prep lab (VWR1305) was monitored inconsistently during the month of June.

**GCMS Dioxins – low resolution (8280A)** *(Steve Rogers, Kevin Sanchez)*

The following items were reviewed: SOPs, maintenance logbooks, standard and analysis logbooks, and standard traceability.

There were no findings in this area.

Concern:

1. The analysis logbook for method 8280A was second reviewed by the same person.

**GCMS Dioxin – high resolution (8290)** *(Clark Pickrell)*

The following items were reviewed: SOPs, maintenance logbooks, standard and analysis logbooks, and standard traceability.

There were no findings in this area.

**HPLC Extraction/Sample Analysis – 8330 and 8330M (nitroguanidine) [HPLC/UV]; 8321 CWM [HPLC/MS]** *(Dennis Gall, Robert Hrabak, Steve Rogers)*

The following items and systems were reviewed: SOPs, project QC requirements, extraction bench worksheets; and spiking procedures; calibration requirements; frequency and criteria of calibration checks; corrective action procedures; standards traceability; maintenance and analysis logbooks; and the condition of laboratory refrigerators including thermometers and temperature logbooks.

Comments:

- Extraction for nitrocellulose is performed here, but analysis is performed by colorimetry in the general chemistry area.
- 8321 analytes include CWM degradates, picric acid and PETN.

Findings:

1. An uncontrolled Quanterra SOP was found in the balance logbook (#113 for QA002). The uncontrolled SOP was marked obsolete during the audit.

2. Balance logbook #113 was last reviewed on 10/11/99.

**GC/MS, semivolatiles (CWM) –extraction and analysis** *(Marcia Reed, Jonathan Reed)*

The following items and systems were reviewed: standard preparation, traceability, and labeling; and calibration criteria and frequency.

Comment:

- The extraction for modified 8270 does not include pH adjustment since it is not pertinent to the target analytes. Surrogates and internal standards added are the same as for 8270C, but QC spiking compounds are per the target compound list.
- Only 3 internal standards and 2 surrogates (BN) are monitored. Tuning with DFPTT is not performed because this compound is not representative of the target compounds.

There are no findings.

**Quality Assurance Management**

*Quality Assurance Program.* STL Sacramento is still working under the Quanterra Corporate QA program. Work is underway to combine STL and Quanterra QA program policies. STL has a quality management plan.

*Method Detection Limit Studies.* MDL studies are performed annually for each method.

Finding:

1. Many MDL studies for the reviewed methods were overdue. The time for data work-up and approval of the MDL seems overly long.

*Standard Operating Procedures.* Standard Operating Procedures (SOP) are available for most of the methods reviewed. Quanterra Corporate SOPs and facility specific SOPs, which are being converted to STL SOPs, are available. During the audit process, it was indicated that SOPs were routinely reviewed by the QA department. If found to be acceptable, the SOP was not updated; however the SOPs in the laboratory areas do not list this review. QA review dates need to be added to the current listing of SOPs. It is also recommended that evidence of this review be added to the SOP within the laboratory areas.

Finding:

1. The required review of SOPs is overdue in many cases. These reviews are not documented on the SOP or SOP log, and it is therefore difficult to determine when and if they are actually done.

*Document Control*. SOPs are issued as controlled copies. Revisions are issued to the original holders of the documents. Logs are kept electronically.

*Performance Evaluation Samples*. PE sample results from the WP and WS studies for the past two years were reviewed. As of January 1999, EPA no longer provides these samples. ERA (Environmental Resource Associates) is the new provider. The results are very good.

*Audits*. Corporate audits were performed annually by Quanterra. The last one was done in November 1999. STL does not perform corporate audits, but planning for the future includes using QA personnel from other laboratories for annual audits. Audits by facility QA are performed periodically, as are data audits. Implementation of corrective action is verified during audits, and a database file has been set up to track previous findings so that an evaluation can be made regarding frequency of findings in each area, and repetition of similar findings.

*Nonconformance/Corrective Action*. Electronic anomaly reports are generated, and are automatically included as part of the project file. Resolution of non-routine anomalies must be accepted by QA before closure. Anomalies are tracked, and a database file has been set up to track previous anomalies so that an evaluation can be made regarding frequency of anomalies in each area, and repetition of similar anomalies.

*Thermometer, Balance and Weight Set Calibrations*. Thermometers used in the lab are calibrated annually against an NIST traceable thermometer. This calibration is performed in-house. Calibration of balances is performed by an outside vendor annually in the laboratory. Weight sets are sent to an outside vendor for calibration every three years.

**Personnel Training**

A training program is in place, documenting proficiency for analytical methods, and resumes listing education and previous experience are available for all technical personnel. Files were well organized in specific sections. Improvements are continually made to facilitate tracking and electronic entries.

**Data Review**

Data review is performed by the analyst (Level 1), by supervisor (Level 2) and by the project manager. Checklists are used.

There are still problems with late reports, and slow response to requests for amendments.

**General Comments**

The staff, including analysts and supervisors, are knowledgeable and proficient in performing the analyses.

A QAS system is in the process of development. This system will computerize client specific requirements. Analysts will be able to access this information directly. This could potentially reduce miscommunication between project managers and analysts, and minimize requests for amendments due to deviations from client requirements. We recommend that this system be brought on-line as soon as possible.

I want to express my appreciation to all laboratory personnel for their interest, courteous assistance and time spent during this audit.

Please respond to the findings and concerns enumerated in the audit report by July 24, 2000.

Very truly yours,

Earth Tech

Elisabeth Fruth

Elisabeth Fruth
QA Manager

cc. Bonnie McNeill

TRUESDAIL LABORATORIES

100 West Broadway, Suite 5000, Long Beach, California [illegible]

9 August 1999

Charles Hamilton
Truesdail Laboratories, Inc.
14201 Franklin Avenue
Tustin, CA 92780-7008

Subject: AUDIT REPORT

Audit Date: August 3, 1999

Audit Scope: Laboratory audit with particular emphasis on AFCEE requirements, compliance with Method 8315M, and other EPA protocol

Auditor: Jim Cirillo (Earth Tech, Long Beach)

Telephone
562.951.2000
Facsimile
562.951.2100

Dear Mr. Hamilton,

Earth Tech conducted a laboratory audit of Truesdail Laboratories on August 3, 1999. The audit focused primarily on the 8315M method for the analysis of hydrazine, monomethylhydrazine (MMH), and unsymmetrical dimethylhydrazine (UDMH). Method 8315M is a modified version of EPA Method 8315A, *Determination of Carbonyl Compounds by High Performance Liquid Chromatography (HPLC)*. The sections audited and discussed below included: Sample Control; Semivolatiles Extractions and Analysis (i.e., Method 8315M); Type II water; SOPs; controlling of documents; PE sample results; personnel training; audits; nonconformance documentation; QC limits and control charts; MDLs; and thermometer and balance calibration records.

**General Comments**

1. The hydrazine method (8315M) used by Truesdail Laboratories is a significant modification of EPA Method 8315A. Included in the OU4, Addendum II QAPP, is a copy of the 8315M SOP. However, the 8315M SOP does not include any discussion about modifications, substitutions or omissions as compared to Method 8315A. It is strongly recommended that the 8315M SOP be updated as soon as possible to include any and all information regarding variances from the EPA method in the hydrazine extraction and analysis procedure.

2. Page 5-32 of the OU4, Addendum II QAPP summarizes the procedure for the analysis of hydrazine in water and soil matrices. The discussion states, in part, that "All appropriate QA/QC procedures specified in this QAPP apply to this laboratory", i.e., Truesdail

Laboratories, when performing the 8315M method. Results of this audit and the validation of data from two Truesdail reports for hydrazine strongly indicate that the QA/QC parameters outlined in the QAPP are either not being strictly followed and/or are not formatted into laboratory documentation. (See the enclosed Earth Tech letter dated 7/29/99 listing comments regarding projects 416036 and 602129.) Please review the QA/QC guidelines in the QAPP and comment on this item. Moreover, please indicate the systems in place ensuring that Earth Tech samples are being analyzed in accordance with the OU4, Addendum II QAPP. (For reference, please see the enclosed pertinent sections from Table 5.7-2 and Table 5.9-1.)

3. Lab generated control charts show that spike recoveries of hydrazine, monomethylhydrazine, and unsymmetrical dimethylhydrazine in soil matrices have consistently been poor. In fact, recoveries of zero percent are not uncommon. Moreover, LCS spike recoveries for the same three compounds in QC-grade sand have generally been as poor as the matrix spike recoveries. The accumulated data therefore indicate that the stability of hydrazines in soil and other solid matrices is questionable and, at present, all hydrazine soil results should be considered estimated. (No more than 10 data points were used in constructing the control charts for hydrazine soil matrices. Therefore, to achieve greater statistical accuracy, it is strongly suggested that when more hydrazine soil data points become available, they be added to the accumulated data.)

**Specific Comments**

***Section:*** ***Sample Control***
***Rupal Patel, Joe Bramblett, Charles Hamilton***

**General Information**

1. The following items and systems were reviewed: Sample receipt, log-in, storage, tracking and anomalies; sample labeling; refrigerator temperature monitoring; Type II water generation; and bottle prep.

2. There was no space on the August temperature log for correction factors. According to Mr. Hamilton, thermometers discovered to be functioning improperly are removed from operation. Does the laboratory document the discovery and removal of malfunctioning thermometers?

**Findings**

1. The lone refrigerator in Sample Control was not operating properly. According to Mr. Hamilton it was discovered the day before the audit that the refrigerator was unable to cool below 10 degrees celsius. (The acceptable temperature range is 4 ± 2 degrees celsius.)

However, Mr. Hamilton stated that the refrigerator was used only to store samples temporarily, particularly since samples were stored in refrigerators in the various departments throughout the lab. Moreover, there was a comment on the August temperature log attached to the door stating a maintenance company had been called to service the refrigerator. Please submit documentation showing that this refrigerator has been serviced and is now operating properly.

**Recommendations**

1. According to Mr. Hamilton, trip blank labels only include the term *Travel Blank*; no initials, prep date or prep batch number is written on the labels. Since at times it proves important to determine the source of contamination detected in trip blanks, a better tracking system is recommended.

***Section:*** ***Hydrazine Sample Prep – (8315M)***
***Der-Haw Wang, Arvinder Sidhu, Yvonne Nguyen, Joe Bramblett***

**General Information**

1. The following items and systems were reviewed: Extraction equipment; and standard preparation, traceability and labeling. (Note that Method 8315A describes an extraction procedure that is unique to the method and therefore no 3000 series extraction methods are referenced.)

**Findings**

1. Extraction SOPs (e.g., 8270, 8080) should reference the pertinent extraction method(s). Several of the extraction SOPs - other than 8315M - available in the extractions lab reference only the analytical method and not the extraction method(s).

2. The 8315M SOP did not include a cover page containing the title and approved signatures. Moreover, only a few of the SOPs in the SOP binder included a cover page listing the title and approved signatures.

3. Certificates of Analysis (CoA) must be available and filed correctly. The CoA's for the Aldrich hydrazine neat standards were not available during the audit. However, Mr. Bramblett forwarded CoA's for hydrazine and UDMH to Earth Tech on 8/6/99. Furthermore, the CoA for MMH had not been received at the time this letter was finalized.

4. Standard labels should at least include references to the standard logbook, material identification, concentration, preparation and expiration date, and the preparers name or initials. The label on standard E09042806 did not identify the analyst who prepared the

standard.

5. Soil results must be reported corrected for moisture content. There was uncertainty amongst the laboratory staff regarding whether or not moisture correction was being performed for 8315M results. Please determine if any hydrazine sample results have been reported to Earth Tech without moisture correction and, if necessary, indicate what type of corrective action will occur. Moreover, please include in the response to this letter evidence that moisture correction will be applied to all future hydrazine soil samples submitted to Truesdail Laboratories.

7. Page 4 of the extraction logbook with the starting date 5/5/99 did not have entered the responsible chemists' initials.

8. The laboratory staff was unsure about the number of calibration check points that were necessary to verify the calibration of balance # 28961 in the extractions lab. Moreover, it was unclear what tolerance limits were in place. Please clarify this finding and indicate what reference the lab uses to justify its balance calibration check policy and balance acceptance limits.

**Recommendations**

1. It would be helpful if logbooks were distinguished by a unique and logical alphanumeric identification. Logbooks are currently identified by the starting and ending usage dates. Since the ending date is not determined until the last page is used, the idenitifier is ambiguous.

***Section:*** ***HPLC (8315M)***
***Der-Haw Wang, Arvinder Sidhu, Yvonne Nguyen, Joe Bramblett***

**General Information**

1. The following items and systems were reviewed: Method 8315M; SOPs; sample storage; and maintenance logbooks and instrument runlogs.

**Findings**

1. It is imperative that the laboratory staff refer to the most current SOP. The version of the 8315M SOP available in the extractions lab was dated 9/94 while the 8315M SOP version contained in the June 1999 addendum to the OU-4 QAPP is dated 12/97.

2. The 8315M SOP should discuss how field samples are to be preserved. The 8315M SOP dated 12/97 does not discuss the preservation of samples.

3. All logbooks must be paginated. Both the maintenance logbook and the instrument runlog for the Shimadzu HPLC are not paginated.

4. All logbooks should consistently document supervisory review; this was not being done in the standard logbook, maintenance logbook and the instrument runlog for the Shimadzu HPLC. Furthermore, the maintenance logbook must indicate when return to control has been achieved. This, again, was not being done for the Shimadzu HPLC.

5. The maintenance logbook for the Shimadzu HPLC should be contained in bound form, not in a spiral notebook. Preferably, the pages should be preprinted with columns for common entries (i.e., date, maintenance performed, comments, return to control, initials). Moreover, initials were entered sporadically.

6. The instrument runlog has a column for time of analysis. However, in the Shimadzu HPLC runlog with the start date 7/24/98, analysis times were not entered for any dates of analysis observed. Analysis time is a crucial QA element and should be entered for either all listed analyses or at least for the first and last analysis of each batch.

7. The dates of analysis should be accurately reflected in the instrument runlog. Entries on 6/24/99 in the Shimadzu HPLC runlog with the start date 7/24/98 suggested that all analyses occurred on 6/24/99. However, there were more than 20 sample runs, each with a run time of approximately 45 minutes, and therefore it is possible that the last few analyses occurred druing the early morning of 6/25/99.

8. Section 8.0 in EPA Method 8315A references Section 7.0 of Method 8000B when discussing quality control procedures, which includes the determination of retention time (RT) windows. The laboratory staff did not appear to understand the process by which RT windows were determined according to Method 8000B. Moreover, in response to a question regarding RT windows, Mr. Wang mentioned that RT windows of "approximately" 0.5 minutes to 1.0 minute were being used. Lastly, long-term RT deviations were not being monitored to prevent unacceptable drift. Please comment on this item and please describe the lab's procedure for determining RT windows and how the lab goes about detecting unacceptable RT drift.

9. Section 7.4.1.4 of EPA Method 8000B, which is referenced in EPA Method 8315A, requires that "at least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project." It is probable that the least concentrated ICAL 8315M standard is not being prepared and analyzed at or below the reporting limits for the three analytes of interest. Please comment on this item.

10. There was confusion among the laboratory staff regarding what the hydrazine reporting limits (RLs) should be. Please indicate how the hydrazine RLs are derived and confirm that all pertinent laboratory staff understand what the RLs are for the Earth Tech project.

**Recommendations**

1. Even though one can determine ICAL standard concentrations by correlating the standard logbook with the instrument runlog, it would prove useful to an outside reviewer if the concentrations were included in the instrument runlog.

2. The instrument runlog had a cluttered appearance. It is recommended that no more than one distinct batch be recorded on each page and that comments and other information be entered more legibly.

***Section:*** ***Quality Assurance/Quality Control***
***Charles Hamilton, Joe Bramblett***

**General Information**

1. The following items and systems were reviewed: Data review/data management; control charts/limits; personnel training; MDL studies (8315M only); document control; Performance Evaluation studies; calibration records for balances, weights and thermometers; audits; and nonconformance/corrective action.

**Findings**

1. Control charts and statistically derived limits were unavailable during the audit. However, faxed copies of the pertinent documents were received on 8/6/99.

I want to express my appreciation to all laboratory personnel for their interest, courteous assistance and time spent during this audit, particularly on such short notice.

Please respond to the findings and concerns enumerated in the audit report by August 30, 1999.

Sincerely,

James Cirillo
Staff Chemist II

CC: Elisabeth Fruth

# ENCLOSURES

27 July 1999

Xuan H. Dang
Truesdail Laboratories, Inc.
14201 Franklin Avenue
Tustin, CA 92780

Project Identifier: Edwards AFB – OU4
Truesdail Lab Reports: 416036, 602129

Dear Xuan,

We have reviewed the above-mentioned laboratory reports for the Edwards AFB OU4 project and pertinent comments are listed below. (The guidance document is "Air Force Research Laboratory, Operable Unit No. 4, Final, Addendum II to Final Field Sampling Plan".) Please provide corrections and clarifications by August 10, 1999.

**General Comments**

1. According to Joe Bramblett, hydrazine samples should be collected in 500 ml amber glass bottles and preserved with either HCl or $H_2SO_4$ to a pH < 2. However, the Truesdail SOP entitled *The Determination of Hydrazine, Monomethylhydrazine, and Unsymmetrical Dimethylhydrazine in Aqueous Solutions by HPLC* does not address sample preservation. This information must be included in the SOP. Furthermore, please indicate the reference used which advocates the preservation of samples to a pH < 2. Lastly, please indicate when the SOP will be updated to include the necessary information regarding sample preservation.

**Specific Comments**

**416036**

1. The date received on the Report listed should be 6/17/99, not 6/18/99. Please correct this discrepancy and submit all pertinent documents.

c:\..\Truesdail_rpt

2. The sample result units listed in the Results Summary should be ug/L, not ug/kg. Please correct this discrepancy and submit all pertinent documents.

3. The Sample Receiving Checklist was not filled out completely. Of particular concern is the absence of pH measurement for any of the four samples. Please submit a record of the pH values measured upon receipt for the samples in question.

4. Placed in the report was an invoice with a Bill To and Ship To address Quality Control Tests, 14201 Franklin Ave., Tustin, CA, 92780. Please explain why this document was included in the report.

5. The Extraction Chronicle for 602084-LCS was not copied correctly. Please submit a legible copy of this document.

6. Why are the LCS/LCSD IDs labeled with 602084 and the method blank ID is labeled with 416036?

7. The ICAL/CCV summary page did not include the %RSD for MMH, UDMH and hydrazine. Also, %RSD results for the ICV were not listed. Finally, the listed CCV acceptance limits for MMH, UDMH and hydrazine were broader than that listed in the OU4 QAPP (i.e., ± 20%). How do the analysts determine if the ICAL, ICV and CCV meet the acceptance criteria outlined in the QAPP?

8. The LCS/LCSD summary page did not list any acceptance limits for %D. Moreover, the percent recovery limit listed on the QA/QC Report should be ±25%, not ±30%. How do the analysts determine if the LCS/LCSD meet the acceptance criterion outlined in the OU4 QAPP?

**602129**

1. The recipient at Truesdail Laboratories lined out entries on the CoC but did not initial the error. Please remind laboratory staff the importance of initializing all erroneous document entries.

2. A copy of the Sample Receiving Checklist was not included with the data package. Please submit this document.

3. Placed in the report was an invoice with a Bill To and Ship To address Quality Control Tests, 14201 Franklin Ave., Tustin, CA, 92780. Please explain why

this document was included in the report.

4. A copy of the instrument runlog was not included with the data package. Please submit this document.

5. The raw data (i.e., the Custom Reports and chromatograms) were not ordered correctly. Please ensure that future reports are properly organized.

6. The Custom Report for Filename L990220 listed 313-MW03-WG2 as the client sample ID. This sample ID was not part of the sample delivery group contained in report 602129. Please correct this discrepancy and submit all pertinent documents.

7. MMH in the ICAL standard 602129-STD1 was not integrated correctly. However, instead of reintegrating the peak, the analyst summed the area counts listed on the Custom Report for the two split peaks. Was the area for MMH properly quantitated? Furthermore, please indicate what the Truesdail Laboratories policy is regarding peak reintegration.

8. The ICAL/CCV summary page did not include the %RSD for MMH, UDMH and hydrazine. Also, %RSD results for the ICV were not listed. Finally, the listed CCV acceptance limits for MMH, UDMH and hydrazine were broader than that listed in the OU4 QAPP (i.e., ± 20%). How do the analysts determine if the ICAL, ICV and CCV met the acceptance criteria outlined in the QAPP?

9. According to the OU4 QAPP, the LCS/LCSD/MS/MSD percent recovery acceptance limit is ±25 %. However, the UDMH 602129-1 MS/MSD percent recoveries were below the LCL and this anomaly was not indicated either by lab qualifiers or by a discussion in a case narrative. Please comment on this item.

Sincerely,
Earth Tech

James Cirillo
Staff Chemist II
CC: Elisabeth Fruth

## TABLE 5.7-2 SUMMARY OF CALIBRATION PROCEDURES
(Page 6 of 13)

| Method | Parameter | Calibration | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| 8290[7] (GC/MS) | Dioxins/Furans | Tune using PFK | Once per 12 hours, prior to sample analysis | Resolving power ≥10,000 at m/z-304.9824 & m/z 380.9760 ± 5 ppm of expected mass | 1) Retune instrument<br>2) Reanalyze PFK |
| | | Window defining mix (WDM) and Column Performance Check Solution (CPSM) | Prior to CCAL, once per 12 hours prior to calibration | Used to set retention times; CPSM must have ≤ 25% valley resolution for 2,3,7,8-TCDD | 1) Readjust windows<br>2) Evaluate system<br>3) Perform troubleshooting<br>4) Reanalyze WDM/CPSM |
| | | Multipoint calibration (5 points, ICAL) | Initially and as required | Int. Std. = %RSD ≤ 30%<br>Natives = %RSD ≤20%<br>Retention time must be within -1 to +3 seconds of labeled IS or 0.005 RRT units. Ion ratios within Table 7.7-6 limits, and S/N ≥2.5 | 1) Evaluate system<br>2) Recalibrate |
| | | Daily continuing calibration standard (CCAL) | Once per 12 hours, prior to sample analysis | % D of IS ≤ 30% from avg. RRF (ICAL). % D of natives ≤ 20% from avg RRF (ICAL). RT must be within -1 to +3 seconds of labeled IS or 0.005 RRT units. Ion ratios are within Table 7.7-6 limits. | 1) Evaluate system<br>2) Reanalyze CCAL<br>3) Recalibrate (ICAL) as necessary |
| Truesdail Modified SW8315[12] | Hydrazines | Multipoint calibration (minimum five points) | Initially and as required. Use RF with area closest to sample area. | r ≥0.995 or average %RSD for RF ≤ 20% | Recalibrate |
| | | Initial calibration verification (ICV) | Immediately after initial calibration | ± 20% | Repeat calibration |
| | | Continuing calibration verification (CCV) | At end of run (maximum 20 samples) | ± 20% | 1) Reanalyze standard<br>2) Recalibrate<br>3) Reanalyze affected samples |

## TABLE 5.9-1. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES

(Page 18 of 33)

| Method | Parameter | QC Performed | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| 8290[3] (GC/HRMS) (Continued) | Dioxins/Furans | Method Blank | 1 per analytical batch, not to exceed 20 samples, per matrix. | Ratio of a given unlabeled PCDD/PCDF isomer ≤ 5% of the appropriate internal standard. | 1) Reanalyze method blank<br>2) If noncompliant and analyte concentration in sample < RL or > 10x blank concentration, report results<br>3) If noncompliant and analyte concentration in sample > RL and < 10x blank concentration, reextract and reanalyze affected samples |
| | | LCS (include natives) | At a frequency of 5% (1 per 20 samples analyzed) | Refer to Table 7.12-1 | 1) Review internal standards, as above<br>2) Evaluate data for useability<br>3) If sample results are ND and RLs are met, no action required<br>4) If samples have positives, reextract and reanlyze samples for analytes outside the acceptance criteria |
| | | MS | 1 per program (As per client request) | Refer to Table 7.12-1 | 1) Review data for useability<br>2) Narrate outliers |
| | | MSD | 1 per program (As per client request) | Refer to Table 7.12-1 | 1) Review data for useability<br>2) Narrate outliers |
| Truesdail Modified SW8315 | Hydrazines | Field | | | |
| | | Equipment Blank | 1 per day of sampling (for reusable equipment) | < RL[b] for all analytes | No laboratory corrective action |
| | | Duplicate/Replicate field samples | 10% | RPD ≤ 45% (W&S) | |

## TABLE 5.9-1. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES

(Page 19 of 33)

| Method | Parameter | QC Performed | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| | | Laboratory | | | |
| Truesdail Modified SW8315 (Continued) | | Method blank | 1 per batch of 20 samples or less | < RL[a] for all analytes | 1) If concentration in associated sample is < RL or > 10X the contamination, note the blank contamination in the case narrative and report sample result. Otherwise:<br>2) Reanalyze blank once<br>3) Determine source of contamination (analyze a system blank)<br>3a) If source is instrument, perform maintainance, reanalyze blank and samples<br>3b) If source is preparation, reprep and reanalyze method blank and all samples (other than those in 1) associated with the contaminated blank. |
| | Hydrazines | Laboratory<br>Method Blank (Continued) | | | |
| | | Matrix Spike/Matrix Spike Duplicate (MS/MSD) or MS and Laboratory Duplicate Sample (must use EAFB sample) | 5% (one per 20 or fewer samples) | See Table 5.12-1 for accuracy (recovery); Precision (RPD) criteria: 20% | 1) Check calculations<br>2) Evaluate the System. (instrument)<br>3) Check LCS: If recoveries are within QC limits, flag data and note in the case narrative as attributable to matrix effects. If LCS recoveries are not within QC limits, see below. |

## TABLE 5.9-1. SUMMARY OF INTERNAL QUALITY CONTROL PROCEDURES



| Method | Parameter | QC Performed | Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|---|---|
| Truesdail Modified SW8315 (Continued) | | Laboratory Control Sample (LCS) | 1 per preparation batch (maximum batch size of 20 samples) | See Table 5.12-1 for accuracy (recovery) criteria | 1) Evaluate the system (instrument). See [b]<br>2) Recheck calculations<br>3) If LCS is high and associated sample results ND, note in case (Continued) |
| SW8330 (HPLC/UV) | | Field<br>Equipment Blank | Once per day of sampling (for reusable equipment) | < RL[a] for all analytes | No laboratory corrective action |
| | | Unspiked Duplicate (aqueous) or Replicate (soil) Samples | 10% (one per 10 samples) | RPD ≤ 45% | No laboratory corrective action |
| | Explosives: Nitroaromatics and Nitroamines | Laboratory<br>Method blank | 1 per batch of 20 samples or less | < RL[a] for all analytes | 1) If concentration in associated sample is < RL or > 10X the contamination, note the blank contamination in the case narrative and report sample result. Otherwise:<br>2) Reanalyze blank once<br>3) Determine source of contamination (analyze a system blank)<br>3a) If source is instrument, perform maintainance, reanalyze blank and samples<br>3b) If source is preparation, reprep and reanalyze method blank and all samples (other than those in 1) associated with the contaminated blank. |

# TRUESDAIL LABORATORIES, INC.

INDEPENDENT TESTING, FORENSIC SCIENCE, AND ENVIRONMENTAL ANALYSES

*Established 1931*

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August 30, 1999

Ms. Elisabeth Fruth
Earth Tech
100 W. Broadway, Ste. 5000
Long Beach, CA 90802-4443

**RE: TRUESDAIL CORRECTIVE ACTION RESPONSE - AUDIT PERFORMED BY MR. CIRILLO ON 8/3/99 OF EPA METHOD 8315M WITHIN AFCEE AND EPA REQUIREMENTS.**

Dear Ms. Fruth:

I would like to thank you for Mr. Jim Cirillo's visit to our laboratory on August 3, 1999. We appreciate the time he took to assist us in making improvements in the quality of work necessary for the AFCEE program.

Mr. Cirillo's letter dated August 9, 1999 expressed his general comments and findings observed during his audit of our facility performed on August 3, 1999. I have responded to many of Mr. Cirillo's general comments and findings. Mr. Joe Bramblett, Organics Section Manager, has addressed many of Mr. Cirillo's concerns that are technical in nature. Please see Mr. Bramblets attached memorandum.

The findings from Mr. Cirillo's QA audit indicated corrective action be taken. The following corrective action taken includes:

**CORRECTIVE ACTION: (refer to the Earth Tech letter dated, 9 August 1999)**

**General Comments**

1. See attached.

2. See attached.

3. See attached.

**

**SPECIFIC COMMENTS**

**Section** **Sample Control**
**Rupal Patel, Joe Bramblett, Charles Hamilton**

**General Information**

2. Attached is a revised temperature log that will be placed into service at the first of September.

   The laboratory documents the discovery and removal of malfunctioning thermometers. We remove the malfunctioning thermometer from service and document the failure by writing on the daily temperature log. This is the same daily temperature log that we use to record the daily temperature when the thermometer is "in-control." We explain when, why and how the thermometer malfunctioned to the best of our ability. The temperature log would become part of the record we keep for 7 years.

**Findings**

1. Attached is the temperature log for the receiving refrigerator in question. Our facilities manager repaired the cooling system in the refrigerator in question. The refrigerator is working properly now. See the August temperature log for the refrigerator in question.

**

**Section: Hydrazine Sample Prep – (8315M)**
**Der-Haw Wang, Arvinder Sidhu, Yvonne Nguyen, Joe Bramblett**

**General Information**

1. Method 8315M is being reviewed and should be finalized in the near future per Joe Bramblett.

**Findings**

1. All SOP's are currently on a revision cycle. We will continue on that cycle, but during revision we will add the applicable extraction methods as reference.

2. Our new format for revised SOP's includes a cover page. Therefore, some of the methods you saw were updated and some were not. Again, we will continue on our revision schedule as stated in #1. See attached.

3. See attached.

4. I have reviewed standard label preparation with the analysts and have emphasized your finding that it would be clearer to have the analysts initials on the label. However, the ID number on the label is referenced to a logbook which incorporates everything you stated (material ID, concentration, preparation, expiration date, and the preparers name and initials). Please note our labels already include the prep number, the material name, concentration, expiration date, and sometimes the analysts initials. See attached.

5. See attached.

7. The chemist inadvertently left off his/her initials. I have mentioned this to this person. All extraction chronicles have been initialed since the 5/12/99 incident. See attached.

8. All balances including the Fisher Model XT410 (S/N 28961) are calibrated by an outside vendor (Watson Brothers, Inc) on a yearly basis. Our analytical balances are cross checked on a daily basis with a Class S weight (1 g) and documented to ±0.0001 g. However, the balance you referenced was not cross checked on a daily basis because of it's accuracy. The balance weighs to ±.01 g. I will start a logbook that will log the weight of a calibrated Class S weight (approximately 5 g). This will be done on a daily basis as our internal check.

**

**Section:** **HPLC (8315M)**
**Der-Haw Wang, Arvinder Sidhu, Yvonne Nguyen, Joe Bramblett**

**Findings**

This entire section is addressed in Mr. Brambletts memorandum. See attached.

**

**Section:** **Quality Assurance/Quality Control**
**Charles Hamilton, Joe Bramblett**

**Findings**

1. Control charts were faxed on 8/6/99. See attached.

If you require more information regarding the quality assurance procedures, please feel free to call me at 714-730-6239, x258 or Mr. Joe Bramblett at x260.

Sincerely,

Chuck Hamilton

Chuck Hamilton, Manager
Quality Assurance

Cc: Mr. Joe Bramblett, TLI Organics Section Manager
Mr. Steve Roesch, TLI Director, Environmental Sciences

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www.truesdail.com

August 31, 1999

Earth Tech
100 West Broadway, Suite 5000
Long Beach, CA 90802-4443

Attn: Mr. Jim Cirillo

**RE: Response to audit of EPA Method 8315M within AFCEE and EPA requirements.**

Dear Mr. Cirillo:

I would like to thank you again for your visit to our laboratory on August 3, 1999. We appreciate the time you took to assist us in making improvements in the quality of work necessary for the AFCEE program.

**GENERAL COMMENTS**

1. A copy of the updated SOP for EPA Method 8315M is enclosed for your review. The use of the designation EPA Method 8315M stems from the approach taken to produce a method compatible with EPA Method 8315 and CARB Method 1004. The primary modification involves switching the role of the derivatizing reagent and the analyte. In 8315M, the derivatizing agent is an aldehyde, while the analytes are hydrazines. We consider our choice of derivatizing agent to be proprietary.

The buffered, aqueous reaction media used in EPA Method 8315 is the same with one modification, which we consider proprietary. The extraction of soils uses acidified, distilled reagent water which is added to the buffered, aqueous reaction media after soil extraction. EPA Method 8315 uses the buffered media as the extraction solvent for aldehydes and ketones.

The extraction solvent, methylene chloride, and the exchange solvent, acetonitrile, are the same solvents used to extract the buffered, aqueous reaction media after the derivatization. We found that the derivatives for EPA Method 8315 and 8315M are less prone to decomposition during solvent exchange in a Turbo-vap concentrator. EPA Method 8315 uses a Kuderna-Danish (K-D) concentrator which subjects the derivatives to higher temperatures in air during the solvent exchange. The Turbo-vap provides a nitrogen headspace.

We also found that the derivative compounds of the hydrazines in 8315M performed well under the same HPLC chromatography conditions as the derivatives in EPA Method 8315. EPA Method 8315 allows modifications to the mobile phase to improve separations.

There have been on going improvements in the method as we gain experience. The current laboratory SOP is enclosed with the proprietary parts, available to the analyst, left out of this copy.

2. We are addressing your specific comments in your letter of July 27, 1999. We are reorganizing the documentation for Lab #602129 to clarify some of the confusion. The second, Lab #416036, is issued to track the QA/QC data supporting Lab #602129 and any other job run in the same batch. Copies of the QA/QC data from Lab #416036 is filed with the customers file and the original QA/QC file, labelled Lab #416036, is filed in our QA/QC department files where, for example, control charts can be prepared and distributed back to the analysts.

3. We have attempted to make it clear that the method is not routine. Hydrazine is very unstable, unlike most of the target compounds found on EPA's Priority Pollutant List. Our data for soil is very limited because the small numbers of soil samples submitted for hydrazine testing. Laboratories that have tested for hydrazine in the past have found that the hydrazines decay rapidly. This may be due to oxidation, reaction with other chemicals commonly found in soil, such as acetone, and other metabolic products like sugars and proteins from microbial active, and possibly binding to silica.

Spikes into the traditional laboratory soil matrix, clean sand, are also low. Again, oxidation and binding to the silica is a possibility. In addition, we have found formaldehyde present in the laboratory sand which is highly reactive with hydrazines. We are still uncertain of what to use for a "blank" soil media for the hydrazines. Formaldehyde is not removed by the normal solvent wash and baking. In fact, this tends to compound the problem.

**SPECIFIC COMMENTS**
**Section: Hydrazine Sample Prep - (8315M)**

**General Information**

**Findings**

2. Method 8315M is being reviewed and should be finalized in the near future. We have added a column oven to improve the reproducibility of retention times. This may require small adjustments in the mobile phase. Once this is completed, the final version of the SOP will be reviewed formally.

3. All Certificates of Analysis (CoA) for primary hydrazine standards are now available and filed correctly.

4. At this time our labels are hand written with limited room for detail. The Extraction Standards logbook remains in the Extraction Lab room. Our logbooks are tracked like a check register in a bank checkbook with opening date and closing date. The Standard ID number incorporates the date prepared. The last two digits refers to the sequence of standards prepared on that day; the standard concentration correlates to the standard concentration reported in the Extraction Logbook.

E-09-04-28-06; Extraction, April 28, 1999, #6 preparation.

This information allows us to quickly find the remaining information, such as material, analyte concentrations, preparation date and expiration date and the preparer's initials. It is our policy to include as a minimum the expiration date and preparer's initials as a minimum and this was an oversight that has been corrected.

5. No soil results have been reported to Earth Tech.

7. Analysts have been reminded to sign Extraction Sample Logbook.

**RECOMMENDATIONS**

1. We are considering a possible numbering scheme for the log books to help identify them. However, each logbook has a unique name and purpose similar to a bank checkbook.

**Section: HPLC (8315M)**

**General Information**

**Findings**

1. The 1994 SOP referred to as EPA Method 8315M is the original EPA Method 8315 (for aldehydes & ketones). This SOP is in revision and will be posted; there have been minimal changes in this method since 1994. The EPA Method 8315M (for hydrazines) dated 12/97 is in revision. This version was written within the first two months of our method development for hydrazines. The analyst have the most current version. Most of the improvements have been completed over the last 18 months and will be reflected in the current revision.

2. The field sample preservation requirement has been included in the current SOP.

3. This has been corrected. The Shimadzu HPLC instrument logbook is an old format which we have already changed on the majority of the instruments in the laboratory. We have also changed to this format for the Shimadzu HPLC instruments since the audit. A copy is enclosed for your review.

4. We have started a formal supervisory review for all logbooks; a bound maintenance logbook has been assigned to the Shimadzu HPLC. We are using the lower section of the instrument logbook to monitor routine maintenance. We will also use this section to indicate return to control. The supervisory review will be used to monitor complete documentation, including analyst's initials, etc.

5. Please see Paragraph 4.

6. The instrument runlog has been modified to reflect the start date and stop date for the batch. Approximate start and stop times will be entered on the runlog. Please note that the date system records the start time and date of each run and is available in the documentation.

7. Please see Paragraph 6.

8. Retention time windows in HPLC are less reproducible from batch to batch in HPLC than in GC. HPLC's generally do not come with thermostated column ovens. We have recently added a thermostated column to our Shimadzu HPLC to control the ambient temperature which varies significantly in the current laboratory location. We have found that this has made a significant improvement in retention time reproducibility. However, it may still not be as reproducible as GC; We are currently evaluating the new RT windows. We have also found that it is necessary to make up the mobile phase in sufficient quantity to last for an entire batch run. The reproducibility of retention times is very sensitive to the smallest difference in mobile phase composition. Our approach for this method has been to closely monitor the retention times of the ICAV and subsequent CCV standard runs to estimate the retention time windows. The method does not have a suitable surrogate for monitoring retention times.

9. The low standard concentration is MMH (5 ppb), UDMH (5 ppb) and Hydrazine (1 ppb). The correct reporting limit or PQL is MMH (5 ppb), UDMH (5 ppb) and Hydrazine (1 ppb). The first report to Earth Tech used estimated detection limits from the analyst's spreadsheet which were incorrect and not based on the MDL and lowest standard concentration. We have corrected this information on the spreadsheet.

10. Please see Paragraph 9.

**RECOMMENDATIONS**

1. We agree. We will add the concentration or range or analyte concentrations to the run logbook to make it easier to ID the standard. Our current data system allows only 8 spaces for the sample queue identification and will be limited to abbreviated ID's.

2. The run log for the Shimadzu HPLC has been converted to the same format that we are using for GC's. The upgrade for all instruments in the laboratory is now complete.

**Section: Quality Assurance/Quality Control**

**General Information**

**Findings**

1. Copies of a current MDL Study and Control Charts are enclosed and available in the laboratory.

Respectfully,

Joe Bramblett, Manager
Organics Section

TRUESDAIL LABORATORIES, INC.

# TEMPERATURE CHART

MONTH _____

LOCATION _____

REQUIRED TEMPERATURE _____

ACCEPTABLE RANGE _____

CORRECTION FACTOR _____

THERMOMETER EXPIRATION _____

| DATE | TEMPERATURE C | INITIAL | COMMENTS |
|---|---|---|---|
| 1 | | | |
| 2 | | | |
| 3 | | | |
| 4 | | | |
| 5 | | | |
| 6 | | | |
| 7 | | | |
| 8 | | | |
| 9 | | | |
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| 25 | | | |
| 26 | | | |
| 27 | | | |
| 28 | | | |
| 29 | | | |
| 30 | | | |
| 31 | | | |

- **Report Out of Range Temperature to Quality Control and/or Supervisor Immediately.**

Return form to the QC Officer at the end of the month. Examine thermometer and verify that it is still "in control" and within calibration dates.

**TRUESDAIL LABORATORIES, INC.**

TEMPERATURE CHART FOR Aug 99

LOCATION LOG-IN RECEIVING LAB

Required Temperature 4 C

Acceptable Range ± 2 C

| DATE | TEMPERATURE C | INITIAL | COMMENTS |
|---|---|---|---|
| 1 | | | |
| 2 | Placed in | Service | nonfunc |
| 3 | 10°C | CBH | not broken |
| 4 | | | |
| 5 | | | |
| 6 | | | |
| 7 | | | |
| 8 | | back in | service CBH 7/19/99 |
| 9 | 3 | CBH | |
| 10 | 3 | CBH | |
| 11 | 3 | DR | |
| 12 | 3 | MN | |
| 13 | 3 | MN | |
| 14 | | | |
| 15 | | | |
| 16 | 3 | CBH | |
| 17 | 3 | CBH | |
| 18 | 3 | CBH | |
| 19 | 4 | CBH | |
| 20 | 4 | CBH | |
| 21 | | | |
| 22 | | | |
| 23 | 3 | CBH | |
| 24 | 4 | CBH | |
| 25 | 4 | CBH | |
| 26 | 3 | CBH | |
| 27 | 3 | CBH | |
| 28 | | | |
| 29 | | | |
| 30 | 3 | CBH | |
| 31 | 3 | CBH | |

Stored CBH 8/3/9

REPORT OUT OF RANGE TEMPERATURE TO THE SUPERVISOR IMMEDIATELY

Return to the QC Officer at the end of the month. Examine thermometer and verify that it is still within it's calibration dates.

# STANDARD OPERATING PROCEDURE FOR:

*The Determination of Hydrazine, Monomethylhydrazine, and Unsymmetrical Dimethylhydrazine in Aqueous Solutions by High Performance Liquid Chromatography (HPLC)*

## Referenced Method

EPA 8315 modified for hydrazine, monomethylhydrazine (MMH), and unsymmetrical dimethylhydrazine (UDMH).

## Scope of Application

This method provides procedures for the determination of hydrazine, MMH, and UDMH in aqueous solutions by derivatization with PROPRIETARY. The method utilizes HPLC with UV/VIS detection. The following compounds may be determined by this method:

Hydrazine
Monomethylhydrazine
Unsymmetrical Dimethylhydrazine

## Interferences

Solvents, reagents, glassware, and other hardware may be contaminated in ways that result in discrete artifacts or elevated baselines. Demonstrate that the reagents and apparatus are free from interference by running laboratory reagent blanks, with each batch of samples consisting of one to ten samples.

## Sample Collection, Preservation & Storage

Liquid samples must be acidified with $H_2SO_4$ to pH less than 2 and refrigerated at 4°C. Aqueous samples should be derivatized and extracted within 3 days of sample collection. All derivatized sample extracts should be analyzed within 3 days after preparation.

## Apparatus

1) 250ml Erlenmeyer flask with Teflon stopcock.

2) 500ml separatory funnel with Teflon stopcock.

3) 200ml TurboVap Sample Tube.

4) TurboVap II Concentration Workstation

5) Sample shaker - wrist-action shaker.

6) Shaker bed.

7) Vials - 10ml, 7ml, and 2ml, clear with Teflon-lined screw caps.

8) 150ml Bucher funnel with medium frit

9) Disposable pipette.

10) High Performance Liquid Chromatograph

    a) Absorbance detector - 360nm

    b) Column - Supelcosil (Supelco) LC-18, 25cm X 4.6mm ID, 5µm particle size.

11) Mobile phase reservoirs.

**Reagents**

1) Acetonitrile (HPLC grade).

2) Methylene chloride (pesticide quality or equivalent).

3) Distilled water.

4) Acetic acid (glacial).

5) Sodium hydroxide pellets.

7) Derivatizing solution. Preparation as follows:

    a) PROPRIETARY.

    b) PROPRIETARY.

    c) PROPRIETARY.

    d) Transfer to 10ml vial.

    e) PROPRIETARY.

8) Sodium hydroxide, NaOH, 5.0 N - Dissolve 20g of NaOH pellets in 100ml distilled water.

9) Acetate buffer, 5 N - Neutralize 50ml of glacial acetic acid to pH 5 with 60ml of 5 N NaOH solution to 174 ml with distilled water.

10) Water - HPLC grade

11) Tetrahydrofuran - HPLC grade.

12) Stock solutions of 10,000 g/ml hydrazine, 10,000mg/ml MMH, and 10,000µg/ml UDMH were prepared by addition of appropriate amounts of each to separate 10ml volumetric flasks containing 5ml of distilled water acidified with hydrochloric acid to pH<2. Dilution to the mark was made with additional acidified distilled water. Calibration standards are prepared by further dilution of aliquots of these stock solutions with acidified distilled water.

**Procedure**

Derivatization and Extraction

1) For liquid samples, proceed Step 5. For soil samples, measure approximately 20g of sample and transfer it to a 250ml Erlenmeyer flask (use bulk sand for Method Blank).

2) Add 110 ml of de-ionized water acidified to pH less than 2 with concentrated sulfuric acid.

3) Seal the Erlenmeyer flask with the stopcock and place it in a wrist-action shaker for half an hour.

4) Filter the sample, and collect the filtrate into another new 250ml Erlenmeyer flask. Proceed Step 6.

5) Take a 100ml aliquot of sample and quantitatively transfer it to a 250ml Erlenmeyer flask (use distilled water for Method Blank).

6) Add 2ml of 5 N acetate buffer, and adjust the pH to 5 with acetic acid or 5.0 N NaOH.

7) PROPRIETARY.

8) Add 1ml of derivatizing solution. Seal the Erlenmeyer flask with the stopcock and place it in a wrist-action shaker. PROPRIETARY.

9) Add 20ml of methylene chloride to a 500ml separatory funnel. Then add the entire sample and shake the separatory funnel for 90 seconds, venting any pressure which develops in the funnel. Let the layers separate.

10) Transfer the methylene chloride layer (bottom layer) to a 200ml TurboVap sample tube (do not allow any water to drain).

11) Add 20 ml of fresh methylene chloride to the 250ml Erlenmeyer flask, swirl, and pour it into the separatory funnel. Shake funnel for 60 seconds, let the layers separate as before and drain the methylene chloride into the 200ml TurboVap sample tube.

12) Repeat step 10 one more time.

13) Add 4 ml of acetonitrile into the 200ml TurboVap sample tube.

14) Set the water temperature of the TurboVap II Concentration Workstation at 39°C and the inlet pressure of nitrogen gas at approximately 10 psi.

15) Blow down to approximately 4ml and add 3ml of acetonitrile into the 200ml TurboVap sample tube.

16) Further concentrate to 3ml.

17) Quantitatively transfer the sample to a 7ml screw cap vial. Rinse the 200mL TurboVap sample tube once with 1ml of acetonitrile.

18) Adjust volume to 5ml.

19) If necessary, filter the sample through a 0.45µ nylon membrane filter prior to HPLC analysis.

20) Pipet 1.5ml of the sample into a 2ml Teflon-lined screw cap vial and proceed with HPLC analysis using the conditions described under HPLC conditions.

Mobile Phase Preparation

Mobile phase A: Prepared by mixing water, acetonitrile, and tetrahydrofuran (THF) in the following proportions - 60:30:10 respectively. For example, to prepare 1000ml of the mobile phase, mix 600ml of water, 300ml of ACN, and 100ml of THF. Shake reservoir to mix reagents thoroughly.

Mobile phase B: Prepared by mixing 40 parts of water with 60 parts of acetonitrile. For example, use 400ml of water and 600ml of acetonitrile to prepare 1000ml of mobile phase. Shake reservoir to mix reagents thoroughly.

**HPLC Conditions**

Instrument: Shimadzu 6A with UV detector
Column: Supelco, LC-18, 25mm x 4.6mm, 5Om
Col. Temp.: Ambient
Flowrate: 1.5ml/min
Detector: Ultraviolet
Wavelength: 360nm
Sensitivity: 0.01 AU
Injection Volume: 25µl
Mobile Phase:

| | |
|---|---|
| Initial: | Pump A (100% mobile phase A). Hold 5 min. |
| Program to: | Linear from 5 min. to 22 min. to 100% Pump B (mobile phase B) |
| Final: | Hold 100% Pump B from 22 min. to 30 min. |
| Equilibration: | Return to 100% Pump A and hold 8 min. |

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# DAILY SAMPLE LOG
# SHIMADZU HPLC #1

**FROM:** 8/9/99 **TO:** _____

**Truesdail Laboratories , Inc.**

**Shimadzu HPLC #1**

**DAILY SAMPLE LOG**

**Date Analyzed:** 8/13/99

**Start Time:** 3:30 PM

**Stop Time:** 8:00 PM

**Inst. Batch No.:** 1

| Sample ID | Dil. Factor | Method No. | Notes (see below)* |
|---|---|---|---|
| ~~602221 - Std #1~~ Dil MP | 1 | 8315M | Re-run samples. |
| 602221 - Std #1 | 1 | | |
| 602221 - Std #2 | 1 | | |
| 602221 - Std #3 | 1 | | |
| 602221 - Std #4 | 1 | | |
| 602221 - Std #5 | 1 | | |
| MP Blank | 1 | | DH. |

**Daily Instrument Maintenance Log**

Pump A Seal replaced - Part # 228-11599-00
8/13/99 A.M. - Return to service, 8/13/99 PM.
8/13/99

**Analyst (Print Name):** JOE BRAMBLETT

*Enter what applies: Client, Standard ID., Quality Control (LCS/MS)