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(1) Out of Range
**Quantitation Report**

Data File: D:\HPCHEM\1\DATA\031905\P0319018.D  
Acq On: 19 Mar 2005 3:54 pm  
Sample: 1.0 PPB CAL  
Misc: 1X 10ML  
MS Integration Params: DIOXANE.P  
Quant Time: Mar 21 12:54 2005  
Vial: 18  
Operator: JG/MS/CLS  
Inst: GCMS1  
Multiplier: 1.00  
Quant Results File: DX031905.RES

Quant Method: D: \HPCHEM\1\METHODS\DX031905.M (RTE Integrator)  
Title: 8260 1,4-Dioxane Ini Cal (05/02/02)  
Last Update: Mon Mar 21 12:54:07 2005  
Response via: Initial Calibration  
DataAcq Meth: DX021605

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<th>QIon</th>
<th>Response</th>
<th>Conc</th>
<th>Units</th>
<th>Dev(Min)</th>
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</table>

System Monitoring Compounds  
2) Dibromofluoromethane (SUI) | 10.07 | 113 | 3733 | 0.12 | ug/L | 0.00 |
Spiked Amount 1.000 | Range 80 - 120 | Recovery = 12.00% #

Target Compounds  
4) 1,4-DIOXANE | 12.43 | 98 | 668 | 1.07 | ug/L | 96 |
6) 1,2,3-Trichloropropane | 0.00 | 75 | 0 | N.D. |
Quantitation Report

Data File: D:\HPCHEM\1\DATA\031905\P0319018.D  Vial: 18
Acq On: 19 Mar 2005  3:54 pm  Operator: JG/MS/CLS
Sample: 1.0 PPB CAL  Inst: GCMS1
Misc: 1X 10ML  Multipl: 1.00
MS Integration Params: DIOXANE.P
Quant Time: Mar 21 12:54 2005  Quant Results File: DX031905.RES

Method: D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 12:54:07 2005
Response via: Initial Calibration

Abundance

TIC: P0319018.D

Time -> 0.00  8.00  9.00  10.00  11.00  12.00  13.00  14.00  15.00  16.00  17.00  18.00  19.00  20.00  21.00  22.00  23.00
23000  22000  21000  20000  19000  18000  17000  16000  15000  14000  13000  12000  11000  10000  9000  8000  7000  6000  5000  4000  3000  2000  1000
1,4-DIOXANE BY METHOD 8250B SIM

Data File Name P0319019.D
Data File Path D:\HPCHEM1\DATA631906SP0319019.D
Sample Name SS/CCV

Date Acquired 3/19/2005 4:27
Operator JG/MS/CL3
Acq. Method File DX021605
GCMS1

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<tr>
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<th>CAL RESPONSE</th>
<th>TARGET RESPONSE</th>
<th>LOW LIMIT</th>
<th>HIGH LIMIT</th>
<th>T/F</th>
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<tbody>
<tr>
<td>Pentfluorobenzene (IS)</td>
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<td>23536</td>
<td>94142</td>
<td>TRUE</td>
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<td>1,4-DIOXANE-d8</td>
<td>5034</td>
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<th>SURROGATE</th>
<th>AMOUNT</th>
<th>% RECOVERY</th>
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<th>T/F</th>
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<tr>
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Quantitation Report

Data File: D:\HPCHEM\1\DATA\031905\P0319019.D
Acq On: 19 Mar 2005 4:27 pm
Sample: SS/CCV
Misc: 1X 10ML
MS Integration Params: DIOXANE.P
Quant Time: Mar 21 12:54 2005
Quant Results File: DX031905.RES

Vial: 19
Operator: JG/MS/CLS
Inst: GCMS1
Multiplier: 1.00

Quant Method: D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 12:54:07 2005
Response via: Initial Calibration
DataAcq Meth: DX021605

Internal Standards

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<tr>
<th>R.T.</th>
<th>QIon</th>
<th>Response</th>
<th>Conc Units</th>
<th>Dev(Min)</th>
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<tbody>
<tr>
<td>1)</td>
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<td>99</td>
<td>46539</td>
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<td>3)</td>
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<tr>
<td>5)</td>
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<td>0.00</td>
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System Monitoring Compounds

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<tr>
<th>Spiked Amount</th>
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<th>Recovery</th>
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<tbody>
<tr>
<td>1.00</td>
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Target Compounds

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<th>Dev(Min)</th>
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<td>6)</td>
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Quantitation Report

Data File: D:\HPCHEM\DATA\O31905\P0319019.D
Acq On: 19 Mar 2005 4:27 pm
Sample: SS/CCV
Misc: 1X 10ML
MS Integration Params: DIOXANE.P
Quant Time: Mar 21 12:54 2005

Method: D:\HPCHEM\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 12:54:07 2005
Response via: Initial Calibration

TIC: P0319019.D

Abundance

METHOD CRITERIA

1. Sequence File is printed and in the file folder? 
   Standard IDs and analyst's initials are present?

2. Initial Calibration met criteria?
   a. Print calibration as Average Response Factor
      (624: RSD ≤ 35%)
      (8260B: ≤ 30% for CCCs, ≤ 15% for all other compounds, SPCCs met Criteria)
      (524.2: RSD ≤ 20%)
   b. If non CCC RSD > 15%, print out the curve as Linear Regression
      r ≥ 0.995 or \( r^2 \geq 0.99 \) (do not force through zero for 8260B)
   c. If non CCC RSD > 15%, print out the curve as Quadratic
      r ≥ 0.995 or \( r^2 \geq 0.99 \) (do not force through zero for 8260B)
   d. Choose option (b or c) with the least negative intercept
   e. Requant the low (RL) standard against the curve
      must be ± 30%, file with the calibration for reference
   f. If samples contain negative values then:
      compare the area counts with the low standard on file
      if <, then report as N.D. with no flag
      if >, then report from RSD curve and flag that curve is out
      or report at an elevated RL as compared to a curve standard

3. Initial Midpoint / LCS / BFB Tune
   (624: use Table 5) (524.2: ±30%) (8260B: see control chart)
   SPCCs met criteria? CCCs met criteria (±20%)?

4. Checked integration of all peaks in Midpoint?

5. Method Blank < Report Limit, if not is data flagged?
   (624: every 20 samples) (524.2: every 12 hours) (8260B: every 12 hours)

6. MS/MSD (every 20 samples)
   (624: use Table 5) (524.2: N/A) (8260B: see Control Chart)

7. All samples met holding time? (Soil 72hr ext, 7/14days water)

8. All water samples checked to be pH < 2? (Note this on the sequence file)

9. LCS every 20 samples
   (624: See Table 5) (524.2: ±30%) (8260B: See Control Chart)

10. Cont. Midpoint / LCS / BFB Tune done every 12 hours
    (624: use Table 5) (524.2: ±30%) (8260B: see control chart)
    SPCCs met criteria? CCCs met criteria (±20%)?

11. Surrogates within acceptance limits
    (624 / 524.2 / 8260B: See Control Chart)

12. Internal Standards within acceptance limits
    (624 / 524.2 / 8260B: response must be -50 to +100%)

13. Manual re-integration(s) performed?
    yes: ________ no: ________

14. Corrective Action Report required?
    yes: ________ (Attached) no: ________

15. Reports impacted by the Corrective Action Report:

Analyst: ___________________ Reviewer / Date: ____________
# DMAP GC/MS 1 DAILY LOG SUMMARY

**DATE:** 3-11-05  
**QC BATCH # (s):** 521128  
**ANALYST:** cfs  
**SEQUENCE FILE:** C:\GCMS1\DATA\05-105.5  
**CALIBRATION METHOD(S):** Ox031905  

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**STANDARD ID NUMBERS**

- **CCV:** 
- **H2O LCS / H20 SPIKE:** 50500010  
- **Internal Std:**  
- **CALIBRATION STD:** NA  
- **IS / Surrogate / BFB:** 5050D11  
- **REVIEWER / DATE:** 4/6/05

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Page 1

12 May 2005 15:36
**1,4-DIOXANE BY METHOD 8260B SIM**

Data File Name: P0511002.D
Data File Path: D:\HPCHEM1\DATA\P0511002.D
Sample Name: pSe1128-ba1

Date Acquired: 5/11/2005 2:56
Operator: cs
Acq. Method File: DX031905
GC MS1

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<th>TARGET RESPONSE</th>
<th>LOW LIMIT</th>
<th>HIGH LIMIT</th>
<th>T/F</th>
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<tbody>
<tr>
<td>Pentafluorobenzene (IS)</td>
<td>47071</td>
<td>39462</td>
<td>23536</td>
<td>94142</td>
<td>TRUE</td>
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<td>1,4-DIOXANE-d8</td>
<td>5034</td>
<td>6966</td>
<td>2517</td>
<td>10068</td>
<td>TRUE</td>
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<tr>
<th>SURROGATE</th>
<th>AMOUNT</th>
<th>% RECOVERY</th>
<th>Low</th>
<th>High</th>
<th>T/F</th>
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<tbody>
<tr>
<td>Dibromofluoromethane (SU1)</td>
<td>0.97</td>
<td>96.5</td>
<td>80</td>
<td>125</td>
<td>TRUE</td>
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<tr>
<th>TARGET ANALYTE</th>
<th>AMOUNT</th>
<th>TRUE VALUE</th>
<th>RECOVER</th>
<th>Low</th>
<th>High</th>
<th>T/F</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,4-DIOXANE</td>
<td>10.68</td>
<td>10.00</td>
<td>106.79</td>
<td>70</td>
<td>130</td>
<td>TRUE</td>
</tr>
</tbody>
</table>
Quantitation Report

Data File: D:\HPCHEM\DATA\051105\P0511002.D
Acq On: 11 May 2005 2:56 pm
Sample: p5e1128-bs1
Misc: 1X 10ML
MS Integration Params: DIOXANE.P
Quant Time: May 12 10:02 2005

Quant Method: D:\HPCHEM\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 07:49:30 2005
Response via: Initial Calibration
DataAcq Meth: DX031905

Internal Standards | R.T. QIon | Response | Conc Units | Dev(Min)
-------------------|----------|----------|------------|--------
1) Pentafluorobenzene (IS) | 10.56 | 99 | 39462 | 1.00 ug/L | 0.00
3) 1,4-DIOXANE-d8 | 12.35 | 64 | 6968 | 25.00 ug/L | 0.00
5) 1,2,3-Trichloropropane-d5 | 0.00 | 79 | 0 | 0.00 ug/L | -15.08

System Monitoring Compounds
2) Dibromofluoromethane (SUI) | 10.07 | 113 | 28790 | 0.97 ug/L | 0.00
Spiked Amount | 1.000 | Range 80 - 120 | Recovery = 97.00%

Target Compounds
4) 1,4-DIOXANE | 12.43 | 88 | 5632 | 10.68 ug/L | 97

(#) = qualifier out of range (m) = manual integration
Evaluate Continuing Calibration Report

Data File: D:\HPCHEM\1\DATA\051105\P0511002.D
Acq On: 11 May 2005 2:56 pm
Sample: p5e1128-bs1
Misc: 1X 10ML
MS Integration Params: DIOXANE.P

Vial: 2
Operator: cs
Inst: GCMS1
Multiplier: 1.00

Method: D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 07:49:30 2005
Response via: Multiple Level Calibration

Min. RRF: 0.100  Min. Rel. Area: 50%  Max. R.T. Dev: 0.50min
Max. RRF Dev: 30%  Max. Rel. Area: 200%

<table>
<thead>
<tr>
<th>Compound</th>
<th>AvgRF</th>
<th>CCRF</th>
<th>%Dev</th>
<th>Area%</th>
<th>Dev(min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 I</td>
<td>Pentafluorobenzene (IS)</td>
<td>1.000</td>
<td>1.000</td>
<td>0.0</td>
<td>#84 0.00</td>
</tr>
<tr>
<td>2 S</td>
<td>Dibromofluoromethane (SU1)</td>
<td>0.756</td>
<td>0.730</td>
<td>3.4</td>
<td>#84 0.00</td>
</tr>
<tr>
<td>3 I</td>
<td>1,4-DIOXANE-d8</td>
<td>1.000</td>
<td>1.000</td>
<td>0.0</td>
<td>138 0.00</td>
</tr>
<tr>
<td>4 T</td>
<td>1,4-DIOXANE</td>
<td>2.130</td>
<td>2.021</td>
<td>5.1</td>
<td>147 0.00</td>
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<tr>
<td>5 I</td>
<td>1,2,3-Trichloropropane-d5</td>
<td>1.000</td>
<td>1.000</td>
<td>0.0</td>
<td>0# -15.08#</td>
</tr>
<tr>
<td>6 T</td>
<td>1,2,3-Trichloropropane</td>
<td>0.000</td>
<td>0.000#</td>
<td>0.0</td>
<td>0# -15.08#</td>
</tr>
</tbody>
</table>

(#): Out of Range  SPCC's out = 0  CCC's out = 0
F0511002.D  DX031905.M  Thu May 12 10:02:46 2005  GCMS1

Pag 57
## 1,4-DIOXANE BY METHOD 8260B SIM

**Data File Name:** P0511003.D  
**Data File Path:** D:\HPCHEM\DATA\0511005\  
**Sample Name:** ps5e1128-bsd1  
**Date Acquired:** 5/11/2005 3:29  
**Operator:** cs  
**Acq. Method File:** DX031905  
**GCMS1**

<table>
<thead>
<tr>
<th>INTERNAL STANDARDS</th>
<th>CAL RESPONSE</th>
<th>TARGET RESPONSE</th>
<th>LOW LIMIT</th>
<th>HIGH LIMIT</th>
<th>T/F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pentfluorobenzene (IS)</td>
<td>47071</td>
<td>40733</td>
<td>23538</td>
<td>94142</td>
<td>TRUE</td>
</tr>
<tr>
<td>1,4-DIOXANE-d8</td>
<td>5034</td>
<td>7703</td>
<td>2517</td>
<td>10068</td>
<td>TRUE</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>SURROGATE</th>
<th>AMOUNT</th>
<th>% RECOVERY</th>
<th>Low</th>
<th>High</th>
<th>T/F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dibromodifluoromethane (SU1)</td>
<td>0.96</td>
<td>96.5</td>
<td>80</td>
<td>125</td>
<td>TRUE</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>TARGET ANALYTE</th>
<th>AMOUNT</th>
<th>TRUE VALUE</th>
<th>RECOVER</th>
<th>Low</th>
<th>High</th>
<th>T/F</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,4-DIOXANE</td>
<td>9.63</td>
<td>10.00</td>
<td>96.29</td>
<td>70</td>
<td>130</td>
<td>TRUE</td>
</tr>
</tbody>
</table>

---

**Note:**  
1. All values are in mg/L.  
2. The table above summarizes the results of the analysis.  
3. The true value for 1,4-DIOXANE is 10.00 mg/L.  
4. The recovery is 96.29%.  
5. The low limit is 70 mg/L, and the high limit is 130 mg/L.  
6. The T/F column indicates the presence or absence of a particular condition.  
7. The operator notes at the bottom of the page indicate the data was reviewed and approved.
Quantitation Report

Data File: D:\HPCHM\1\DATA\051105\P0511003.D
Acq On : 11 May 2005 3:29 pm
Sample : p5e1128-bsd1
Misc : 1X 10ML
MS Integration Params: DIOXANE.P
Quant Time: May 12 10:02 2005

Vial: 3
Operator: cs
Inst : GCMS1
Multiplier: 1.00

Quant Results File: DX031905.RES

Quant Method : D:\HPCHM\1\METHODS\DX031905.M (RTE Integrator)
Title : 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update : Mon Mar 21 07:49:30 2005
Response via : Initial Calibration
DataAcq Meth : DX031905

Internal Standards

<table>
<thead>
<tr>
<th>R.T.</th>
<th>Qion</th>
<th>Response</th>
<th>Conc Units</th>
<th>Dev(Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1)</td>
<td>Pentafluorobenzene (IS)</td>
<td>10.56</td>
<td>99</td>
<td>40733</td>
</tr>
<tr>
<td>2)</td>
<td>1,4-DIOXANE-d8</td>
<td>12.35</td>
<td>64</td>
<td>7703</td>
</tr>
<tr>
<td>3)</td>
<td>1,2,3-Trichloropropane-d5</td>
<td>0.00</td>
<td>79</td>
<td>0</td>
</tr>
</tbody>
</table>

System Monitoring Compounds

<table>
<thead>
<tr>
<th>R.T.</th>
<th>Qion</th>
<th>Response</th>
<th>Conc Units</th>
<th>Dev(Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2)</td>
<td>Dibromofluoromethane (SU1)</td>
<td>10.07</td>
<td>113</td>
<td>29696</td>
</tr>
<tr>
<td>Spiked Amount</td>
<td>1.000</td>
<td>Range 80 - 120</td>
<td>Recovery = 96.00%</td>
<td></td>
</tr>
</tbody>
</table>

Target Compounds

<table>
<thead>
<tr>
<th>R.T.</th>
<th>Qion</th>
<th>Response</th>
<th>Conc Units</th>
<th>Dev(Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>4)</td>
<td>1,4-DIOXANE</td>
<td>12.43</td>
<td>88</td>
<td>5614</td>
</tr>
</tbody>
</table>

(#)= qualifier out of range (m)= manual integration
Quantitation Report

Data File : D:\HPCHEM\1\DATA\051105\P0511003.D
Acq On : 11 May 2005 3:29 pm
Sample : p5el128-bsd1
Misc : 1X 10ML
MS Integration Params: DIOXANE.P
Quant Time: May 12 10:02 2005

Operator: cs
Inst : GCMS1
Multiplier: 1.00

Quant Results File: DX031905.RES

Method : D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)
Title : 6260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update : Mon Mar 21 07:49:30 2005
Response via : Initial Calibration

TIC: P0511003.D

Abundance

Time->

0  1000  2000  3000  4000  5000  6000  7000  8000  9000  10000  11000  12000  13000  14000  15000  16000  17000  18000  19000  20000  21000  22000  23000

8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00 21.00 22.00 23.00

Dichloromethane (SU)1
Perfluorotributylamine (SU)1
1,4-Dioxane

P0511003.D  DX031905.M  Thu May 12 10:03:24 2005  GCMS1
Page 60
Quantitation Report (QT Reviewed)

Data File : D:\HPCHEM\1\DATA\051105\P0511004.D
Acq On : 11 May 2005  4:02 pm
Sample : p5e1128-blk1
Misc : 1X 10ML
MS Integration Params: DIOXANE.P
Quant Time: May 12 10:03 2005

Quant Method : D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)
Title : 8260  1,4-Dioxane Ini. Cal. (05/02/02)
Last Update : Mon Mar 21 07:49:30 2005
Response via : Initial Calibration
DataAcq Meth : DX031905

Quant Results File: DX031905.RES

Internal Standards                    R.T. QIon  Response  Conc Units  Dev(Min)

1) Pentfluorobenzene (IS)  10.56  99    43608 /1.00 ug/L  0.00
3) 1,4-DIOXANE-d8     12.35  64    8456  25.00 ug/L  0.00
5) 1,2,3-Trichloropropane-d5  0.00  79    0.00 0.00 ug/L  -15.08

System Monitoring Compounds
2) Dibromofluoromethane (SU1)  10.07  113   31916  0.97 ug/L  0.00
Spiked Amount  1.000  Range 80 - 120  Recovery = 97.00%

Target Compounds
4) 1,4-DIOXANE         12.43  88    208  0.33 ug/L  89

(#)= qualifier out of range  (m)= manual integration

P0511004.D  DX031905.M   Thu May 12 10:03:45 2005   GCMS1
Quantitation Report

Data File: D:\HPCHEM\1\DATA\051105\P0511004.D  Vial: 4
Acq On: 11 May 2005 4:02 pm  Operator: cs
Sample: p5e1128-bik1  Inst: GCMS1
Misc: 1X 10ML  Multipl: 1.00
MS Integration Params: DIOXANE.P
Quant Time: May 12 10:03 2005  Quant Results File: DX031905.RES

Method: D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 07:49:30 2005
Response via: Initial Calibration

Abundance

TIC: P0511004.D

Time->

0.00 1.00 2.00 3.00 4.00 5.00 6.00 7.00 8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00 21.00 22.00 23.00
#1
Pentafluorobenzene (IS)
Concen: 1.00 µg/L
RT: 10.56 min Scan# 596
Delta R.T. -0.00 min
Lab File: P0511004.D
Acq: 11 May 2005 4:02 pm
Tgt Ion: 99 Resp: 43608
Ion Ratio Lower Upper
99 100
137 24.1 3.8 43.8

#2
Dibromofluoromethane (SUI)
Concen: 1.00 µg/L
RT: 10.07 min Scan# 511
Delta R.T. -0.00 min
Lab File: P0511004.D
Acq: 11 May 2005 4:02 pm
Tgt Ion: 113 Resp: 31916
#3
1,4-DIOXANE-d8
Concen: 25.00 µg/L
RT: 12.35 min Scan# 867
Delta R.T. -0.00 min
Lab File: P0511004.D
Acq: 11 May 2005 4:02 pm
Tgt Ion: 64 Resp: 8456
Ion Ratio Lower Upper
64 100
96 112.9 72.7 172.7

#4
1,4-DIOXANE
Concen: 0.33 µg/L
RT: 12.43 min Scan# 873
Delta R.T. -0.00 min
Lab File: P0511004.D
Acq: 11 May 2005 4:02 pm
Tgt Ion: 88 Resp: 208
Ion Ratio Lower Upper
88 100
58 75.5 15.8 115.8
87 7.5 0.0 59.5
### Quantitation Report

**Data File**: D:\HPCHEM\1\DATA\051105\P0511006.D  
**Acq On**: 11 May 2005 5:07 pm  
**Sample**: poe0059-02  
**Misc**: 1X 10ML  
**MS Integration Params**: DIOXANE.P  
**Quant Time**: May 12 10:04 2005  
**Quant Results File**: DX031905.RES

**Vial**: 6  
**Operator**: cs  
**Inst**: GCMS1  
**Multiplier**: 1.00

#### Quant Method:
D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)

**Title**: 8260 1,4-Dioxane Ini. Cal. (05/02/02)

**Last Update**: Mon Mar 21 07:49:30 2005

**Response via**: Initial Calibration

**DataAcq Meth**: DX031905

#### Internal Standards

<table>
<thead>
<tr>
<th></th>
<th>R.T. QIon</th>
<th>Response</th>
<th>Conc Units</th>
<th>Dev (Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Pentafluorobenzene (IS)</td>
<td>10.56</td>
<td>99</td>
<td>30937</td>
<td>1.00 ug/L</td>
</tr>
<tr>
<td>3) 1,4-DIOXANE-d8</td>
<td>12.35</td>
<td>64</td>
<td>6052</td>
<td>25.00 ug/L</td>
</tr>
<tr>
<td>5) 1,2,3-Trichloropropane-d5</td>
<td>0.00</td>
<td>79</td>
<td>0</td>
<td>0.00 ug/L</td>
</tr>
</tbody>
</table>

#### System Monitoring Compounds

| 2) Dibromofluoromethane (SU1) | 10.07 | 113 | 23452 | 1.00 ug/L | 0.00 |

**Spiked Amount**: 1.000  
**Range**: 80 - 120  
**Recovery**: 100.00%

#### Target Compounds

| 4) 1,4-DIOXANE | 12.43 | 88 | 388 | 0.85 ug/L | 91 |

(#) = qualifier out of range (m) = manual integration

---

P0511006.D  DX031905.M  Thu May 12 10:04:06 2005  GCMS1  

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Quantitation Report

Data File: D:\HPCHEM\DATA\051105\P0511006.D
Acq On: 11 May 2005 5:07 pm
Sample: pce0059-02
Misc: 1X 10ML
MS Integration Params: DIOXANE.P
Quant Time: May 12 10:04 2005

Method: D:\HPCHEM\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 07:49:30 2005
Response via: Initial Calibration

TIC: P0511006.D

Abundance

Time->

8.00 9.00 10.00 11.00 12.00 13.00 14.00 15.00 16.00 17.00 18.00 19.00 20.00 21.00 22.00 23.00
#1
Pentafluorobenzene (IS)
Concen: 1.00 ug/L
RT: 10.56 min Scan# 596
Delta R.T. -0.00 min
Lab File: P0511006.D
Acq: 11 May 2005 5:07 pm
Tgt Ion: 99 Resp: 30937
Ion Ratio Lower Upper
99 100
137 24.5 3.8 43.8

#2
Dibromofluoromethane (SU1)
Concen: 1.00 ug/L
RT: 10.07 min Scan# 511
Delta R.T. -0.00 min
Lab File: P0511006.D
Acq: 11 May 2005 5:07 pm
Tgt Ion: 113 Resp: 23451
1,4-DIOXANE-d8

Concen: 25.00 µg/L
RT: 12.35 min Scan# 867
Delta R.T. -0.00 min
Lab File: P0511006.D
Acq: 11 May 2005 5:07 pm

Tgt Ion: 64 Resp: 6052
Ion Ratio Lower Upper
64 100
96 114.5 72.7 172.7

1,4-DIOXANE

Concen: 0.85 µg/L
RT: 12.43 min Scan# 873
Delta R.T. -0.00 min
Lab File: P0511006.D
Acq: 11 May 2005 5:07 pm

Tgt Ion: 88 Resp: 388
Ion Ratio Lower Upper
88 100
58 74.3 15.8 115.8
87 9.5 0.0 59.5
Quantitation Report

Data File: \HPCHEM\DATA\051105\P0511007.D
Acq On: 11 May 2005 5:40 pm
Sample: poe0059-02ms1
Misc: 1X 10ML
MS Integration Params: DIOXANE.p
Quant Time: May 12 10:04 2005
Quant Results File: DX031905.RES

Quant Method: \HPCHEM\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 07:49:30 2005
Response via: Initial Calibration
DataAcq Meth: DX031905

Internal Standards | R.T. | QIon | Response | Conc Units | Dev(Min)
1) Pentafluorobenzene (IS) | 10.56 | 99 | 37664 | 1.00 ug/L | 0.00
2) 1,4-DIOXANE-d8 | 12.35 | 64 | 6138 | 25.00 ug/L | 0.00
3) 1,2,3-Trichloropropane-d5 | 0.00 | 79 | 0 | 0.00 ug/L | -15.08

System Monitoring Compounds
2) Dibromofluoromethane (SU1) | 10.07 | 113 | 28032 | 0.98 ug/L | 8.00
Spiked Amount | 1.000 | Range 80 - 120 | Recovery = 98.00%

Target Compounds
4) 1,4-DIOXANE | 12.43 | 88 | 4817 | 10.37 ug/L | Qvalue

((#) = qualifier out of range (m) = manual integration

P0511007.D DX031905.M Thu May 12 10:04:20 2005 GCMS1
Quantitation Report  (QT Reviewed)

Data File : D:\HPCHEM\1\DATA\051105\P0511008.D  
Acq On :  11 May 2005  6:13 pm  
Sample : poe0059-02msd1  
Misc :  1X 10ML  
MS Integration Params: DIOXANE.P  
Quant Time: May 12 10:04 2005  
Quant Results File: DX031905.RES

Vial: 8  
Operator: cs  
Inst : GCMS1  
Multiplier: 1.00

Quant Method : D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)  
Title : 8260  1,4-Dioxane Ini. Cal. (05/02/02)  
Last Update : Mon Mar 21 07:49:30 2005  
Response via : Initial Calibration  
DataAcq Meth : DX031905

<table>
<thead>
<tr>
<th>Internal Standards</th>
<th>R.T.</th>
<th>Qion</th>
<th>Response</th>
<th>Conc Units</th>
<th>Dev(Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Pentafluorobenzene (IS)</td>
<td>10.56</td>
<td>99</td>
<td>39551</td>
<td>1.00 ug/L</td>
<td>0.00</td>
</tr>
<tr>
<td>3) 1,4-DIOXANE-d8</td>
<td>12.35</td>
<td>64</td>
<td>7597</td>
<td>25.00 ug/L</td>
<td>0.00</td>
</tr>
<tr>
<td>5) 1,2,3-Trichloropropane-d5</td>
<td>0.00</td>
<td>79</td>
<td>0</td>
<td>0.00 ug/L</td>
<td>-15.08</td>
</tr>
</tbody>
</table>

System Monitoring Compounds
2) Dibromofluoromethane (SU1)  10.07  113  30374  1.02 ug/L  0.00
Spiked Amount  1.000  Range 80 - 120  Recovery = 102.00%

Target Compounds
4) 1,4-DIOXANE  12.43  88  5777  10.05 ug/L  98

(#)= qualifier out of range  (m) = manual integration
Quantitation Report

Data File: D:\HPCHEM\1\DATA\051105\P0511012.D
Acq On: 11 May 2005 8:24 pm
Sample: poe0151-01
Misc: 10mL Dioxane, 5-12-05
MS Integration Params: DIOXANE.P
Quant Time: May 12 10:11 2005

Vial: 12
Operator: cs
Inst: GCMS1
Multiplier: 1.00

Quant Results File: DX031905.RES

Quant Method: D:\HPCHEM\1\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 07:49:30 2005
Response via: Initial Calibration
DataAcq Meth: DX031905

<table>
<thead>
<tr>
<th>Internal Standards</th>
<th>R.T. (min)</th>
<th>QIon</th>
<th>Response</th>
<th>Conc (ug/L)</th>
<th>Units</th>
<th>Dev (Min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1) Pentfluorobenzene (IS)</td>
<td>10.56</td>
<td>99</td>
<td>34886</td>
<td>1.00</td>
<td>ug/L</td>
<td>0.00</td>
</tr>
<tr>
<td>3) 1,4-DIOXANE-d8</td>
<td>12.35</td>
<td>64</td>
<td>6295</td>
<td>25.00</td>
<td>ug/L</td>
<td>0.00</td>
</tr>
<tr>
<td>5) 1,2,3-Trichloropropane-d5</td>
<td>0.00</td>
<td>79</td>
<td>0</td>
<td>0.00</td>
<td>ug/L</td>
<td>-15.08</td>
</tr>
</tbody>
</table>

System Monitoring Compounds
2) Dibromofluoromethane (SU1) 10.07 113 26643 1.01 ug/L 0.00
Spiked Amount 1.000 Range 80 - 120 Recovery = 101.00%

Target Compounds
4) 1,4-DIOXANE 12.43 88 177 0.37 ug/L

Qvalue

(#) = qualifier out of range (m) = manual integration

P0511012.D DX031905.M Thu May 12 10:11:33 2005 GCMS1

Page 73
Quantitation Report

Data File: D:\HPCHEM\DATA\051105\P0511012.D
Vial: 12
Acq On: 11 May 2005  8:24 pm
Sample: pso0151-01
Operator: cs
Misc: 1X 10ML
Inst: GCMS1
Multipl: 1.00
MS Integration Params: DIOXANE.P
Quant Time: May 12 10:11 2005
Quant Results File: DX031905.RES

Method: D:\HPCHEM\METHODS\DX031905.M (RTE Integrator)
Title: 8260 1,4-Dioxane Ini. Cal. (05/02/02)
Last Update: Mon Mar 21 07:49:30 2005
Response via: Initial Calibration

TIC P0511012.D

Time-->  8.00  9.00  10.00  11.00  12.00  13.00  14.00  15.00  16.00  17.00  18.00  19.00  20.00  21.00  22.00  23.00

P0511012.D DX031905.M Thu May 12 10:11:33 2005 GCMS1
#1
Pentafluorobenzene (IS)
Concen: 1.00 ug/L
RT: 10.56 min Scan# 595
Delta R.T. -0.01 min
Lab File: P0511012.D
Acq: 11 May 2005 8:24 pm
Tgt Ion: 99 Resp: 34886
Ion Ratio Lower Upper
99 100
137 23.8 3.8 43.8

#2
Dibromofluoromethane (SU1)
Concen: 1.00 ug/L
RT: 10.07 min Scan# 511
Delta R.T. -0.00 min
Lab File: P0511012.D
Acq: 11 May 2005 8:24 pm
Tgt Ion: 113 Resp: 26643
<table>
<thead>
<tr>
<th>Lab Number</th>
<th>Initial (ml)</th>
<th>Final (ml)</th>
<th>Surrogate 1</th>
<th>Spike 1</th>
<th>Spike 2</th>
<th>Surrogate 2</th>
<th>Spike 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>PE1128-BLKI</td>
<td>05/11/05 00:00</td>
<td>10</td>
<td>10</td>
<td>POE009-01</td>
<td>10</td>
<td>POE009-02</td>
<td>10</td>
</tr>
<tr>
<td>PE1128-BB1</td>
<td>05/11/05 00:00</td>
<td>10</td>
<td>10</td>
<td>POE009-01</td>
<td>10</td>
<td>POE009-02</td>
<td>10</td>
</tr>
<tr>
<td>PE1128-MM3</td>
<td>05/11/05 00:00</td>
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<td>POE009-01</td>
<td>10</td>
<td>POE009-02</td>
<td>10</td>
</tr>
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</table>

Matrix: Water

Prepared using: GCMS - EPA 5050 GCMS
Prepared using: GCMS - EPA 5050 GCMS
Prepared using: GCMS - EPA 5050 GCMS
Prepared using: GCMS - EPA 5050 GCMS
Analytical Standard Record
Del Mar Analytical - Phoenix
5050010

Description: 1,4-Dioxane SSC 10 ppm
Standard Type: Analyte Spike
Solvent: MeOH #44337
Final Volume (mls): 1
Vials: 1
Expires: 06/02/05
Prepared: 05/02/05
Prepared By: Corey Schrader
Department: GCMS
Last Edit: 05/02/05 11:59 by cs

1,4-Dioxane SSC 10ppm

<table>
<thead>
<tr>
<th>Analyte</th>
<th>CAS Number</th>
<th>Concentration (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,4-Dioxane</td>
<td>123-91-1</td>
<td>10</td>
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</tbody>
</table>

Parent Standards used in this standard

<table>
<thead>
<tr>
<th>Standard</th>
<th>Description</th>
<th>Prepared</th>
<th>Prepared By</th>
<th>Expires</th>
<th>Last Edit</th>
<th>Amount (mls)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5050008</td>
<td>1,4-Dioxane SS 2000 ppm STOCK</td>
<td>05/02/05</td>
<td>Corey Schrader</td>
<td>06/02/05</td>
<td>05/02/05 11:41 by c</td>
<td>0.005</td>
</tr>
</tbody>
</table>

Elizabeth Wueschner
Review By

05-11-2005
Date
## Analytical Standard Record

**Del Mar Analytical - Phoenix**

**5050008**

<table>
<thead>
<tr>
<th>Description</th>
<th>1,4-Dioxane SS 2000 ppm STOCK</th>
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<tbody>
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<td>Other Solution</td>
<td>Prepared:</td>
<td>05/02/05</td>
</tr>
<tr>
<td>Solvent:</td>
<td>MeOH</td>
<td>Prepared By:</td>
<td>Corey Schrader</td>
</tr>
<tr>
<td>Final Volume (mLs):</td>
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<td>Department:</td>
<td>GCMS</td>
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<tr>
<td>Vials:</td>
<td>1</td>
<td>Last Edit:</td>
<td>05/02/05 11:41 by cs</td>
</tr>
</tbody>
</table>

O2SI, 1,4-Dioxane 2000 ppm in Methanol PART#020223-01 LOT#109885
CRACKED NEW AMPULE -- original log in #5010214

<table>
<thead>
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<th>Analyte</th>
<th>CAS Number</th>
<th>Concentration (ppm)</th>
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<td>1,4-Dioxane</td>
<td>123-91-1</td>
<td>2000</td>
</tr>
</tbody>
</table>

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Elizabeth Wueschner 05-11-2005
Reviewed By          Date
Analytical Standard Record
Del Mar Analytical - Phoenix
5050011

<table>
<thead>
<tr>
<th>Description:</th>
<th>IS/SURR MIX DIOXANE250/10/10PPM</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Standard Type:</td>
<td>Surrogate Spike</td>
<td>Prepared:</td>
<td>05/02/05</td>
</tr>
<tr>
<td>Solvent:</td>
<td>MeOH/EMD#44337</td>
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<td>Corey Schrader</td>
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<td>Final Volume (mls):</td>
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<td>Department:</td>
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<tr>
<td>Vials:</td>
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<td>Last Edit:</td>
<td>05/02/05 12:01 by cs</td>
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</tbody>
</table>

IS/SURR MIX for 1,4-Dioxane: 1,4-Dioxane-d8 at 250 ppm, Pentfluorobenzene at 10 ppm, Dibromofluoromethane at 10 ppm

<table>
<thead>
<tr>
<th>Analyte</th>
<th>CAS Number</th>
<th>Concentration (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,4-Dichlorobenzene d4</td>
<td>3855-82-1</td>
<td>10</td>
</tr>
<tr>
<td>1,4-Difluorobenzene</td>
<td>540-36-3</td>
<td>10</td>
</tr>
<tr>
<td>1,4-Dioxane-d8</td>
<td>17647-74-4</td>
<td>250</td>
</tr>
<tr>
<td>4-Bromofluorobenzene</td>
<td>460-00-4</td>
<td>10</td>
</tr>
<tr>
<td>Chlorobenzene-d5</td>
<td>3114-55-4</td>
<td>10</td>
</tr>
<tr>
<td>Dibromofluoromethane</td>
<td>1868-53-7</td>
<td>10</td>
</tr>
<tr>
<td>Pentafluorobenzene</td>
<td>NA</td>
<td>10</td>
</tr>
<tr>
<td>Toluene-d8</td>
<td>2037-26-5</td>
<td>10</td>
</tr>
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</table>

Parent Standards used in this standard

<table>
<thead>
<tr>
<th>Standard</th>
<th>Description</th>
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<th>Prepared By</th>
<th>Expires</th>
<th>Last Edit</th>
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<td>5040415</td>
<td>8260 Surr.2000PPM</td>
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<td>Melissa Spencer</td>
<td>05/25/05</td>
<td>04/25/05 11:50 by ²</td>
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<td>5040458</td>
<td>8260 Internal Standard</td>
<td>04/27/05</td>
<td>Melissa Spencer</td>
<td>05/27/05</td>
<td>04/27/05 09:35 by ³</td>
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<td>5050009</td>
<td>1,4-Dioxane-d8 10000 PPB</td>
<td>05/02/05</td>
<td>Corey Schrader</td>
<td>06/02/05</td>
<td>05/02/05 11:42 by ²</td>
<td>0.025</td>
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Elizabeth Wueschner  
05-11-2005
Reviewed By
Date
# Analytical Standard Record

**Del Mar Analytical - Phoenix**

5040415

<table>
<thead>
<tr>
<th>Description:</th>
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<td>Surrogate Spike</td>
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<td>Solvent:</td>
<td>MEOH</td>
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<tr>
<td>Final Volume (mls):</td>
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<tr>
<td>Vials:</td>
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</tr>
<tr>
<td>Expires:</td>
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</tr>
<tr>
<td>Prepared:</td>
<td>04/25/05</td>
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<tr>
<td>Prepared By:</td>
<td>Melissa Spencer</td>
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<td>Department:</td>
<td>GCMS</td>
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<tr>
<td>Last Edit:</td>
<td>04/25/05 11:50 by MS</td>
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ABSOLUTE, PART#21002, LOT#060304, 3 COMP @ 2000 ug/mL
CRACKED NEW AMPULE--original log in #5020381

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<tr>
<th>Analyte</th>
<th>CAS Number</th>
<th>Concentration (ppm)</th>
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<tbody>
<tr>
<td>4-Bromofluorobenzene</td>
<td>460-00-4</td>
<td>2000</td>
</tr>
<tr>
<td>Dibromofluoromethane</td>
<td>1868-53-7</td>
<td>2000</td>
</tr>
<tr>
<td>Toluene-d8</td>
<td>2037-26-5</td>
<td>2000</td>
</tr>
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</table>

Reviewed By

Date
### Analytical Standard Record

**Del Mar Analytical - Phoenix**  
**5040458**

<table>
<thead>
<tr>
<th>Description</th>
<th>8260 INTERNAL STANDARD</th>
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<td>Other Solution</td>
<td>Prepared:</td>
<td>04/27/05</td>
</tr>
<tr>
<td>Solvent:</td>
<td>N/A</td>
<td>Prepared By:</td>
<td>Melissa Spencer</td>
</tr>
<tr>
<td>Final Volume (mLs):</td>
<td>1</td>
<td>Department:</td>
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<td>Vials:</td>
<td>1</td>
<td>Last Edit:</td>
<td>04/27/05 09:35 by MS</td>
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</tbody>
</table>

Absolute PART#20013, LOT#081604, 2000PPM  
CRACKED NEW AMPULE--ORIGINAL LOG-IN ID#4120170

<table>
<thead>
<tr>
<th>Analyte</th>
<th>CAS Number</th>
<th>Concentration (ppm)</th>
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<tbody>
<tr>
<td>1,4-Dichlorobenzene d4</td>
<td>3855-82-1</td>
<td>2000</td>
</tr>
<tr>
<td>1,4-Difluorobenzene</td>
<td>540-36-3</td>
<td>2000</td>
</tr>
<tr>
<td>Chlorobenzene-d5</td>
<td>3114-55-4</td>
<td>2000</td>
</tr>
<tr>
<td>Pentafluorobenzene</td>
<td>NA</td>
<td>2000</td>
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Reviewed By:  
Date:
**Analytical Standard Record**

**Del Mar Analytical - Phoenix**

5050009

<table>
<thead>
<tr>
<th>Description:</th>
<th>1,4-Dioxane-d8 10000 PPB</th>
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<tbody>
<tr>
<td>Standard Type:</td>
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<td>Vials:</td>
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</tr>
<tr>
<td>Prepared:</td>
<td>05/02/05</td>
</tr>
<tr>
<td>Prepared By:</td>
<td>Corey Schrader</td>
</tr>
<tr>
<td>Department:</td>
<td>GCMS</td>
</tr>
<tr>
<td>Last Edit:</td>
<td>05/02/05 11:42 by cs</td>
</tr>
</tbody>
</table>

Absolute Part# 92785, Lot# 022301, 1,4-Dioxane-d8, 10mg/mL in methanol

ORIGINAL LOG-IN ID#5010501

<table>
<thead>
<tr>
<th>Analyte</th>
<th>CAS Number</th>
<th>Concentration (ppm)</th>
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<tr>
<td>1,4-Dioxane-d8</td>
<td>17647-74-4</td>
<td>10000</td>
</tr>
</tbody>
</table>

Elizabeth Wueschner 05-11-2005
Reviewed By Date
### ACTION ITEMS

1. **Case Narrative**
   - Deficiencies
   - 

2. **Out of Scope**
   - Analyses
   - 

3. **Analyses Not Conducted**
   - 

4. **Missing Hardcopy Deliverables**
   - 

5. **Incorrect Hardcopy Deliverables**
   - 

6. **Deviations from Analysis Protocol, e.g.,**
   - Holding Times
   - GC/MS Tune/Inst. Perform
   - Calibrations
   - Blanks
   - Surrogates
   - Matrix Spike/Dup LCS
   - Field QC
   - Internal Standard Performance
   - Compound Identification and Quantitation
   - System Performance

### COMMENTS
- Acceptable as reviewed.

---

* Subcontracted analytical laboratory is not meeting contract and/or method requirements.
* Differences in protocol have been adopted by the laboratory but no action against the laboratory is required.
DATA VALIDATION REPORT

NPDES Monitoring

ANALYSIS: SEMIVOLATILES

SAMPLE DELIVERY GROUP: IOE0230

Prepared by

AMEC Denver Operations
550 South Wadsworth Boulevard, Suite 500
Lakewood, Colorado 80226
1. INTRODUCTION

Task Order Title: NPDES Monitoring  
Contract Task Order #: 3131500110  
SDG#: IOE0230  
Project Manager: B. McIlvaine  
Matrix: Water  
Analysis: Semivolatiles  
QC Level: Level IV  
No. of Samples: 1  
No. of Reanalyses/Dilutions: 0  
Reviewer: M. Pokorny  
Date of Review: June 21, 2005

The samples listed in Table 1 were validated based on the guidelines outlined in the AMEC Data Validation Procedure for Levels C and D Semivolatile Organics (DVP-3, Rev. 2), EPA Method 625, and the National Functional Guidelines For Organic Data Review (2/94). Any deviations from these procedures are documented herein. Qualifiers were applied in cases where the data did not meet the required QC criteria or where special consideration by the data user is required. Data qualifiers were placed on Form Is with the associated qualification codes. Analytes that were rejected for any reason are denoted on the Form I as having only the “R” data qualifier and associated qualification code(s) denoting the reason for rejection. Any additional problems with the data that may have resulted in an estimated value were not denoted by a qualification code since the data had already been rejected.
Table 1. Sample Identification

<table>
<thead>
<tr>
<th>Client ID</th>
<th>EPA ID</th>
<th>Lab No.</th>
<th>Matrix</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outfall 012</td>
<td>Outfall 012</td>
<td>IOE0230-01</td>
<td>water</td>
<td>625</td>
</tr>
</tbody>
</table>
2. DATA VALIDATION FINDINGS

2.1 SAMPLE MANAGEMENT

The sample in this SDG was received at the laboratory within the temperature limits of 4°C ±2°C. The analysis did not require preservation, and no preservation was noted in the field. The COC noted that the sample was received intact. No qualifications were required.

2.1.2 Chain of Custody

The COC was signed and dated by both field and laboratory personnel. The COC accounted for the analysis presented in this SDG. As the sample was couriered directly to the laboratory, custody seals were not required. No qualifications were required.

2.1.3 Holding Times

The water sample was extracted within seven days of collection and analyzed within 40 days of collection. No qualifications were required.

2.2 GC/MS TUNING

The DFTPP tunes met the criteria specified in Method 625, and the sample was analyzed within 12 hours of the DFTPP injection time. No qualifications were required.

2.3 CALIBRATION

The initial calibration associated with this SDG was dated 05/04/05. The average RRFs were ≥0.05 and the %RSDs were ≤35% or $r^2$ ≥0.995 for both target compounds listed on the sample summary form. A representative number of average RRFs and %RSDs were checked from the raw data, and no calculation or transcription errors were noted. The continuing calibration associated with the sample analysis was analyzed 05/09/05. The RRFs for both target compounds were ≥0.05, and the %Ds were ≤20%. A representative number of RRFs, $r^2$ values, and %Ds were checked from the raw data, and no calculation or transcription errors were noted. No qualifications were required.

2.4 BLANKS

One method blank (5E05051-BLK1) was extracted and analyzed with this SDG. No target compounds were reported in the method blank. Review of the raw data indicated no false negatives. No qualifications were required.

2.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

One blank spike/blank spike duplicate pair (5E05051-BS1/5E05051-BSD1) was extracted and analyzed with this SDG. All percent recoveries and RPDs were within the laboratory QC limits. A
representative number of recoveries and RPDs were calculated from the raw data and no calculation or transcription errors were found. No qualifications were required.

2.6 SURROGATE RECOVERY

The sample surrogate recoveries were within the laboratory QC limits. A representative number of recoveries were calculated from the raw data, and no transcription or calculation errors were noted. No qualifications were required.

2.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

No MS/MSD analyses were associated with this SDG. Evaluation of method accuracy and precision was based on blank spike/blank spike duplicate results. No qualifications were required.

2.8 FIELD QC SAMPLES

Field QC samples were evaluated, and if necessary, qualified based on method blanks and other laboratory QC results affecting the usability of the field QC data. Any remaining detects were used to evaluate the associated site sample. Following are findings associated with field QC samples:

2.8.1 Field Blanks and Equipment Rinsates

There were no field QC samples associated with this SDG. No qualifications were required.

2.8.2 Field Duplicates

There were no field duplicate samples associated with this SDG. No qualifications were required.

2.9 INTERNAL STANDARDS PERFORMANCE

The internal standard area counts and retention times were within the control limits established by the continuing calibration standards: -50%/+100% for internal standard areas and ±30 seconds for retention times. A representative number of recoveries were checked from the raw data, and no transcription or calculation errors were noted. No qualifications were required.

2.10 COMPOUND IDENTIFICATION

The laboratory analyzed for naphthalene and n-nitrosodimethylamine by EPA Method 625. Review of the sample chromatogram, retention times, and spectra indicated no problems with target compound identification. No qualifications were required.
2.11 COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS

Compound quantification is verified at a Level IV data validation. No calculation or transcription errors were found. The reporting limits were supported by the low level of the initial and the method detection limit study. No qualifications were required.

2.12 TENTATIVELY IDENTIFIED COMPOUNDS

TICs were not reported by the laboratory for this SDG. No qualifications were required.

2.13 SYSTEM PERFORMANCE

Review of the raw data indicated no problems with system performance. No qualifications were required.
# DRAFT: ACID & BASE/NEUTRALS BY GC/MS (EPA 625)

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>Batch</th>
<th>Reporting Limit</th>
<th>Sample Result</th>
<th>Dilution Factor</th>
<th>Date</th>
<th>Data Analyzed Qualifiers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample ID: JOE0230-01 (DRAFT: Outfall 012 - Water) Reporting Units: µg/l</td>
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<td></td>
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<td></td>
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<tr>
<td>Naphthalene</td>
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<td>05/05/05</td>
<td></td>
</tr>
<tr>
<td>N-Nitrosodimethylamine</td>
<td>EPA 625</td>
<td>SE05051</td>
<td>3.7</td>
<td>23</td>
<td>0.962</td>
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<td>(30-120%)</td>
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<tr>
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<td></td>
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<tr>
<td>Surrogate: 2,4,6-Trichlorophenol (43-120%)</td>
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<td></td>
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<td>Surrogate: Nitrobenzene-d5 (45-120%)</td>
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<tr>
<td>Surrogate: 2-Fluorobiphenyl (45-120%)</td>
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<td>Surrogate: Terphenyl-d14 (45-120%)</td>
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<td></td>
</tr>
</tbody>
</table>

Sampled: 05/03/05

**AMEC VALIDATED**

**LEVEL IV**

The results pertain only to the samples tested in the laboratory. This report shall not be reproduced except in full, without written permission from Del Mar Analytical.
## CONTRACT COMPLIANCE SCREENING FORM FOR HARDCOPY DATA

**AMEC Earth & Environmental**
550 South Wadsworth Boulevard
Suite 500
Lakewood, CO 80226

**Package ID** T711TF70
**Task Order** 313150010
**SDG No.** IOE0230

**Laboratory** Del Mar Analytical
**Reviewer** L. Calvin
**Analysis/Method** TFH/Purgeable by Method 8015M

### Date: June 16, 2005

### Action Items

1. **Case Narrative Deficiencies**

2. **Out of Scope Analyses**

3. **Analyses Not Conducted**

4. **Missing Hardcopy Deliverables**

5. **Incorrect Hardcopy Deliverables**

6. **Deviations from Analysis Protocol**
   - e.g., Holding Times
   - GC/MS Tune/Inst. Performance
   - Calibration
   - Method blanks
   - Surrogates
   - Matrix Spike/Dap LCS
   - Field QC
   - Internal Standard Performance
   - Compound Identification
   - Quantitation
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### Comments

Acceptable as reviewed.

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DATA VALIDATION REPORT

NPDES Monitoring

ANALYSIS: TPH/Purgeable

SAMPLE DELIVERY GROUP: IOE0230

Prepared by
AMEC Denver Operations
550 South Wadsworth Boulevard, Suite 500
Lakewood, Colorado 80226
1. INTRODUCTION

Task Order Title: NPDES Monitoring
Contract Task Order #: 313150010
SDG#: IOE0230
Project Manager: B. McIlvaine
Matrix: Water
Analysis: TPH-Purgeable
QC Level: Level IV
No. of Samples: 2
No. of Reanalyses/Dilutions: 0
Reviewer: L. Calvin
Date of Review: June 15, 2005

The samples listed in Table 1 were validated based on the general guidelines outlined in the AMEC Data Validation Procedure for Levels C and D Extractable Total Fuel Hydrocarbons by GC (DVP-8, Rev. 2), USEPA SW-846 Method 8015M, and validation guidelines outlined in the USEPA CLP National Functional Guidelines for Organic Data Review (2/94). Any deviations from these procedures are documented herein. Qualifiers were applied in cases where the data did not meet the required QC criteria or where special consideration by the data user is required. Data qualifiers were placed on Form Is with the associated qualification codes. Analytes that were rejected for any reason are denoted on the Form I as having only the “R” data qualifier and associated qualification code(s) denoting the reason for rejection. Any additional problems with the data that may have resulted in an estimated value were not denoted by a qualification code since the data had already been rejected.
Table 1. Sample identification

<table>
<thead>
<tr>
<th>Client ID</th>
<th>EPA ID</th>
<th>Lab No.</th>
<th>Matrix</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outfall 012</td>
<td>Outfall 012</td>
<td>IOE0230-01</td>
<td>water</td>
<td>8015M/GRO</td>
</tr>
<tr>
<td>Trip Blank</td>
<td>Trip Blank</td>
<td>IOE0230-02</td>
<td>water</td>
<td>8015M/GRO</td>
</tr>
</tbody>
</table>
2. DATA VALIDATION FINDINGS

2.1 SAMPLE MANAGEMENT

The following are findings associated with sample management:

2.1.1 Sample Preservation, Handling, and Transport

The samples in this SDG were received at Del Mar Analytical on ice within the temperature limits of 4°C ±2°C, at 4°C. The Del Mar Analytical case narrative noted that the samples were received intact, and the COC indicated the samples were properly preserved. No qualifications were required.

2.1.2 Chain of Custody

The COC was signed and dated by both field and laboratory personnel. The EFH analysis (rather than the GRO analysis) was requested in error on the COC for the Trip Blank sample. The sample was analyzed correctly. As the samples were couriered directly to the laboratory, custody seals were not required. No qualifications were required.

2.1.3 Holding Times

The water samples were analyzed within 14 days of collection. No qualifications were required.

2.2 CALIBRATION

One gasoline standard initial calibration dated 11/22/04 was associated with the sample analyses. The %RSD for GRO (C4-C12) were within the QC limit of ≤20%. An initial calibration verification (ICV) was not provided in the data package. The %Ds for all CCVs bracketing the sample analyses were within the Method QC limit of ≤15%. The %RSD and %Ds were recalculated from the raw data and no transcription or calculation errors were noted. No qualifications were required.

2.4 METHOD BLANKS

Two water method blanks (5E11043-BLK1 and 5E12047-BLK1) were associated with the sample analyses. GRO (C4-C12) was not detected above the MDL in either method blank. Review of the raw data indicated no false negative results. No qualifications were required.

2.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

Two water method blank spikes (5E11043-BS1 and 5E12047-BS1) were associated with the sample analyses. GRO (C4-C12) was recovered within the laboratory-established QC limits of 70-140%. The recoveries were checked from the raw data, and no calculation or transcription errors were noted. No qualifications were required.
2.6 SURROGATE RECOVERY

The samples were fortified with the surrogate compound 4-bromofluorobenzene (BFB). Surrogate recoveries were within the laboratory-established QC limits of 65-140%. Recoveries were calculated from the raw data and no transcription or calculation errors were noted. No qualifications were required.

2.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD analyses were not performed on the site sample in this SDG. Evaluation of method accuracy was based on the blank spike results. No qualifications were required.

2.8 FIELD QC SAMPLES

Field QC samples are evaluated, and if necessary, qualified based on method blanks and laboratory QC samples for usability. Any remaining detects are used to evaluate the associated samples. The following are findings associated with field QC samples:

2.9.1 Trip Blanks, Field Blanks, and Equipment Rinsates

Sample Trip Blank was the trip blank associated with site sample Outfall 012. GRO (C4-C12) was not detected above the MDL in the trip blank. Review of the raw data indicated no false negative result. There were no field blank or equipment rinsate samples associated with this SDG. No qualifications were required.

2.9.2 Field Duplicates

There were no field duplicate samples in this SDG.

2.10 COMPOUND IDENTIFICATION

The laboratory analyzed for GRO (C4-C12) by Method 8015M. Compound identification is verified at a Level IV validation. Review of chromatograms and retention times indicated no problems with compound identification for the samples in this SDG. No qualifications were required.

2.11 COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS

Compound quantification was verified for this SDG by recalculating any sample detects, blank spike recoveries, and a representative number of surrogate recoveries. Reporting limits were supported by the low level standard of the initial calibration and by the laboratory MDL. The results were reported in mg/L (ppm). No qualifications were required.
## DRAFT: VOLATILE FUEL HYDROCARBONS (EPA 5030/CADHS Mod. 8015)

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>Batch</th>
<th>Reporting Units</th>
<th>MDL</th>
<th>Reporting Limit</th>
<th>Sample Dilution Factor</th>
<th>Result</th>
<th>Date</th>
<th>Extracted Analyzed, Qualifiers</th>
</tr>
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<tbody>
<tr>
<td>Sample ID: IOE0230-01 (DRAFT: Outfall 012 - Water) - cont.</td>
<td></td>
<td></td>
<td>mg/l</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>GRO (C4 - C12)</td>
<td>EPA 8015 Mod.</td>
<td>SE12047</td>
<td>0.50</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td>Surrogate: 4-BFB (FID) (65-140%)</td>
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<td></td>
<td></td>
<td>mg/l</td>
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Sampled: 05/03/05

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114 %

102 %

AMEC VALIDATED 
LEVEL IV
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   Analyses

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Unusual problems found with the data that have been described in Section 2.1, "Data Validation Findings." The number following the asterisk (*) will indicate the subsection where a description of the problem can be found (eg. *1 would indicate a sample was not within temperature limits). Unusual problems found with the data that have been described in Section 2.1, "Data Validation Findings." The number following the asterisk (*) will indicate the subsection where a description of the problem can be found (eg. *1 would indicate a sample was not within temperature limits).
<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Organics</th>
<th>Inorganics</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>The analyte was analyzed for, but was not detected above the reported sample quantitation limit.</td>
<td>The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.</td>
</tr>
<tr>
<td>J</td>
<td>The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.</td>
<td>The associated value is an estimated quantity.</td>
</tr>
<tr>
<td>N</td>
<td>The analysis indicates the presence of an analyte for which there is presumptive evidence to make a &quot;tentative identification.&quot;</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>NJ</td>
<td>The analysis indicates the presence of an analyte that has been &quot;tentatively identified&quot; and the associated numerical value represents its approximate concentration.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>UJ</td>
<td>The analyte was not deemed above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.</td>
<td>The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.</td>
</tr>
<tr>
<td>R</td>
<td>The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.</td>
<td>The data are unusable. (Note: Analyte may or may not be present).</td>
</tr>
</tbody>
</table>
## Qualification Code Reference Table

<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Organics</th>
<th>Inorganics</th>
</tr>
</thead>
<tbody>
<tr>
<td>H</td>
<td>Holding times were exceeded.</td>
<td>Holding times were exceeded.</td>
</tr>
<tr>
<td>S</td>
<td>Surrogate recovery was outside QC limits.</td>
<td>The sequence or number of standards used for the calibration was incorrect. Correlation coefficient is &lt;0.995.</td>
</tr>
<tr>
<td>C</td>
<td>Calibration %RSD or %D were noncompliant.</td>
<td>%R for calibration is not within control limits.</td>
</tr>
<tr>
<td>R</td>
<td>Calibration RRF was &lt;0.05.</td>
<td>Presumed contamination from preparation (method) blank.</td>
</tr>
<tr>
<td>B</td>
<td>Presumed contamination from preparation (method) blank.</td>
<td>Laboratory Blank Spike/Blank Spike Duplicate %R was not within control limits.</td>
</tr>
<tr>
<td>L</td>
<td>Laboratory Blank Spike/Blank Spike Duplicate %R was not within control limits.</td>
<td>MS recovery was poor.</td>
</tr>
<tr>
<td>Q</td>
<td>MS/MSD recovery was poor or RPD high.</td>
<td>Duplicates showed poor agreement.</td>
</tr>
<tr>
<td>E</td>
<td>Not applicable.</td>
<td>ICP ICS results were unsatisfactory.</td>
</tr>
<tr>
<td>I</td>
<td>Internal standard performance was unsatisfactory.</td>
<td>ICP Serial Dilution %D were not within control limits.</td>
</tr>
<tr>
<td>A</td>
<td>Not applicable.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>M</td>
<td>Tuning (BFB or DFTPP) was noncompliant.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>T</td>
<td>Presumed contamination from trip blank.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>+</td>
<td>False positive – reported compound was not present. Not applicable.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>-</td>
<td>False negative – compound was present but not reported.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>F</td>
<td>Presumed contamination from FB, or ER.</td>
<td>Presumed contamination from FB or ER.</td>
</tr>
<tr>
<td>S</td>
<td>Reported result or other information was incorrect.</td>
<td>Reported result or other information was incorrect.</td>
</tr>
<tr>
<td>?</td>
<td>TIC identity or reported retention time has been changed.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>D</td>
<td>The analysis with this flag should not be used because another more technically sound analysis is available.</td>
<td>The analysis with this flag should not be used because another more technically sound analysis is available.</td>
</tr>
<tr>
<td>P</td>
<td>Instrument performance for pesticides was poor.</td>
<td>Post Digestion Spike recovery was not within control limits.</td>
</tr>
<tr>
<td>DNQ</td>
<td>The compound was detected between the MDL and the RL and, by definition, is considered an estimated value.</td>
<td>The compound was detected between the MDL and the RL and, by definition, is considered an estimated value.</td>
</tr>
<tr>
<td>*#, *#</td>
<td>Unusual problems found with the data that have been described in Section 2., &quot;Data Validation Findings.&quot; The number following the asterisk (*) will indicate the subsection where a description of the problem can be found (eg., *1 would indicate a sample was not within temperature limits).</td>
<td>Unusual problems found with the data that have been described in Section 2., &quot;Data Validation Findings.&quot; The number following the asterisk (*) will indicate the subsection where a description of the problem can be found (eg., *1 would indicate a sample was not within temperature limits).</td>
</tr>
</tbody>
</table>
DATA VALIDATION REPORT

NPDES Monitoring

ANALYSIS: TPH/Extractable

SAMPLE DELIVERY GROUP: IOE0230

Prepared by
AMEC Denver Operations
550 South Wadsworth Boulevard, Suite 500
Lakewood, Colorado 80226
1. INTRODUCTION

Task Order Title: NPDES Monitoring
Contract Task Order #: 313150010
SDG#: IOE0230
Project Manager: B. McIlvaine
Matrix: Water
Analysis: TPH-Extractable
QC Level: Level IV
No. of Samples: 1
No. of Reanalyses/Dilutions: 0
Reviewer: L. Calvin
Date of Review: June 15, 2005

The samples listed in Table 1 were validated based on the general guidelines outlined in the AMEC Data Validation Procedure for Levels C and D Extractable Total Fuel Hydrocarbons by GC (DVP-8, Rev. 2), USEPA SW-846 Method 8015B, and validation guidelines outlined in the USEPA CLP National Functional Guidelines for Organic Data Review (2/94). Any deviations from these procedures are documented herein. Qualifiers were applied in cases where the data did not meet the required QC criteria or where special consideration by the data user is required. Data qualifiers were placed on Form Is with the associated qualification codes. Analytes that were rejected for any reason are denoted on the Form I as having only the “R” data qualifier and associated qualification code(s) denoting the reason for rejection. Any additional problems with the data that may have resulted in an estimated value were not denoted by a qualification code since the data had already been rejected.
Table 1. Sample identification

<table>
<thead>
<tr>
<th>Client ID</th>
<th>EPA ID</th>
<th>Lab No.</th>
<th>Matrix</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outfall 012</td>
<td>Outfall 012</td>
<td>IOE0230-01</td>
<td>water</td>
<td>8015B</td>
</tr>
</tbody>
</table>
2. DATA VALIDATION FINDINGS

2.1 SAMPLE MANAGEMENT

The following are findings associated with sample management:

2.1.1 Sample Preservation, Handling, and Transport

The sample in this SDG was received at Del Mar Analytical laboratory on ice within the temperature limits of 4°C ±2°C. The Del Mar Analytical case narrative noted that the sample containers were received intact. No qualifications were required.

2.1.2 Chain of Custody

The COC was signed and dated by both field and laboratory personnel, and accounted for the analysis presented in this SDG. The EFH analysis (rather than the GRO analysis) was requested in error on the COC for the Trip Blank sample. The sample was analyzed correctly. As the site sample was couriered directly to the laboratory, custody seals were not required. No qualifications were required.

2.1.3 Holding Times

The sample was extracted within seven days of sample collection and analyzed within 40 days of extraction. No qualifications were required.

2.2 CALIBRATION

The initial calibration associated with the sample analysis was analyzed on 04/05/05. The %RSD was within the QC limit of ≤20%. The %Ds for the initial calibration verification (ICV) and continuing calibrations associated with the sample analysis were ≤15%. The %RSD and %Ds were recalculated from the raw data and no transcription or calculation errors were noted. No qualifications were required.

2.4 METHOD BLANKS

One method blank (5E06055-BLK1) was extracted and analyzed with the sample in this SDG. EFH (C13-C22) was not present above the MDL in the method blank or in the instrument blank analyzed at the beginning of the analytical sequence. Review of the chromatograms showed no false negatives. No qualifications were required.

2.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

One method blank spike/blank spike duplicate pair (5E06055-BS1/BSD1) was extracted and analyzed with the sample in this SDG. The laboratory reported recoveries of alkane range C13-C28 from spiked diesel. The recoveries were within the laboratory-established QC limits of 40-120%,
and the RPD was within the QC limit of ≤25%. The recoveries and RPD were checked from the raw data, and no calculation or transcription errors were noted. No qualifications were required.

2.6 SURROGATE RECOVERY

The sample was fortified with the surrogate compound n-octacosane. The sample surrogate recovery was within the laboratory-established QC limits of 40-125%. The recovery was calculated from the raw data and no transcription or calculation errors were noted. No qualifications were required.

2.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

There were no MS/MSD analyses associated with the sample of this SDG. Evaluation of method accuracy and precision was based on the BS/BSD results. No qualifications were required.

2.8 FIELD QC SAMPLES

Field QC samples are evaluated, and if necessary, qualified based on method blanks and laboratory QC samples for usability. Any remaining detects are used to evaluate the associated sample. The following are findings associated with field QC samples:

2.9.1 Field Blanks and Equipment Rinsates

There were no field blank or equipment rinseate samples associated with the site sample in this SDG. No qualifications were required.

2.9.2 Field Duplicates

There were no field duplicate samples associated with this SDG.

2.10 COMPOUND IDENTIFICATION

The laboratory analyzed for EFH n-alkane range C13-C22 by EPA SW-846 Method 8015B. Compound identification is verified at a Level IV validation. Review of chromatograms and retention times indicated no problems with compound identification for this SDG. No qualifications were required.

2.11 COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS

Compound quantification was verified for this SDG by recalculating any sample detect, blank spike recoveries, and a representative number of surrogate recoveries. Reporting limits were supported by the low level standard of the initial calibration and by the laboratory MDL. Results were reported in mg/L (ppm). No qualifications were required.
**DRAFT: EXTRACTABLE FUEL HYDROCARBONS (CADHS/8015 Modified)**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>Batch</th>
<th>MDL Limit</th>
<th>Reporting Limit</th>
<th>Sample Dilution</th>
<th>Result</th>
<th>Factor</th>
<th>Extracted</th>
<th>Analyzed</th>
<th>Qualifiers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample: IOE0230-01 (DRAFT: Outfall 012 - Water) - cont.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>EFH (C13 - C22)</td>
<td>EPA 8015B</td>
<td>SE06055</td>
<td>0.082</td>
<td>0.50</td>
<td></td>
<td>0.71</td>
<td>0.971</td>
<td>05/05/05</td>
<td>05/06/05</td>
<td>73 %</td>
</tr>
<tr>
<td>Surrogate: n-Octacosane (40-125%)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Sample: 05/03/05*  

**AMEC VALIDATED**

**LEVEL IV**
## ACTION ITEMS*

1. **Case Narrative Deficiencies**
   
2. **Out of Scope Analyses**
   
3. **Analyses Not Conducted**
   
4. **Missing Hardcopy Deliverables**
   
5. **Incorrect Hardcopy Deliverables**
   
6. **Deviations from Analysis Protocol, e.g.,**
   - Qualifications required for calibration outliers.
   - Holding Times
   - GC/MS Tune/Inst. Perform
   - Calibrations
   - Blanks
   - Surrogates
   - Matrix Spike/Dup LCS
   - Field QC
   - Internal Standard Performance
   - Compound Identification and Quantitation
   - System Performance

## COMMENTS*

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* Subcontracted analytical laboratory is not meeting contract and/or method requirements.

* Differences in protocol have been adopted by the laboratory but no action against the laboratory is required.
DATA VALIDATION REPORT

NPDES Monitoring

ANALYSIS: VOLATILES

SAMPLE DELIVERY GROUP: IOE0230

Prepared by

AMEC Denver Operations
550 South Wadsworth Boulevard, Suite 500
Lakewood, Colorado 80226
1. INTRODUCTION

Task Order Title: NPDES Monitoring
Contract Task Order #: 313150010
SDG#: IOE0230
Project Manager: B. McIlvaine
Matrix: Water
Analysis: Volatiles
QC Level: Level IV
No. of Samples: 2
No. of Reanalyses/Dilutions: 0
Reviewer: M. Pokorny
Date of Review: June 21, 2005

The samples listed in Table 1 were validated based on the guidelines outlined in the AMEC Data Validation Procedure for Levels C and D Volatile Organics (DVP-2, Rev. 2), EPA Method 624 and the National Functional Guidelines For Organic Data Review (2/94). Any deviations from these procedures are documented herein. Qualifiers were applied in cases where the data did not meet the required QC criteria or where special consideration by the data user is required. Data qualifiers were placed on Form Is with the associated qualification codes. Analytes that were rejected for any reason are denoted on the summary forms as having only the “R” data qualifier and associated qualification code(s) denoting the reason for rejection. Any additional problems with the data that may have resulted in an estimated value were not denoted by a qualification code since the data had already been rejected.
Table 1. Sample identification

<table>
<thead>
<tr>
<th>Client ID</th>
<th>EPA ID</th>
<th>Lab No.</th>
<th>Matrix</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outfall 012</td>
<td>Outfall 012</td>
<td>IOE0230-01</td>
<td>water</td>
<td>624</td>
</tr>
<tr>
<td>Trip Blank</td>
<td>Trip Blank</td>
<td>IOE0230-02</td>
<td>water</td>
<td>624</td>
</tr>
</tbody>
</table>
2. DATA VALIDATION FINDINGS

2.1 SAMPLE MANAGEMENT

The following are findings associated with sample management:

2.1.1 Sample Preservation, Handling, and Transport

The samples in this SDG were received at the laboratory within the temperature limits of 4°C ±2°C. The samples were properly preserved. The COC noted that the samples were received intact; however, information regarding absence of headspace was not provided. No qualifications were required.

2.1.2 Chain of Custody

The COC was signed and dated by both field and laboratory personnel. The COC accounted for the analyses presented in this SDG. As the samples were couriered directly to the laboratory, custody seals were not required. No qualifications were required.

2.1.3 Holding Times

The samples were analyzed within 14 days of collection. No qualifications were required.

2.2 GC/MS TUNING

The ion abundance windows shown on the quantitation reports were consistent with those specified in EPA Method 624, and all ion abundances were within the established windows. The samples and associated QC were analyzed within 12 hours of the BFB injection time. The BFB summary report was verified from the raw data and no discrepancies between the summary report and the raw data were noted. No qualifications were required.

2.3 CALIBRATION

Two initial calibrations dated 03/26/05 and 05/10/05 were associated with this SDG. The average RRFs were ≥0.05 for the target compounds listed on the sample result summaries. The %RSDs were ≤35% for all applicable target compounds. Two continuing calibrations dated 05/09/05 and 05/12/05 were associated with the sample analyses in this SDG. For the continuing calibration dated 05/09/05, the %Ds for all target compounds were ≤20% in the continuing calibration except for the %Ds for MTBE and 1,2,3-trichloropropane. MTBE and 1,2,3-trichloropropane were qualified as estimated nondetects, "UI," in the site sample of this SDG. For the continuing calibration dated 05/12/05, the %Ds for all target compounds were ≤20% in the continuing calibration except for the %D for DIPE. The trip blank required no qualification. The RRFs were ≥0.05 for the target compounds listed on the sample result summaries. A representative number of %RSDs and average RRFs from the initial calibrations, and %Ds and RRFs from the continuing calibrations were recalculated from the raw data, and no calculation or transcription errors were found. No further qualifications were required.
2.4 BLANKS

Two water method blank (5E09023-BLK1 and 5E12005-BLK1) were associated with the sample analyses. There were no detects above the MDLs for the target compounds listed on the sample result summaries. The method blank raw data showed no evidence of false negatives. No qualifications were required.

2.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

Two water blank spikes (5E09023-BS1 and 5E12005-BS1) were associated with the sample analyses. All recoveries were within the laboratory-established QC limits. A representative number of recoveries were recalculated from the raw data and no calculation or transcription errors were found. No qualifications were required.

2.6 SURROGATE RECOVERY

The surrogates were recovered within the QC limits of 80-120% in the samples and associated QC. A representative number of surrogate recoveries were recalculated from the raw data and no calculation or transcription errors were found. No qualifications were required.

2.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

An MS/MSD was not analyzed with this SDG. Method accuracy was evaluated based on blank spike results. No qualifications were required.

2.8 FIELD QC SAMPLES

Field QC samples were evaluated, and if necessary, qualified based on method blanks and other laboratory QC results affecting the usability of the field QC data. Any remaining detects were used to evaluate the associated site sample. Following are findings associated with field QC samples:

2.8.1 Trip Blanks

Sample Trip Blank was the trip blank associated with this SDG. There were no target compounds detected above the MDLs in the trip blank. No qualifications were required.

2.8.2 Field Blanks and Equipment Rinsates

There were no field QC samples associated with this SDG. No qualifications were required.

2.8.3 Field Duplicates

There were no field duplicate samples associated with this SDG.
2.9 INTERNAL STANDARDS PERFORMANCE

Internal standard area counts and retention times for the samples in this SDG were within the control limits established by the continuing calibration standards: ±100%/±50% for internal standard areas and ±0.50 minutes for retention times. A representative number of internal standard areas and retention times were verified from the raw data, and no calculation or transcription errors were noted. No qualifications were required.

2.10 COMPOUND IDENTIFICATION

Target compound identification was verified at a Level IV data validation. The laboratory analyzed for five volatile target compounds by EPA Method 624. Chromatograms, retention times, and spectra for the samples and QC were examined and no target compound identification problems were noted. No qualifications were required.

2.11 COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS

Compound quantification is verified at a Level IV data validation. The reporting limits were supported by the lowest concentrations of the initial calibration standard and by the MDL study. As there were no sample detects in this SDG, compound quantitation was verified by recalculating a representative number of blank spike and surrogate recoveries from the raw data. Results were reported in μg/L (ppb). No calculation or transcription errors were noted. No qualifications were required.

2.12 TENTATIVELY IDENTIFIED COMPOUNDS

The laboratory did not provide TICs for this SDG. No qualifications were required.

2.13 SYSTEM PERFORMANCE

A review of the chromatograms and other raw data showed no identifiable problems with system performance. No qualifications were required.
DRAFT: PURGEABLES BY GC/MS (EPA 624)

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>Batch</th>
<th>MDL</th>
<th>Reporting Limit</th>
<th>Sample Dilution Factor</th>
<th>Extracted Date</th>
<th>Sample Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2-Dibromoethane (EDB)</td>
<td>EPA 624</td>
<td>SE09023</td>
<td>0.32</td>
<td>2.0</td>
<td>ND</td>
<td>05/09/05</td>
<td>05/10/05</td>
</tr>
<tr>
<td>Methyl-tet-butyl Ether (MTBE)</td>
<td>EPA 624</td>
<td>SE09023</td>
<td>0.32</td>
<td>5.0</td>
<td>ND</td>
<td>05/09/05</td>
<td>05/10/05</td>
</tr>
<tr>
<td>1,2,3-Trichloropropane</td>
<td>EPA 624</td>
<td>SE09023</td>
<td>0.85</td>
<td>10</td>
<td>ND</td>
<td>05/09/05</td>
<td>05/10/05</td>
</tr>
<tr>
<td>Di-isopropyl Ether (DIPE)</td>
<td>EPA 624</td>
<td>SE09023</td>
<td>0.25</td>
<td>5.0</td>
<td>ND</td>
<td>05/09/05</td>
<td>05/10/05</td>
</tr>
<tr>
<td>tert-Butanol (TBA)</td>
<td>EPA 624</td>
<td>SE09023</td>
<td>3.1</td>
<td>25</td>
<td>ND</td>
<td>05/09/05</td>
<td>05/10/05</td>
</tr>
</tbody>
</table>

Surrogate: Dibromofluoromethane (80-120%)
Surrogate: Toluene-d8 (80-120%)
Surrogate: 4-Bromofluorobenzene (80-120%)

Sample ID: IOE0230-01 (DRAFT: Outfall 012 - Water)
Reporting Units: ug/l

Sample ID: IOE0230-02 (DRAFT: Trip Blank - Water)

AMERICAN ENVIRONMENTAL CORPORATION

DRAFT REPORT
DRAFT REPORT
DATA SUBJECT TO CHANGE

The results pertain only to the sample tested in the laboratory. This report shall not be reproduced except in full, without the written permission from Del Mar Analytical.
CONTRACT COMPLIANCE SCREENING FORM FOR HARDCOPY DATA

AMEC Earth & Environmental
550 South Wadsworth Boulevard
Suite 500
Lakewood, CO 80226

Package ID T711VO110
Task Order 313150010
SDG No. IOE0230

Laboratory Del Mar
Reviewer M. Pokorny
Analysis/Method Volatiles (1,4-dioxane)

No. of Analyses 1
Date: June 21, 2005
Reviewer’s Signature

<table>
<thead>
<tr>
<th>ACTION ITEMS</th>
<th>COMMENTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Case Narrative Deficiencies</td>
<td>Acceptable as reviewed.</td>
</tr>
<tr>
<td>2. Out of Scope Analyses</td>
<td></td>
</tr>
<tr>
<td>3. Analyses Not Conducted</td>
<td></td>
</tr>
<tr>
<td>4. Missing Hardcopy Deliverables</td>
<td></td>
</tr>
<tr>
<td>5. Incorrect Hardcopy Deliverables</td>
<td></td>
</tr>
</tbody>
</table>

* Subcontracted analytical laboratory is not meeting contract and/or method requirements.

b Differences in protocol have been adopted by the laboratory but no action against the laboratory is required.
DATA VALIDATION REPORT

NPDES Monitoring

ANALYSIS: VOLATILES

SAMPLE DELIVERY GROUP: IOE0230

Prepared by

AMEC—Denver Operations
550 South Wadsworth Boulevard, Suite 500
Lakewood, Colorado 80226
1. INTRODUCTION

Task Order Title: NPDES Monitoring  
Contract Task Order #: 313150010  
Sample Delivery Group #: IOE0230  
Project Manager: B. McIlvaine  
Matrix: Water  
Analysis: Volatiles (1,4-dioxane)  
QC Level: Level IV  
No. of Samples: 1  
No. of Reanalyses/Dilutions: 0  
Reviewer: M. Pokorny  
Date of Review: June 21, 2005

The samples listed in Table 1 were validated based on the guidelines outlined in the AMEC Data Validation Procedure for Levels C and D Volatile Organics (DVP-2, Rev. 2), EPA Method SW-846 8260B and the National Functional Guidelines For Organic Data Review (2/94). Any deviations from these procedures and guidelines are documented herein. Qualifiers were applied in cases where the data did not meet the required QC criteria or where special consideration by the data user is required. Data qualifiers were placed on Form Is with the associated qualification codes. Analytes that were rejected for any reason are denoted on the Form I as having only the “R” data qualifier and associated qualification code(s) denoting the reason for rejection. Any additional problems with the data that may have resulted in an estimated value were not denoted by a qualification code since the data had already been rejected.
Table 1. Sample identification

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<tr>
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<th>EPA ID</th>
<th>Lab No.</th>
<th>Matrix</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outfall 012</td>
<td>Outfall 012</td>
<td>IOE0230-01</td>
<td>water</td>
<td>8260B</td>
</tr>
</tbody>
</table>
2. DATA VALIDATION FINDINGS

2.1 SAMPLE MANAGEMENT

Following are findings associated with sample management:

2.1.1 Sample Preservation, Handling, and Transport

The sample in this SDG was received at the Del Mar within the temperature limits of 4°C ±2°C. The sample was subcontracted to Del Mar (Phoenix) for 1,4-dioxane analysis, and the sample was received within the temperature limits of 4°C ±2°C. The sample was properly preserved. The COC and transfer COC noted that the sample was received intact; however, information regarding absence of headspace was not provided. No qualifications were required.

2.1.2 Chain of Custody

The COC and transfer COC were signed by field and laboratory personnel. As the sample was couriered directly to the laboratory from the field, custody seals were not required. According to the transfer COC, there were no custody seals present on the cooler received by Del Mar Analytical in Arizona. No qualifications were required.

2.1.3 Holding Times

The sample was analyzed within 14 days of collection. No qualifications were required.

2.2 GC/MS TUNING

The ion abundance windows were consistent with those specified in EPA Method 8260B. All ion abundances were within the established windows, and the sample was analyzed within 12 hours of the BFB injection time. No qualifications were required.

2.3 CALIBRATION

One initial calibration, dated 03/19/05, was associated with this SDG. The average RRF for 1,4-dioxane was ≥0.05 and the %RSD was ≤35%. The laboratory reported the continuing calibration and the blank spike (P5E1128-BS1) from the same analysis. As the analysis cannot be reported as both a CCV and a blank spike, the reviewer evaluated P5E1128-BS1 as the continuing calibration. The RRF for 1,4-dioxane was ≥0.05; and, the %D was ≤20%. The r² value and average RRF for 1,4-dioxane in the initial calibration, and the %D and RRF for 1,4-dioxane in the continuing calibration were recalculated from the raw data, and no calculation or transcription errors were found. No qualifications were required.
2.4 BLANKS

One water method blank (P5E1128-BLK1) was associated with this SDG. Target compound 1,4-dioxane was not detected above the MDL in the method blank. The method blank raw data showed no evidence of a false negative. No qualifications were required.

2.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

The laboratory analyzed a blank spike/blank spike duplicate pair (P5E1128-BS1/BS1D) with this SDG; however, P5E1128-BS1 was reported as the CCV (see section 2.3); therefore, P5E1128-BS1D was evaluated as a single blank spike. The recovery for 1,4-dioxane was within the QC limits of 70-130%. The recovery was recalculated from the raw data and no calculation or transcription errors were found. No qualifications were required.

2.6 SURROGATE RECOVERY

The sample and QC were fortified with dibromofluoromethane. The surrogate was recovered within the laboratory QC limits of 80-125%. The surrogate recovery for the sample was recalculated from the raw data and no calculation or transcription errors were found. No qualifications were required.

2.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

No MS/MSD analyses were associated with this SDG. Evaluation of method accuracy was based on blank spike results. No qualifications were required.

2.8 FIELD QC SAMPLES

Field QC samples were evaluated, and if necessary, qualified based on method blanks and other laboratory QC results affecting the usability of the field QC data. Any remaining detects were used to evaluate the associated site sample. Following are findings associated with field QC samples:

2.8.1 Trip Blanks

The sample in this SDG had no associated trip blank. No qualifications were required.

2.8.1.1 Field Blanks and Equipment Rinsates

The site sample in this SDG had no associated field QC samples. No qualifications were required.

2.8.2 Field Duplicates

There were no field duplicate samples associated with this SDG.
2.9 INTERNAL STANDARDS PERFORMANCE

Internal standard area counts and retention times for the sample were within the control limits established by the continuing calibration standard: +100%-50% for internal standard areas and ±0.50 minutes for retention times. Internal standard areas and retention times were verified from the raw data, and no calculation or transcription errors were noted. No qualifications were required.

2.10 COMPOUND IDENTIFICATION

Target compound identification was verified at a Level IV data validation. The laboratory analyzed for 1,4-dioxane by Method 8260B/SIM. Chromatograms, retention times, and spectra for the sample and QC were examined and no target compound identification problems were noted. No qualifications were required.

2.11 COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS

Compound quantification is verified at a Level IV data validation. The reporting limit was supported by the lowest concentration of the initial calibration standards and by the undated MDL supplied by the laboratory. Compound quantitation was verified by recalculating blank spike and surrogate recoveries from the raw data. No calculation or transcription errors were noted. No qualifications were required.

2.12 TENTATIVELY IDENTIFIED COMPOUNDS

TICs are not typically reported for SIM methods.

2.13 SYSTEM PERFORMANCE

A review of the chromatograms and other raw data showed no identifiable problems with system performance. No qualifications were required.
## DRAFT: 1,4-DIOXANE BY GC/MS (EPA 5030B/8260B)

### Analyte

<table>
<thead>
<tr>
<th>Sample ID: IOE0230-01 (DRAFT: Outfall 012 - Water) - cont.</th>
<th>Method</th>
<th>Batch</th>
<th>MDL</th>
<th>Reporting Limit</th>
<th>Sample Dilution</th>
<th>Result Factor Extracted</th>
<th>Analyzed Qualifiers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surrogate: Dibromofluoromethane (80-125%)</td>
<td>EPA 8260B</td>
<td>PSE1128</td>
<td>4.9</td>
<td>10</td>
<td>ND</td>
<td>101%</td>
<td></td>
</tr>
</tbody>
</table>
### ACTION ITEMS

1. **Case Narrative Deficiencies**

2. **Out of Scope Analyses**

3. **Analyses Not Conducted**

4. **Missing Hardcopy Deliverables**

5. **Incorrect Hardcopy Deliverables**

6. **Deviations from Analysis Protocol, e.g.,**
   - Holding Times
   - GC/MS Tune/Inst. Performance
   - Calibrations
   - Blanks
   - Surrogates
   - Matrix Spike/Dup LCS
   - Field QC
   - Internal Standard Performance
   - Compound Identification and Quantitation
   - System Performance

### COMMENTS

Acceptable as reviewed.

---

*a Subcontracted analytical laboratory is not meeting contract and/or method requirements.

*b Differences in protocol have been adopted by the laboratory but no action against the laboratory is required.
<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Organics</th>
<th>Inorganics</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>The analyte was analyzed for, but was not detected above the reported sample quantitation limit.</td>
<td>The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.</td>
</tr>
<tr>
<td>J</td>
<td>The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.</td>
<td>The associated value is an estimated quantity.</td>
</tr>
<tr>
<td>N</td>
<td>The analysis indicates the presence of an analyte for which there is presumptive evidence to make a &quot;tentative identification.&quot;</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>NJ</td>
<td>The analysis indicates the presence of an analyte that has been &quot;tentatively identified&quot; and the associated numerical value represents its approximate concentration.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>UJ</td>
<td>The analyte was not deemed above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.</td>
<td>The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.</td>
</tr>
<tr>
<td>R</td>
<td>The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.</td>
<td>The data are unusable. (Note: Analyte may or may not be present).</td>
</tr>
</tbody>
</table>
## Qualification Code Reference Table

<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Organics</th>
<th>Inorganics</th>
</tr>
</thead>
<tbody>
<tr>
<td>H</td>
<td>Holding times were exceeded.</td>
<td>Holding times were exceeded.</td>
</tr>
<tr>
<td>S</td>
<td>Surrogate recovery was outside QC limits.</td>
<td>The sequence or number of standards used for the calibration was incorrect</td>
</tr>
<tr>
<td>C</td>
<td>Calibration %RSD or %D were noncompliant.</td>
<td>Correlation coefficient is &lt;0.995.</td>
</tr>
<tr>
<td>R</td>
<td>Calibration RRF was &lt;0.05.</td>
<td>%R for calibration is not within control limits.</td>
</tr>
<tr>
<td>B</td>
<td>Presumed contamination from preparation (method) blank.</td>
<td>Presumed contamination from preparation (method) or calibration blank.</td>
</tr>
<tr>
<td>L</td>
<td>Laboratory Blank Spike/Blank Spike Duplicate %R was not within control limits.</td>
<td>Laboratory Control Sample %R was not within control limits.</td>
</tr>
<tr>
<td>Q</td>
<td>MS/MSD recovery was poor or RPD high.</td>
<td>MS recovery was poor.</td>
</tr>
<tr>
<td>E</td>
<td>Not applicable.</td>
<td>Duplicates showed poor agreement.</td>
</tr>
<tr>
<td>I</td>
<td>Internal standard performance was unsatisfactory.</td>
<td>ICP ICS results were unsatisfactory.</td>
</tr>
<tr>
<td>A</td>
<td>Not applicable.</td>
<td>ICP Serial Dilution %D were not within control limits.</td>
</tr>
<tr>
<td>M</td>
<td>Tuning (BF or DFTPP) was noncompliant.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>T</td>
<td>Presumed contamination from trip blank.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>+</td>
<td>False positive – reported compound was not present. Not applicable.</td>
<td>False negative – compound was present but not reported.</td>
</tr>
<tr>
<td>-</td>
<td>False negative – compound was present but not reported.</td>
<td>False negative – compound was present but not reported.</td>
</tr>
<tr>
<td>F</td>
<td>Presumed contamination from FB, or ER.</td>
<td>Presumed contamination from FB or ER.</td>
</tr>
<tr>
<td>S</td>
<td>Reported result or other information was incorrect.</td>
<td>Reported result or other information was incorrect.</td>
</tr>
<tr>
<td>?</td>
<td>TIC identity or reported retention time has been changed.</td>
<td>TIC identity or reported retention time has been changed.</td>
</tr>
<tr>
<td>D</td>
<td>The analysis with this flag should not be used because another more technically sound analysis is available.</td>
<td>The analysis with this flag should not be used because another more technically sound analysis is available.</td>
</tr>
<tr>
<td>P</td>
<td>Instrument performance for pesticides was poor.</td>
<td>Post Digestion Spike recovery was not within control limits.</td>
</tr>
<tr>
<td>DNQ</td>
<td>The compound was detected between the MDL and the RL and, by definition, is considered an estimated value.</td>
<td>The compound was detected between the MDL and the RL and, by definition, is considered an estimated value.</td>
</tr>
<tr>
<td>*#</td>
<td>Unusual problems found with the data that have been described in Section 2.9, &quot;Data Validation Findings.&quot; The number following the asterisk (*) will indicate the subsection where a description of the problem can be found (e.g. *1 would indicate a sample was not within temperature limits).</td>
<td>Unusual problems found with the data that have been described in Section 2.9, &quot;Data Validation Findings.&quot; The number following the asterisk (*) will indicate the subsection where a description of the problem can be found (e.g. *1 would indicate a sample was not within temperature limits).</td>
</tr>
</tbody>
</table>
DATA VALIDATION REPORT

NPDES Monitoring

ANALYSIS: PERCHLORATE
SAMPLE DELIVERY GROUP: IOE0230

Prepared by
AMEC—Denver Operations
550 South Wadsworth Boulevard, Suite 500
Lakewood, Colorado 80226
1. INTRODUCTION

Task Order Title: NPDES Monitoring
Contract Task Order #: 313150010
Sample Delivery Group #: IOE0230
Project Manager: B. McIlvaine
Matrix: Water
Analysis: Perchlorate
QC Level: Level IV
No. of Samples: 1
Reviewer: P. Meeks
Date of Review: June 15, 2005

The samples listed in Table 1 was validated based on the guidelines outlined in the AMEC Data Validation Procedures SOP DVP-6, Rev. 2, USEPA Methods for Chemical Analysis of Water and Wastes Method 314.0, and validation guidelines outlined in the USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review (2/94). Any deviations from these procedures and guidelines are documented herein. Qualifiers were applied in cases where the data did not meet the required QC criteria or where special consideration by the data user is required. Data qualifiers were placed on Form Is with the associated qualification codes. Analytes that were rejected for any reason are denoted on the Form I as having only the "R" data qualifier and associated qualification code(s) denoting the reason for rejection. Any additional problems with the data that may have resulted in an estimated value were not denoted by a qualification code since the data had already been rejected.
Table 1. Sample identification

<table>
<thead>
<tr>
<th>Client ID</th>
<th>EPA ID</th>
<th>Laboratory ID</th>
<th>Matrix</th>
<th>COC Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outfall 012</td>
<td>Outfall 012</td>
<td>IOE0230-01</td>
<td>Water</td>
<td>Perchlorate</td>
</tr>
</tbody>
</table>


2. DATA VALIDATION FINDINGS

2.1 SAMPLE MANAGEMENT

Following are findings associated with sample management:

2.1.1 Sample Preservation, Handling, and Transport

The sample in this SDG was received at the laboratory within the temperature limits of 4°C ± 2°C. The analysis did not require preservation and no preservation was noted in the field. No qualifications were required.

2.1.2 Chain of Custody

The COC was signed and dated by field and laboratory personnel, and accounted for the sample and analysis presented in this SDG. No qualifications were required.

2.1.3 Holding Times

The holding time was assessed by comparing the date of collection with the date of analysis. The 28-day analytical holding time for perchlorate was met, and no qualifications were required.

2.2 CALIBRATION

The initial calibration correlation coefficient associated with this SDG was ≥0.995. The IPC-MA recovery was within the control limits of 80-120%. The ICV, CCV, and IPC recoveries were within the control limits of 90-110%. The ICCS was recovered above the control limits at 175%; however, as perchlorate was not detected in the site sample, no qualifications were required.

2.3 BLANKS

The method blank result reported on the summary form and in the raw data for the blank analysis associated with the sample was a nondetect at the reporting limit. No qualifications were required.

2.4 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

The laboratory control sample associated with this SDG was recovered within the method control limits of 85-115%. No qualifications were required.

2.5 SURROGATES RECOVERY

Surrogate recovery is not applicable to the analysis presented in these SDGs.
2.6 LABORATORY DUPLICATES

No MS/MSD or duplicate analyses were performed in association with the sample in this SDG; therefore, no assessment was made with respect to this criterion.

2.7 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

No MS/MSD analyses were performed in association with the sample in this SDG; therefore, no assessment was made with respect to this criterion. Method accuracy was assessed based on the LCS result.

2.8 FURNACE ATOMIC ABSORPTION QC

Furnace atomic absorption was not utilized for the analysis of this sample; therefore, furnace atomic absorption QC is not applicable.

2.9 ICP SERIAL DILUTION

ICP serial dilution is not applicable to the analysis presented in this data validation report.

2.10 SAMPLE RESULT VERIFICATION

A Level IV review was performed for the samples in these data packages. Calculations were verified, and the sample result reported on the Form 1 was verified against the raw data. No transcription errors or calculation errors were noted. No qualifications were required.

2.11 FIELD QC SAMPLES

Field QC samples are evaluated, and if necessary, qualified based only on laboratory blanks. Any remaining detects are used to evaluate the associated sample. The following are findings associated with field QC samples:

2.11.1 Field Blanks and Equipment Rinsates

The sample in this SDG had no associated field QC samples. No qualifications were required.

2.11.2 Field Duplicates

There were no field duplicate pairs associated with this SDG.
### DRAFT: INORGANICS

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Method</th>
<th>Batch Limit</th>
<th>Reporting Limit</th>
<th>Sample Result</th>
<th>Dilution Factor</th>
<th>Extracted Date</th>
<th>Analyzed Date</th>
<th>Qualifiers</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sample ID: IOE0230-01</strong> (DRAFT: Outfall 012 - Water) - cont.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ammonia-N (Distilled)</td>
<td>EPA 350.2</td>
<td>5E05099</td>
<td>0.30</td>
<td>0.50</td>
<td>ND</td>
<td>05/05/05</td>
<td>05/05/05</td>
<td></td>
</tr>
<tr>
<td>Biochemical Oxygen Demand</td>
<td>EPA 405.5</td>
<td>5E04069</td>
<td>0.59</td>
<td>2.0</td>
<td>1.5</td>
<td>05/04/05</td>
<td>05/09/05</td>
<td></td>
</tr>
<tr>
<td>Oil &amp; Grease</td>
<td>EPA 411.1</td>
<td>5E06041</td>
<td>0.94</td>
<td>5.0</td>
<td>ND</td>
<td>05/06/05</td>
<td>05/18/05</td>
<td></td>
</tr>
<tr>
<td>Total Dissolved Solids</td>
<td>SM2540C</td>
<td>5E04104</td>
<td>10</td>
<td>10</td>
<td>250</td>
<td>05/04/05</td>
<td>05/04/05</td>
<td></td>
</tr>
<tr>
<td>Total Suspended Solids</td>
<td>EPA 160.2</td>
<td>5E08025</td>
<td>10</td>
<td>10</td>
<td>11</td>
<td>05/08/05</td>
<td>05/08/05</td>
<td></td>
</tr>
</tbody>
</table>

**Sample ID: IOE0230-01** (DRAFT: Outfall 012 - Water) - cont.

- **Reporting Units:** mg/l
- **Sample ID:** IOE0230-01
- **Method:** EPA 160.5
- **Batch Limit:** 5E05078
- **Reporting Limit:** 0.10
- **Sample Result:** 0.10
- **Dilution Factor:** 1
- **Extracted Date:** 05/05/05
- **Analyzed Date:** 05/05/05

**Sample ID: IOE0230-01** (DRAFT: Outfall 012 - Water) - cont.

- **Reporting Units:** NTU
- **Method:** EPA 180.1
- **Batch Limit:** 5H05095
- **Reporting Limit:** 0.040
- **Sample Result:** 30
- **Dilution Factor:** 1
- **Extracted Date:** 05/05/05
- **Analyzed Date:** 05/05/05

**Sample ID: IOE0230-01** (DRAFT: Outfall 012 - Water) - cont.

- **Reporting Units:** µg/l
- **Method:** EPA 314.0
- **Batch Limit:** 5E10060
- **Reporting Limit:** 0.80
- **Sample Result:** ND
- **Dilution Factor:** 1
- **Extracted Date:** 05/10/05
- **Analyzed Date:** 05/10/05

*Analysis not validated*

---

**AMEC VALIDATED**

**LEVEL IV**

**DRAFT REPORT**
**DRAFT REPORT**
**DATA SUBJECT TO CHANGE**

The results pertain only to the samples tested in the laboratory. This report shall not be reproduced, except in full, without written permission from Del Mar Analytical.

IOE0230 - Page 7 of 23
**CONTRACT COMPLIANCE SCREENING FORM FOR HARDCOPY DATA**

AMEC Earth & Environmental
550 South Wadsworth Boulevard
Suite 500
Lakewood, CO 80226

Laboratory Del Mar
Reviewer P. Meeks
Analysis/Method General Minerals

<table>
<thead>
<tr>
<th>Package ID</th>
<th>T711WC156</th>
</tr>
</thead>
<tbody>
<tr>
<td>Task Order</td>
<td>313150010</td>
</tr>
<tr>
<td>SDG No.</td>
<td>IOE0230</td>
</tr>
</tbody>
</table>

| No. of Analyses | 1 |
| Date:           | 06/15/05 |
| Reviewer's Signature | [Signature] |

### ACTION ITEMS

1. **Case Narrative deficiencies**

2. **Out of Scope Analyses**

3. **Analyses Not Conducted**

4. **Missing Hardcopy Deliverables**

5. **Incorrect Hardcopy Deliverables**

6. **Deviations from Analysis Protocol, e.g.,**
   - Qualifications applied for CCV recovery outliers and detects below the reporting limit.
   - Holding Times
   - GC/MS Tune/Inst. Performance
   - Calibrations
   - Blanks
   - Surrogates
   - Matrix Spike/Dup LCS
   - Field QC
   - Internal Standard Performance
   - Compound Identification and Quantitation
   - System Performance

### COMMENTS

- *Subcontracted analytical laboratory is not meeting contract and/or method requirements.*
- *Differences in protocol have been adopted by the laboratory but no action against the laboratory is required.*
# Data Qualifier Reference Table

<table>
<thead>
<tr>
<th>Qualifier</th>
<th>Organics</th>
<th>Inorganics</th>
</tr>
</thead>
<tbody>
<tr>
<td>U</td>
<td>The analyte was analyzed for, but was not detected above the reported sample quantitation limit.</td>
<td>The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.</td>
</tr>
<tr>
<td>J</td>
<td>The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.</td>
<td>The associated value is an estimated quantity.</td>
</tr>
<tr>
<td>N</td>
<td>The analysis indicates the presence of an analyte for which there is presumptive evidence to make a &quot;tentative identification.&quot;</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>NJ</td>
<td>The analysis indicates the presence of an analyte that has been &quot;tentatively identified&quot; and the associated numerical value represents its approximate concentration.</td>
<td>Not applicable.</td>
</tr>
<tr>
<td>UJ</td>
<td>The analyte was not deemed above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.</td>
<td>The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.</td>
</tr>
<tr>
<td>R</td>
<td>The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.</td>
<td>The data are unusable. (Note: Analyte may or may not be present).</td>
</tr>
</tbody>
</table>