Department of Toxic Substances Control

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MEMORANDUM

TO: Gerard Abrams
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FROM: Fred Seto, Ph.D.
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DATE: September 20, 2004

SUBJECT: Boeing Rocketdyne – Santa Susana Field Laboratory RFI
Review of Lockheed Martin Report on Audit of Data Packages
Columbia Analytical Services: Methods 8270 - Semi-Volatile Organics (SVOC), 8270SIM - Polyaromatic Hydrocarbons (PAHs), 8080 – Polychlorinated Biphenyls (PCBs) and 8082 – PCBs
Pace Analytical: Method 8290 – Dioxin/Furan

The Department of Substances Control (DTSC) has contracted with Lockheed Martin (LM) to perform electronic and hard copy audits of the laboratory data. LM audited the electronic and hard copy data for the following data packages:

SDG	No. of Samples	Sample ID	Method	Laboratory
L9704259	1-Soil	RS001	8270 (SVOC)	Columbia
L9704318	1-Soil	RS592	8270 (SVOC)	Columbia
L9703923	10- Soil	RF262, RF282, RF286 RF287, RF289, RF861 RF866, RF873, RF874 RF877	8270SIM (PAHs)	Columbia

SDG	No. of Samples	Sample ID	Method	Laboratory
L9800196	1-Soil	RS652	8080 (PCBs)	Columbia
L9803443	15 – Soil	RS824 - RS826, RS828, RS830, RS836 – RS845	8082 (PCBs)	Columbia
RJ028	12 – Soil	200546-01 RJ028 200546-02 RJ029 200546-03 RJ030 200546-05 RJ545 200546-06 RJ546 200546-13 RJ032 200546-16 RJ035 200546-17 RJ036 200571-01 RJ553 200571-09 RJ561 200571-11 RJ563 200571-03A RJ555	8290 (Dioxin)	Pace

The Hazardous Materials Laboratory (HML) of DTSC received an audit report. HML has reviewed the LM audit report. We have the following summary/comments:

LM performed the audit of the hard and electronic copy of Methods 8270 - SVOC, 8270SIM – PAHs and 8080/8082 – PCBs with the following approach by using Chemstation as appropriate:

1) The hard copy was compared to the standard EPA Contract Laboratory Program data packages requirements.
2) The electronic copy was reviewed to verify that information provided in the hard copy matched the corresponding information in the electronic form.
3) Where the required information was available for GC/MS analyses, all response factors (RFs), Average RFs, and % RSD for all initial calibration (ICAL) standards and percent difference (%D) values for all continuing calibration standards were verified using LM Software Program (Checkmate).
4) Chromatograms and GC/MS spectra were examined to verify the appropriateness and accuracy of any manually integrated instrument responses.

LM performed the audit of the hard and electronic copy of Methods 8290 – Dioxins with the following approach:

1) The data package was examined to verify the presence of the applicable reporting forms for the method-required standards, instrument checks and quality control (QC) samples, and client sample results; logs documenting standard preparation, sample extraction, and analytical activities; and the raw data from all analyses.

2) The reported data for analytical system performance (e.g. mass spectrometer resolution, GC column resolution); instrument initial and daily calibration verification standards (e.g., retention time (RT) windows, response factors (RFs), PCDD/PCDF isotope ratios); and QC measurements (e.g., internal standard (ISTD), clean-up standard, spike recoveries) were reviewed.

3) Approximately 20% of each type of data was recalculated/verified by the auditor.

LM evaluated, as appropriate, the results of the analytical system performance, tuning, initial calibration (ICAL), continuing calibration verification (CCV), method blanks, surrogates, internal standards, laboratory control samples (LCS), matrix spike/matrix spike duplicates (MS/MSD), retention times and manual integration of some quality control samples and client samples and verified some QC and client sample results. The detailed audit results are summarized in Tables 1 to 6A. The acceptability of the sample results is given in Table 7.

SDG L9704259 – Method 8270 (SVOC) by Columbia

As shown in Table 1, SDG L9704259, ICAL tune hard copy was not provided but the tune from the electronic copy met the ion abundance criteria. Other tunes met the ion abundance criteria.

The ICAL results were within the control limit of 15% RSD except hexachlorocyclopentadiene (15.6%), diethylphthalate (42.1%), 4,6-dinitro-2-methylphenol (15.8%), Carbazole (15.8%) and 2,4-dinitrophenol (26.8%). RF20 and RF40 values reported for diethylphthalate did not match with the RF values calculated by the auditor. Using the calculated RF values, RSD result was 42%. The laboratory reported 16.46% RSD.

The CCV results were within the control limit of 20 %D except for phenol (21%) and di-octylphthalate (23.2%).

The MB showed diethylphthalate 24.78 ug/L (820 ug/kg). Diethylphthalate was reported as non-detect on the analytical report and was noted on the quantitation report as system contaminant. Since diethylphthalate was positively identified and detected above the PQL of 330 ug/kg, its value should be reported.

The LCS and surrogate recoveries were satisfactory.

The MS/MSD recoveries reported for phenol (MS=124%, MSD=130%) did not match with the recoveries calculated by the auditor. The auditor calculated the MS recovery as 144% and MSD recovery as 150%, thus exceeding the control limit of 5% to 112 %. All other MS/MSD recoveries were satisfactory.

The manual integrations performed were examined by the auditor and were determined to be appropriate and accurately performed.

The sample RS001 had a detected diethylphthalate of 12.72 ug/L (460 ug/kg) but was reported non-detect on the analytical report. Since diethylphthalate was positively identified and detected above the PQL of 330 ug/kg, its value should have been reported and flagged as possible contaminant since diethylphthalate in the MB had a value of 24.78 ug/L (820 ug/kg).

SDG L9704318 – Method 8270 (SVOC) by Columbia

As shown in Table 2, SDG L9704318, the electronic copy for the tune was not provided. The tune from the hard copy met ion abundance criteria.

ICAL results were within the control limits of 15% RSD except 2,6-dinitrotoluene (15.94%), 3-nitroaniline (36.5%), 4,6-dinitro-2-methylphenol (17.3%), carbazole (21.7%), 3,3'-dichlorobenzidine (16.1%) and benzo(k)fluoranthene (16.6%)

Manual integrations were examined. The manual integrations of ICAL standards 4-nitrophenol (20 ppm), 2,4-dinitrophenol (80 ppm), 3-nitroaniline (100 ppm) and 2,4-dinitrophenol (100 ppm) were not properly performed.

ICAL RF60 values reported for bis(2-chloroethyl)ether, 4-nitroaniline and carbazole did not match the RF60 values calculated by the auditor.

MB and LCS electronic copy were not provided but results from the hard copy were satisfactory.

MS/MSD electronic copy was not provided. The MS/MSD samples were analyzed on 12/30/97 and 12/31/97 when ICAL and samples were analyzed on 1/8/98. The extraction log was not provided, so the auditor could not verify that MS/MSD samples were extracted in the same batch as the sample RS592. Phenol MS/MSD recoveries (115%, 118%) exceeded the control limits (5-112%).

Surrogate recovery of 2-fluorobiphenyl (118%) exceeded the control limit of 10% to 115%.

Benzo(g,h,i)perylene result was incorrectly reported as 4500 ug/Kg as the auditor calculated the result to be 5400 ug/Kg.

SDG L9703923 – Method 8270SIM (PAHs) by Columbia

As shown in Table 3, the electronic and hard copy tunes associated with ICAL were not provided. Tune analyses associated with daily calibration standards were satisfactory.

ICAL standards manual integrations were found to be appropriate and accurately performed. The RF1.0 value and %RSD reported for benzo(g,h,i)perylene did not match the RF and % RSD calculated by the auditor. ICAL results were within the control limits of 15% RSD except for indeno(1,2,3-c,d)pyrene (19.4%), dibenzo(a,h)anthracene (15.6%) and benzo(g,h,i)perylene (26.3%).

Indeno(1,2,3-c,d)pyrene and benzo[g,h,i]perylene were detected in the sample RF877. Using average RF values reported, the auditor was able to duplicate on-column concentrations for these two compounds. However, the laboratory should have used a regression line fitted to the initial calibration as an alternate procedure to determine extract concentrations of these compounds.

Continuing Calibration Verification, Method Blank, and Laboratory Control Sample results were satisfactory.

MS/MSD (sample RF262) provided appears to be not related to the SDG. No extraction log but the QA/QC report indicated extraction date as 11/25/97, which was different from samples, MB and LCS. MS/MSD electronic copy was not provided. Based on the QA/QC report, MS/MSD results were satisfactory.

SDG L9800196 – Method 8080 (PCBs) by Columbia

ICAL result was satisfactory. The laboratory selected only one peak to report retention time and response for each calibration standard. As per method 8082, the laboratory should have selected a minimum of three peaks.

Continuing Calibration Verification, Method Blank, and Laboratory Control Sample results were satisfactory.

As noted in the MS/MSD QA/QC report, the percent recoveries of Aroclor 1260 were not reported by the laboratory due to the interference from peaks of other target compounds. Surrogate recoveries reported for LCS and MS/MSD could not be verified because the quantitation reports were not provided. But surrogate recovery for sample RS652 was within the control limits.

The electronic files for the sample analyses were not provided for review.

SDG L9803443 – Method 8082 (PCBs) by Columbia

As shown in Table 5, ICAL electronic files were not provided. Hard copy ICAL results were satisfactory. Electronic files for 2 of 7 CCVs were not provided. Sample RS838 was not bracketed by CCV analyses. CCVs percent difference (%D) results were not provided. The auditor calculated %D for 2 peaks, one each for Arochlor 1016 and 1260, from each CCV and the %D results were satisfactory. MB, LCS, MSD/MSD and surrogate results were satisfactory.

Electronic copy of samples RS830, RS838 and RS843 were not provided.

SDG RJ028 – Method 8290 (Dioxin/Furan) by Pace

As shown in Table 6, Mass Spectrometer tune/resolution documentation was not available.

The column performance/resolution could not be verified because some necessary documents were not available.

Initial Calibration results were satisfactory. Results of the daily calibration verification standards (CVS) were mostly satisfactory but some CVS (12 hr,12 min to 14 hr, 45 min) exceeded the allowed 12 hour interval between CVS analyses.

Three method blanks (MB) were prepared: Batch 8/3-4/00, batch 8/21-22/00 and batch 8/7-8/00. For batches 8/3-4/00 and 8/21-22/00, the Relative Ion Abundance ratios for reported identified PCDDs/PCDFs, ISTDs and recovery standards were satisfactory. For batch 8/7-8/00, results ISTDs and recovery standard were not provided and the Relative Ion Abundance ratios exceeded the control limits. The exceeded ratios were flagged with "I" indicating the presence of an interfering compound. They are:

a. 1,2,3,4,6,7,8-HpCDF, ratio = 1.35 (control limits 0.88 -1.20)
b. OCDF, ratio = 1.11 (control limits 0.76 – 1.02)

The MB contained target isomers:

a. Batch 8/3-4/00: OCDD at 2.0 ng/kg (flagged "J", below calibration range).
b. Batch 8/7-8/00: 1,2,3,4,6,7,8-HpCDF, OCDF, OCDD (~36 ng/Kg) and other HpCDD. Amounts were not provided.
c. Batch 8/21-22/00: OCDD at 9.1 ng/Kg (flagged "J")

Only one set of the 3 matrix spike/matrix spike duplicate results was provided. The results were satisfactory.

Laboratory Control Sample results were satisfactory.

Conclusion/Recommendations

For reasons discussed above with the details provided in Tables 1 to 7, the data acceptability for the various SDGs is as follows:

1. The results for SDG L9704259 by Method 8270B (SVOC) should be acceptable except for phenol (330 ug/Kg) which should be qualified as estimate because the associated CCV value exceeded the 20% criterion. The test results higher than the PQL of 330 ug/kg for diethylphthalate should have been reported and flagged to indicate that diethylphthalate was found in the MB.

2. The results for SDG L9704318 by Method 8270B (SVOC) should be acceptable except benzo(k)fluoranthene and carbazole which should be qualified as estimates because the ICAL exceeded the 15% criterion. The reported 4500 ug/kg for benzo(ghi)perylene is incorrect. The correct result is 5400 ug/kg.

3. The results for SDG L9703923 by Method 8270SIM (PAHs) should be acceptable except indeno(123-cd)pyrene (3900 ug/kg) and benzo(g,h,i)perylene (3700 ug/kg) for sample RF877. These results should be qualified as estimates because the ICAL exceeded the 15% criterion. Sample RF262 could not be evaluated as its quantitation report was not available.

4. The results of SDG L9800196 by Method 8082 (PCBs) should be qualified as estimates due to calibration and quantitation using 1 peak only instead of 3 peaks as recommended by the method.

5. The results of SDG L9803443 by Method 8081 (PCBs) should be acceptable.

6. The results for SDG RJ028 by Method 8290 (Dioxin/Furan) should be acceptable/provisionally acceptable except for some compounds. These compounds should be qualified as estimates due to the presence of interferences. They are:

   Sample ID. 200546-101-RJ028: 1,2,3,7,8-PeCDF; 1,2,3,4,7,8-HxCDF 1,2,3,7,8,9-HxCDF; 1,2,3,6,7,8-HxCDF
   Sample ID. 200546-02-RJ029: 1,2,3,6,7,8-HxCDF
   Sample ID. 200546-03-RJ030: 1,2,3,4,6,7,8-HpCDF
   Sample ID. 200546-05-RJ545: 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDD 1,2,3,4,6,7,8-HpCDF; 1,2,3,6,7,8-HxCDF
   Sample ID. 200546-06-RJ546: 2,3,7,8-TCDF; 1,2,3,6,7,8-HxCDF
   Sample ID. 200546-17-RJ036: 2,3,4,7,8-PeCDF; 1,2,3,4,7,8-HxCDF 1,2,3,7,8,9-HxCDD
   Sample ID. 200571-01-RJ553: 1,2,3,4,6,7,8-HpCDD
   Sample ID. 200571-09-RJ561: OCDF

   Some compounds should be qualified as unacceptable due to blank contamination. They are:

   Sample ID. 200546-13-RJ032: Total HpCDD and OCDD
   Sample ID. 200571-01-RJ553 and 200571-09-RJ561: OCDD
   Sample ID. 200571-11-RJ563: 1,2,3,4,6,7,8-HpCDD; Total HpCDD and OCDD

   Some isomers for sample ID. 200546-02-RJ029, 200546-03-RJ030 and 200546-05-RJ546 were inconclusive because the *Detected Peak List* forms were not submitted. The auditor could not verify the ion abundance ratios even though they appear to be acceptable.

If you have any questions, please contact me or Lorna Garcia at (510)540-3003.

CC: Thomas Li, Ph.D.
Cindy Dingman
Lorna Garcia
James Cheng

[redacted]

TABLE 1: SDG L9704259 AUDIT SUMMARY– COLUMBIA ANALYTICAL SERVICES, INC.

<table>
<tr><th>[illegible]</th><th>[illegible] Analyzed 12/11/97 - 12/24/97 Sample ID: [illegible]</th></tr>
<tr><td>DFTPP Tunes</td><td>DFTPP tune analyses were performed, as required, before the first initial calibration standard and before the CCV standard. Since the hard copy for the tune (analyzed on 12/11/1997, at 17:19) associated with ICAL standards was not provided, the Auditor evaluated data only from the electronic file. For the second tune (analyzed on 12/23/1997, at 21:18), both electronic and hard copy data were evaluated. Both tune analyses met the Ion abundance criteria.</td></tr>
<tr><td>Initial Calibration Standards (ICAL)

All System Performance Check Compounds (SPCCs)

Calibration Check Compounds (CCCs)</td><td>
In the analyses of the ICAL standards, manual integrations performed for the following compounds:
<table>
<tr><th>ICAL Standard</th><th>Compound</th></tr>
<tr><td>20,40,60,80 PPM</td><td>2,4-Dinitrophenol, Indeno (1,2,3-c,d) pyrene</td></tr>
<tr><td>100 PPM</td><td>2,4-Dinitrotoluene, Di-Butylphthalate, Indeno(1,2,3-c,d)pyrene</td></tr>
</table>
Chromatograms of the peaks after manual integration were not included with each standard quantitation report. Quantitation reports for all ICAL standards were generated before manual integrations were performed. The manual integration were examined by the auditor and determined to be appropriate, and accurately performed.

SPCCs met minimum RF limits. CCCs met the method requirement of <30% RSD for the RFs of the ICAL standards.

The Auditor was unable to duplicate RF20 and RF40 values reported for diethylphthalate on the Response Factor Report (Page # 2039). The Auditor calculated RF values resulted in the %RSD value of 42.1%, which exceeded 15% method criteria. The laboratory reported %RSD as 16.46%. Comparison of the laboratory reported RF values and the Auditor calculated values is included in the following table:
<table>
<tr><th>Standard Conc.</th><th>Lab Reported RF</th><th>Calculated RF</th><th>Compound Area</th><th>Internal Std Area</th><th>Internal Std</th></tr>
<tr><td>20 PPM</td><td>1.977</td><td>3.310</td><td>7449622</td><td>4501888</td><td>40 PPM</td></tr>
<tr><td>40 PPM</td><td>1.412</td><td>2.044</td><td>9700364</td><td>4746807</td><td>40 PPM</td></tr>
<tr><td>60 PPM</td><td>1.540</td><td>1.540</td><td>9252411</td><td>4004943</td><td>40 PPM</td></tr>
<tr><td>80 PPM</td><td>1.454</td><td>1.454</td><td>11681111</td><td>4016682</td><td>40 PPM</td></tr>
<tr><td>100 PPM</td><td>1.332</td><td>1.332</td><td>16421823</td><td>4932607</td><td>40 PPM</td></tr>
</table>
Four (non-CCC) target compounds exhibited %RSD >15% for the RFs in the ICAL standards. These were hexachlorocyclopentadiene (15.6%), diethylphthalate (42.1%), 4,6-dinitro-2-methylphenol, and Carbazole (15.8%). Also one SPCC compound, 2,4-dinitrophenol (26.8%), exceeded %RSD criteria of 15%.

Diethylphthalate was detected in the method blank at 24.78 ug/L concentration but was reported as a non-detect by the laboratory on the Analytical Report (Page # 2029-R).

Diethylphthalate was also detected in the sample RS001 at 12.72 ug/L concentration but was reported as a non-detect by the laboratory on the Analytical Report (Page # 2031-R).
</td></tr>
<tr><td>Continuing Calibration Verification Standard (CCV)</td><td>
This data package included one CCV standard that was analyzed in the same 12-hour sequence as the client sample RS001.

Two Calibration Check Compounds (CCCs), phenol (21%) and di-octylphthalate (23.2%), did not meet the percent difference (%D) criteria of < 20% as specified in the Method 8270B. Method 8270B states, " After the system performance check is met, CCCs listed in Table 4 are used to check the validity of the initial calibration. If the percent difference for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% drift) for any one CCC, corrective action must be taken." It was not apparent from the data submitted for this SDG that the laboratory took any corrective actions. It is possible that the %D limit for CCC compounds in CCV standard was not applicable to this data as per the project data and measurement quality objectives (DQOs/MQOs). The auditor could not confirm that possibility since the DQOs/MQOs were not available.

Manual Integrations were performed for phenol, 2,4-dinitrophenol, Carbazole, and benzo[g,h,i]perylene. Chromatograms of the peaks after manual integration were not included with the Quantitation Report. On the Quantitation Report only phenol and benzo[g,h,i]perylene compound results were labeled as "m" indicating that their peaks were manually integrated. The manual integrations were examined by the auditor and determined to be appropriate, and accurately performed.
</td></tr>
</table>

[illegible]	Method 8270 (SVOC) Analyzed 12/11/97 - 12/24/97 Sample ID: RS001 1-soil
Method Blanks (MB)	One method blank was extracted and analyzed with this SDG. Diethylphthalate was identified in the MB by the GC/MS system at 24.78 ug/L concentration (equivalent to 820 ug/Kg) but was reported as a non-detect by the laboratory on the Analytical Report (Page # 2029-R). On the Quantitation Report, this compound was labeled by the analyst as System Contaminant. Since diethylphthalate was positively identified and detected above the Practical Quantitation Limit (PQL) of 300 ug/Kg, it should have been reported as a detect.
Laboratory Control Sample (LCS)	One LCS was prepared with the same extraction batch as the method blank and the client sample. All spike compounds and surrogates met the method's percent recovery (%R) criteria.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Sample RS001 was used to prepare the MS/MSD samples. Reported %R values for phenol in MS (124%) and MSD (130%) samples did not match with the ones calculated by the Auditor. Using the data reported on the QA/QC Report, Auditor calculated MS and MSD %R of phenol as 144% and 150% respectively. All 11 spiked compounds, except phenol, met the %R limits as specified in the Method 8270B. Percent recovery of phenol in MS (144%) and MSD (150%) exceeded the upper limit of criteria window (5 - 112%). All 11 spiked compounds met the method's relative percent difference (RPD) limits. Sample results were not affected because LCS analysis met all the method specific requirements.
COC Forms, Sample Receipt, Sample Extraction, Standard, and Instrument Run Logs	Standard Logs were not submitted. Only one page of the MS Instrument Run Log was submitted. This page listed the analyses performed on 12/23/97 and 12/24/97. Instrument run log for the ICAL standards analyzed on 12/11/97 was not included in the data package.
Client Samples	Diethylphthalate was identified in sample RS001 by the GC/MS system at 12.72 ug/L concentration (equivalent to 460 ug/Kg) but was reported as a non-detect by the laboratory on the Analytical Report (Page # 2031-R). On the Quantitation Report, concentration of this compound was crossed out but no initials were found next to this manual editing. Since diethylphthalate was positively identified and detected above the Practical Quantitation Limit (PQL) of 330 ug/Kg, it should have been reported as a detect. Phenol and bis (2-ethylhexyl) phthalate were detected in the sample and their results were reported as 330 ug/Kg and 980 ug/Kg respectively. A copy of the Quantitation Report was submitted only for identification of the phenol peak. Two Tentatively Identified Compounds (TICs) were reported for Sample RS001. On the Analytical Report, estimated concentrations of both TICs were reported as 220 & 250 ug/Kg. It appeared that these results were not reported on the dry basis as indicated on the TIC Analytical Report (page # 2036). Percent solids for RS001 was reported as 92%. The estimated concentrations of the two TICs were calculated by the Auditor as 230 ug/Kg and 270 ug/Kg.
Overview Comments	The data were generated by the Columbia Analytical Services laboratory and reviewed by the auditor using ChemStation software. The laboratory did not specify which revision of method 8270 was used for analysis. Since this data were generated in 1997, the Auditor used method 8270B (September 1994). Hard copy was not provided for DFTPP tune associated with initial calibration standards. However, electronic file for the tune analysis performed on 12/11/1997, at 17:19 was present in the same data folder where ICAL standard files were located. The Auditor was unable to duplicate 20 PPM and 40 PPM standard response factor (RF) values reported for diethylphthalate on the Response Factor Report (Page # 2039). MS/MSD percent recoveries for phenol did not match with the values calculated by the Auditor using the information reported on the MS/MSD Summary QA/QC Report (Page # 2034). Internal Standard (ISTD) Summary sheets, documenting shifts in retention time and changes in compound response (peak area), were not provided.

[redacted]

TABLE 2: SDG L9704318 AUDIT SUMMARY– COLUMBIA ANALYTICAL SERVICES, INC.

<table>
<tr><th>SVOC</th><th>Method 8270 (SVOC) Analyzed 12/30/97 -01/08/98 Sample ID: RS592 [illegible]</th></tr>
<tr><td>DFTPP Tunes</td><td>DFTPP tune analysis was performed, as required, before the first initial calibration standard. Hard copy data was provided for this tune but the electronic file was missing. Tune data reported in the data package met ion abundance criteria as specified in Table 3 of Method 8270B.</td></tr>
<tr><td>Initial Calibration Standards (ICAL)
System Performance Check Compounds (SPCCs)
Calibration Check Compounds (CCCs)</td><td>Manual integrations were performed for the following compounds:
<table>
<tr><th>Compound</th><th>20 PPM</th><th>40 PPM</th><th>60 PPM</th><th>80 PPM</th><th>100 PPM</th></tr>
<tr><td>Bis (2-Chloroethyl)ether</td><td></td><td></td><td>x</td><td></td><td></td></tr>
<tr><td>4-Methylphenol</td><td></td><td></td><td></td><td>x</td><td>x</td></tr>
<tr><td>Benzoic acid</td><td>x</td><td></td><td></td><td></td><td></td></tr>
<tr><td>4-Chloroaniline</td><td></td><td></td><td></td><td>x</td><td></td></tr>
<tr><td>3-Nitroaniline</td><td>x</td><td>x</td><td></td><td>x</td><td>x</td></tr>
<tr><td>2,4-Dinitrophenol</td><td></td><td></td><td></td><td>x</td><td>x</td></tr>
<tr><td>2,4-Dinitrotoluene</td><td></td><td></td><td></td><td></td><td>x</td></tr>
<tr><td>4-Nitrophenol</td><td>x</td><td>x</td><td>x</td><td>x</td><td></td></tr>
<tr><td>Diethylphthalate</td><td></td><td></td><td></td><td></td><td>x</td></tr>
<tr><td>4-Nitroaniline</td><td></td><td>x</td><td>x</td><td>x</td><td></td></tr>
<tr><td>Carbazole</td><td>x</td><td>x</td><td>x</td><td></td><td></td></tr>
<tr><td>Benzo(b)fluoranthene</td><td></td><td></td><td></td><td></td><td>x</td></tr>
<tr><td>Benzo (k) fluoranthene</td><td></td><td></td><td></td><td>x</td><td>x</td></tr>
</table>
Chromatograms of the peaks after manual integration were not included with each standard Quantitation Report. All the compounds mentioned above were correctly identified with "m" on all the Quantitation Reports except for 60 PPM ICAL standard. The Quantitation Reports for 60 PPM ICAL standard did not identify 4-nitroaniline and Carbazole results with "m" identifier. The manual integrations were examined by the auditor. Manual integrations were not properly performed for the following compounds: 20 PPM of 4-Nitrophenol, 80 PPM of 2,4-Dinitrophenol, 100 PPM of 3-Nitroaniline and 2,4-Dinitrophenol.

SPCCs met minimum RF limits, and CCCs met the ICAL method requirement of <30% RSD.

The Auditor was unable to duplicate RF60 values reported for bis(2-chloroethyl)ether, 4-nitroaniline, and Carbazole on the Response Factor Report (Page # 4001 & 4002).
<table>
<tr><th>Compound</th><th>Lab Reported RF</th><th>Calculated RF</th><th>Compound Area</th><th>IS Area</th><th>IS Conc.</th></tr>
<tr><td>Bis(2-chloroethyl)ether</td><td>1.943</td><td>1.660</td><td>1730284</td><td>694707</td><td>40</td></tr>
<tr><td>4-Nitroaniline</td><td>0.159</td><td>0.143</td><td>330128</td><td>1535811</td><td>40</td></tr>
<tr><td>Carbazole</td><td>0..222</td><td>0.201</td><td>739885</td><td>2454066</td><td>40</td></tr>
</table>
Six (non-CCC) target compounds exhibited %RSDs > 15% for the RFs: 2,6-Dinitrotoluene =15.94%, 3-Nitroaniline = 36.5%, 4,6-Dinitro-2-methylphenol = 17.3%, Carbazole = 21.7%, 3,3'-Dichlorobenzidine =16.1%, Benzo(k)fluoranthene = 16.6%. One SPCC compound, 2,4-dinitrophenol (28.7%), did not meet %RSD criteria of <15%.

Benzo(k)fluoranthene and Carbazole were detected in the sample. It was not clear from the hard copy data that the laboratory used different procedures other than average RF, to quantitate results of these compounds. Since the %RSD exceeded the criteria for the ICAL standards, there was no indication that the laboratory used calibration curves to quantitate benzo(k)fluoroanthene and Carbazole concentrations. The reported results for these two compounds should be considered estimated.</td></tr>
<tr><td>Continuing Calibration Verification Standard (CCV)</td><td>CCV standard was not required because sample RS592, method blank, and LCS analyses were performed in the same 12-hour sequence as the initial calibration.</td></tr>
<tr><td>Method Blank</td><td>One method blank, SB -12/23, was correctly extracted and analyzed with sample RS592. Electronic data file was not submitted for SB -12/23 analyzed on 01/08/1998, at 9:02 pm. No contaminants were detected in the blank.</td></tr>
<tr><td>Laboratory Control Sample</td><td>One aqueous LCS was extracted on 12/23/97 with sample RS592 and analyzed on 01/08/98 at 9:52 pm. Electronic data file for this analysis was not submitted. All spike compounds met the method's percent recovery criteria.</td></tr>
<tr><td>Matrix Spike/Matrix Spike Duplicate (MS/MSD)</td><td>Sample CHMW5A-1 (Lab Code L9704210-001) was used to prepare the MS/MSD samples. The Auditor was not sure why this sample was used for MS/MSD analyses and their results were included in the data package for this SDG. These MS/MSD samples were analyzed on 12/30/1997 and 12/31/1997 respectively. These samples were analyzed before the ICAL standards associated with sample RS592.</td></tr>
</table>

QA/QC	Method 8270 (SVOC) Analyzed 12/30/97 -01/08/98 Sample ID: RS592 1- soil
	No extraction log was present for these samples so the Auditor could not verify that MS/MSD were extracted in the same batch as the sample RS592. Electronic data were not included for these analyses Percent recovery of phenol (MS -115% & MSD -118%) exceeded upper limit of the criteria window.
COC Forms, Sample Receipt Logs, Sample Extraction Logs, Standard Logs, and Instrument Run Logs	Standard Logs were not submitted.
Client Samples	Sample RS592 was extracted on 12/23/1997 and analyzed on 01/08/1998. Sample was analyzed at 10-fold dilution. Benzo(g,h,i)perylene result was incorrectly reported as 4500 ug/Kg. Using the on-column concentration reported on the Quantitation Report, Auditor calculated the result as 5400 ug/Kg. Six Tentatively Identified Compounds (TICs) were reported for this sample. Reference library Spectra were not included in the data package for any of these TICs. Percent solids for this sample was reported as 85%. All TIC concentrations were reported without correcting them for percent dryness. For example estimated concentration of hexadecane was reported as 8200 ug/Kg (Basis: Dry) on the TIC Analytical Report. Using the information from Library Search Compound Report (page # 4052), Auditor calculated concentration as 9300 ug/Kg. The Auditor noted transcription error on the TIC Analytical Report. TIC with retention time 24.19 minutes was reported as anthracene, 4-methyl (CAS # 000779-02-2). Compound with this CAS Number was identified as anthracene, 9-methyl on the Library Search Compound Report. Surrogate recovery of 2-fluorobiphenyl (118%) exceeded the upper limit of the criteria window (30-115). The Method 8270B clearly specifies that all surrogates must meet percent recovery criteria. Instead, the laboratory used the criteria specified in the CLP Statement of Work (SOW 2/88) that allows up to two surrogates to fail percent recovery requirement. Manual integrations were performed for two target compounds, benzo(b)fluoranthene and benzo(k)fluoranthene. Manual integrations were correctly performed.
Overview Comments	The data were generated by the Columbia Analytical Services laboratory and reviewed by the auditor using ChemStation software. The laboratory did not specify which revision of method 8270 was used for analysis. Since this data were generated in 1997, the Auditor used method 8270B (September 1994). Electronic data were **not** provided for the instrument performance check (decafluorotriphenylphosphene [DFTPP] tune) associated with initial calibration standards. Electronic data files were also missing for method blank (SB-12/23) and LCS (SL-12/23) analyzed with this SDG. The original data package did not contain electronic data files for the following analyses: a. DFTPP – Date/Time of analysis: 01/08/98 at 4:32 pm, b. GC/MS File ID: 0101001.D c. Method Blank: SB-12/23 d. LCS: SL-12/23 On May28, 2003 Lockheed Martin sent a request to provide electronic data for method blank and LCS analyses. The DPTPP data was not requested because the laboratory indicated that the electronic data file was irretrievable. Additional data was delivered to Lockheed Martin via CD (designated Rocketdyne CD2) that included three separate folders identified as GC07, L9704318-17, and MS02. This new CD included two new data files corresponding to Method Blank SB-12/23 and LCS SL-12/23 analyses. The remaining data files on the CD were previously included on the CD called Rocketdyne 2 that was submitted with the original data package. These new electronic data files were evaluated using ChemStation software. In the LCS analysis, manual integrations were correctly performed for 3-nitroaniline, and 4-nitrophenol. No problems were noted for Method Blank SB-12/23.

[illegible]	[illegible]
	The Auditor was unable to duplicate the 60 PPM standard response factor (RF) values reported for bis (2-chloroethyl)ether, 4-nitroaniline, and Carbazole on the Response Factor Report (Page # 4001 & 4002). No individual chromatograms of any of the manually integrated peaks in ICAL standards were provided in the data package. Reference library spectra confirming the presence of Tentatively Identified Compounds in sample RS592 were not included. Internal Standard (ISTD) Summary sheets, documenting shifts in retention time and changes in compound response (peak area), were not provided in the data package.

[redacted]

TABLE 3: SDG L9703923 AUDIT SUMMARY – COLUMBIA ANALYTICAL SERVICES

<table>
<tr><th>GC/MS</th><th>Method 8270SIM Analyzed :12/04/1997 -12/08/1997
Sample ID: RF262, RF282, RP286, RF287, RF289, RF861, [illegible], RF873, [illegible]</th></tr>
<tr><td>DFTPP Tunes</td><td>The auditor was not able to confirm that DFTPP tune analysis was performed, as required, before the initial calibration standards because both hard copy and electronic data were missing.

DFTPP tune analyses were correctly performed before the daily calibration standards. All tune analyses reported in the data package met ion abundance criteria as specified in Table 3 of Method 8270B.</td></tr>
<tr><td>Initial Calibration Standards (ICAL)

System Performance Check Compounds (SPCCs)

Calibration Check Compounds (CCCs)</td><td>In the analyses of the ICAL standards, manual integrations were performed for the following compounds:
<table>
<tr><th>Compound</th><th>0.2 ppm</th><th>0.4 ppm</th><th>0.8 ppm</th><th>1.0 ppm</th><th>4.0 ppm</th></tr>
<tr><td>N-Nitrosodimethylamine</td><td>x</td><td></td><td></td><td>32 cpds & IS were Manually Integrated on 2 separate days</td><td></td></tr>
<tr><td>Naphthalene</td><td>x</td><td></td><td></td><td></td><td></td></tr>
<tr><td>2-Fluorobiphenyl</td><td>x</td><td></td><td></td><td></td><td></td></tr>
<tr><td>Indeno (1,2,3-c,d) pyrene</td><td>x</td><td>x</td><td>x</td><td></td><td></td></tr>
<tr><td>Dibenzo(a,h)anthracene</td><td>x</td><td>x</td><td>x</td><td></td><td></td></tr>
<tr><td>Benzo[g,h,i]perylene</td><td>x</td><td>x</td><td>x</td><td></td><td></td></tr>
</table>
All compounds from the 1.0 PPM standard were manually integrated on December 5,1997 and again on December 18, 1997. The electronic data file included manual integration results obtained on December 18, 1997 while the hard copy data matched with the results obtained on December 5, 1997.

Chromatograms of the peaks after manual integration were not included with each standard Quantitation Report. The manual integrations were examined by the auditor and determined to be appropriate, and accurately performed.

All SPCCs met minimum RF limits, and all CCCs met the method requirement of <30% RSD for the RFs across the five concentration levels of the ICAL standards.

The Auditor was unable to duplicate the **RF1.0** value reported for **benzo[g,h,i]perylene** on the Response Factor Report (Page # 2001). The laboratory reported RF, %RSD values and the Auditor calculated values are included in the following table.
<table>
<tr><th colspan="2">Laboratory Reported</th><th colspan="4">Auditor Calculated</th></tr>
<tr><th>RF</th><th>%RSD</th><th colspan="2">Based on Hard Copy</th><th colspan="2">Based on Electronic File</th></tr>
<tr><td>1.079</td><td>26.30</td><td>0.978</td><td>28.1</td><td>1.1167</td><td>25.1</td></tr>
</table>
Three (non-CCC) target compounds exhibited %RSDs > 15% for the RFs in the ICAL standards. They were, indeno(1,2,3-c,d)pyrene (19.4%), dibenzo(a,h)anthracene (15.6%),and benzo(g,h,i)perylene (26.3%).

Indeno(1,2,3-c,d)pyrene and benzo[g,h,i]perylene were detected in the sample RF877 analyzed on 12/8/1997, at 00:18 (Dilution Factor = 100). Using average RF values reported on the Response Factor Report (Page # 2008), the auditor was able to duplicate on-column concentrations for these two compounds.

The laboratory should have used the regression line fitted to the initial calibration as an alternate procedure (see method 8270B, section 7.3.5.1) to determine extract concentrations of these compounds.</td></tr>
<tr><td>Continuing Calibration Verification Standard (CCV)</td><td>This data package included four CCV standards. CCV standards were analyzed at the required frequency. Percent difference of all CCC compounds met method specified criteria of <20%.</td></tr>
</table>

[illegible]	[illegible] Analyzed 12/04/1997 -12/08/1997 Sample ID: RF262, [illegible], RF286, RF287, RF289, RF861, RF866, RF873, RF874, RF877 10 –soil
Method Blank	One method blank, SB -11/26, was extracted on 11/26/1997 along with nine field samples included in this SDG. Sample RF262, RF262MS, and RF262MSD were extracted on 11/25/199. Method blank associated with this extraction batch and analyzed on 12/07/97 at 4:44 am. No contaminants were detected in the blank.
Laboratory Control Sample (LCS)	One LCS, SL-11/26 was extracted on 11/26/97 with all nine samples included in this SDG and analyzed on 12/07/97 at 5:38 am. All spike compounds met percent recovery criteria as stated in the Table 6 of Method 8270B.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Sample RF262 (Lab Code L9703846-010) was used to prepare the MS/MSD samples. This sample did not belong to this SDG. RF262MS and RF262MSD samples were analyzed on 12/06/1997 and 12/07/1997 respectively. The only information reported for sample RF262 was page 1016 of the Analytical Report. On this report, Service Request field indicated L9703846 as the CAS SDG Number. Analytical reports of all other field samples indicated Service Request as L9703923. Since the Service Request field information for RF262 did not match with rest of the samples, the auditor concluded that the sample RF262 belong to a different SDG. No extraction log was present for these samples. QA/QC Report (page # 1015) indicated extraction date as 11/25/1997, which was different from all samples, method blank, and LCS. Electronic data files were not included for MS, MSD, and sample RF262. All percent recovery values and RPD values reported on the QA/QC Report met method specific criteria.
COC Forms, Sample Receipt, Sample Extraction, Standard, and Instrument Run Logs	COC Forms, Sample Receipt Logs, and Standard Logs were not submitted.
Client Samples	There were nine field samples present in this SDG. Three of the samples were also analyzed at secondary dilution because few compounds exceeded calibration range in the original analyses. Analytical Reports for Tentatively Identified Compounds (TICs) were not submitted for any samples. Percent Solids information was not reported for seven out of nine samples - RF262, RF282, RF286, RF289, RF861, RF873, and RF874. All undiluted samples except RF287 met surrogate recovery criteria. RF262 (Dilution Factor = 1). This sample was used for MS and MSD analysis. Only Analytical Report of Selected Ion Monitoring (SIM) Semivolatile Organic Compounds was submitted for this sample (page # 1016). Auditor could not evaluate any results because Quantitation Report and Electronic data files were not provided with the SDG. QA/QC Report submitted for MS/MSD analyses indicated that this sample was extracted on 11/25/1997 and analyzed on 12/07/1997. However. Auditor could not confirm this information because Extraction Log for 11/25/1997 date was not included in the hard copy data package. RF282 (Dilution Factor = 1). No target compounds were detected in this sample. RF286 (Dilution Factor = 1). No target compounds were detected in this sample. RF287 (Dilution Factor = 1). Percent recovery of surrogate terphenyl-d14 (>700%) exceeded the upper limit of the criteria window. The laboratory reported presence of non-target compounds as the reason for this unusual high percent recovery. However no TICs were reported for this sample. Area count of the internal standard chrysene-d12 (121584) was outside the criteria the lower limit of the criteria window (403088 - 16112352). This criteria window was based on the internal standard area count from 1.0 PPM CCV standard analyzed on 12/07/1997, at 10:44. Bis(2-ethylhexyl)phthalate concentration (29.8 ug/L) exceeded the upper limit of the initial calibration curve (4.0 ug/L). The laboratory correctly re-analyzed this sample at a higher dilution to detect this compound below the upper limit of the initial calibration curve. RF287DL (Dilution Factor = 10). Bis(2-ethylhexyl)Phthalate (0.89 ug/L) was detected below the upper limit of the initial calibration curve (4.0 ug/L).

[illegible]	Method [illegible] Analyzed: 12/04/1997 -12/08/1997 Sample ID: [illegible] RF873, RF874, RF877 10 – soil
	RF289 (Dilution Factor = 1). No target compounds were detected in this sample. RF861DL (Dilution Factor = 10). No target compounds were detected in this sample. Auditor was unable to determine why this sample was analyzed at 10 fold dilution. Since no target compounds were detected in this diluted analysis, the laboratory should have analyzed this sample at a lower dilution to verify that no target compounds were diluted out. RF866DL (Dilution Factor = 10). Concentrations of three target compounds exceeded the upper limit of the initial calibration curve. They were Phenanthrene, fluoranthene, and pyrene. The laboratory correctly re-analyzed this sample at a higher dilution to detect these compounds below the upper limit of the initial calibration curve. Manual integrations were correctly performed for benzo(b)fluoranthene and benzo(k) fluoranthene. RF866DLDL (Dilution Factor = 20). Phenanthrene, fluoranthene, and pyrene were detected below the upper limit of the initial calibration curve. Manual integrations were correctly performed for benzo(b) fluoranthene and benzo(k)fluoranthene. RF873DL (Dilution Factor = 10). No target compounds were detected above Practical Quantitation Limits (PQLs) and therefore were reported as non-detects by the laboratory. The Auditor was unable to determine why this sample was analyzed at 10-fold dilution. Since no target compounds were detected above PQLs in this diluted analysis, the laboratory should have analyzed this sample at a lower dilution to verify that no target compounds were diluted out. Manual integrations were correctly performed for naphthalene and 2-methylnaphthalene peaks. RF874 (Dilution Factor = 1). No target compounds were detected in this sample. RF877DL (Dilution Factor = 100). Concentrations of three target compounds exceeded the upper limit of the initial calibration curve. They were Phenanthrene, fluoranthene, and pyrene. The laboratory correctly re-analyzed this sample at a higher dilution to detect these compound below the upper limit of the initial calibration curve. Manual integrations were correctly performed for benzo(b)fluoranthene and benzo(k) fluoranthene. Indeno(1,2,3-c,d)pyrene and benzo(g,h,i)perylene were also detected in this sample. It appears that the laboratory used average RF from the initial calibration to quantitate these samples. These two compounds did not meet initial calibration %RSD criteria of 15% as defined in method 8270B. The laboratory should have used the regression line fitted to the initial calibration as an alternate procedure (see method 8270B, section 7.3.5.1) to determine extract concentrations of these compounds. RF877DLDL (Dilution Factor = 1000). Phenanthrene, fluoranthene, and pyrene were detected below the upper limit of the initial calibration curve.
Overview Comments	The data were generated by the Columbia Analytical Services laboratory and reviewed by the auditor using ChemStation software. The laboratory did not specify which revision of method 8270 was used for analysis. Since this data were generated in 1997, the Auditor used method 8270B (September 1994). Both electronic data file and hard copy results were missing for the instrument performance check (decafluorotriphenylphosphene [DFTPP] tune) associated with initial calibration standards. Electronic data files were not provided for sample RF262, RF262MS and RF262MSD analyses. Hard copy of the Quantitation Report was not provided for RF262. The Auditor was unable to duplicate 1.0 PPM standard response factor (RF) values reported for benzo [g,h,i]perylene on the Response Factor Report (Page # 2001). No individual chromatograms of any of the manually integrated peaks in ICAL standards were provided. Internal Standard (ISTD) Summary sheets, documenting shifts in retention time and changes in compound response (peak area), were not provided.

TABLE 4: SDG L9800196 AUDIT SUMMARY – COLUMBIA ANALYTICAL SERVICES

[illegible]	Method 8082 – PCB Analyzed: 02/03/1998 - 02/04/1998 Sample ID RS652 - 1 soil
Initial Calibration Standards (ICAL)	The laboratory performed five point calibration for Aroclor 1254 and Aroclor 1260. As per method 8082, when PCBs are to be quantitatively determined as Aroclors, the initial calibration consists of two parts, 1) a five point initial calibration using mixture of Aroclor 1016 and 1260, and 2) single point calibration using other five Aroclors. Exception to this requirement is when only a few Aroclors are of interest for a specific project. In that situation a five point initial calibration of each of the Aroclors of interest (e.g., Aroclor 1254) is the only requirement. It appears from the calibration data submitted that Aroclor 1254 was the only Aroclor of interest for this project. However neither the case narrative nor the Chain of Custody (COC) forms documented this requirement. The laboratory selected only one peak to report retention time and response for each calibration standard. As per method 8082, the laboratory should have selected a minimum of three peaks for each Aroclor. The laboratory did not include Quantitation Reports with surrogate compound information for any of the ICAL standards. Therefore, the Auditor could not verify the surrogate calibration factors reported for five standards on the Response Factor Report (page # 2001) The percent standard deviation (%RSD) of Response Factors (RF) between five standards was less than method specified 20% limit.
Continuing Calibration Verification Standard (CCV)	The laboratory used 1.0 PPM Aroclor 1260 standard for CCV analysis. One CCV was analyzed before the five point initial calibration and sample analysis. The second one was analyzed after the sample analysis. The percent difference between the reported calibration factors from both CCV standard and the average calibration factor from the initial calibration was less than 15%. Retention time and Response of only one Aroclor 1260 peak was reported.
Method Blanks	One method blank (SB-1/29) was extracted on 01/29/1998 with the sample and analyzed on 02/04/1998, at 02:40 a.m. No hits were reported for this analysis. Surrogate percent recovery was within the criteria established by the laboratory.
Laboratory Control Sample (LCS)	One LCS (SL-1/29) was extracted on 01/29/1998 with the sample and analyzed on 02/04/1998, at 03:23 a.m. Aroclor 1260 was used as a spike compound. The percent recovery of Aroclor 1260 (139%) was within the acceptance limit reported on the QA/QC Report (page # 1005). The laboratory did not include the Quantitation Report for the surrogate compound. Therefore, the Auditor could not verify the percent recovery reported on the QA/QC Report (page #2043R).
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Sample RS652 (Dilution factor = 10) was used to prepare the MS/MSD samples. Aroclor 1260 was used as a spike compound. As per the footnote on MS/MSD QA/QC Report, the percent recoveries of Aroclor 1260 were not reported by the laboratory due to interference from peaks of other target compounds. The laboratory did not include the Quantitation Report for the surrogate compound. Therefore, the Auditor could not verify the percent recovery reported on the QA/QC Report (page # 2043R).
COC Forms, Sample Receipt, Sample Extraction , Standard and Instrument Run Logs	Standard Logs were not submitted.

Client Samples	Sample RS652 was analyzed at a 10 fold dilution. Aroclor 1254 was reported as detected at 6000 ug/kg concentration. Surrogate percent recovery (127%) was within the QC criteria window (45-140) reported on the QC/QC Report (page # 1003). The Auditor could not verify if the surrogate met retention time criteria because surrogate retention times were not reported for either initial or continuing calibration standards.
Overview Comments	The data was generated by the Columbia Analytical Services laboratory. The laboratory identified Analysis Method as 8080 in the data package. The laboratory used wide-bore capillary columns DB-608 and DB-1701 (0.53mm x 30m). SW-846 method 8082 specifies to use these types of wide-bore capillary columns. On the other hand, SW-846 method 8080 specifies the use of GC column of 4mm x 1.8m dimensions. Therefore, the Auditor used method 8082 (December 1996) to evaluate this package. Electronic data files were not submitted for any analysis. Method 8082 clearly specifies the use of decachlorobiphenyl (DCB) as a surrogate compound when PCBs are to be determined as Aroclors. Instead of DCB, the laboratory used tetrachloro-meta-xylene (TCMX) as a surrogate compound. As per method 8082, TCMX is used when PCBs are quantitated as congeners by internal standard method.

TABLE 5: SDG L9803443 AUDIT SUMMARY – COLUMBIA ANALYTICAL SERVICES

[illegible]	[illegible]
Initial Calibration Standards (ICAL)	The laboratory performed five point calibration using a mixture of Aroclor 1016 and Aroclor 1260 as defined in method 8082. Calculated values of average retention times and retention time windows for each Aroclor peak and surrogate were not reported. %RSD of calibration factors met the method specific criteria of <20%RSD. Electronic files were not submitted.
Continuing Calibration Verification Standard (CCV)	The laboratory used a mixture of Aroclor 1016 and 1260 standard (1.0 PPM) for CCV analysis. There were seven CCV analyses reported with this SDG. Electronic data files were missing for two CCV analyses (File ID 1028039.D and 1028051.D) All samples except RS838 (Lab ID 3440-10) were correctly bracketed by CCV analyses as per the method requirement. This sample was analyzed on 10/30/1998, at 1:03 p.m. Data package contained a CCV standard analyzed before this sample (10/30/1998, at 4:01 a.m.) but no CCV standard data was included to indicate a CCV analysis after sample RS838. The laboratory should have analyzed CCV standard on 10/30/1998 between 1:03 p.m. and 4:01 p.m. to meet 12-hour frequency and bracketing criteria as specified in the method. For any CCV standard, the laboratory did not report percent difference (%D) values between the CCV calibration factors and the average calibration factor from the initial calibration. Due to time limitation, the auditor calculated %D values for only two peaks, one each for Aroclor 1016, and 1260, from each CCV. The auditor calculated %D values were less than 15% as required by method 8082.
Method Blanks	One method blank (SB-10/19) was extracted on 10/19/1998 with all samples and analyzed on 10/23/1998, at 12:45 p.m. No hits were reported for this analysis. Quantitation Report indicating Aroclor 1016 and 1260 as non-detects was included in the data package. Surrogate percent recovery was within the criteria.
Laboratory Control Sample (LCS)	One LCS (SL-10/19) was extracted on 10/19/1998 with all samples and analyzed on 10/23/1998, at 1:28 p.m. Aroclor 1260 was used as a spike compound. The percent recovery of Aroclor 1260 (108%) was within the acceptance limit of 78-140%. Surrogate percent recovery was within the criteria.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Sample RS824 was used to prepare the MS/MSD samples. Aroclor 1260 was used as a spike compound. The percent recoveries of Aroclor 1260 in MS (143%) and MSD (125%) were within the acceptance limit of 62-154% reported on the QA/QC Report (page # 7018). Relative Percent Difference (RPD) between MS/MSD percent recoveries was 14%. No criteria for RPD were specified on the QA/QC Report (page # 7018).
COC Forms, Sample Receipt, Sample Extraction, Standard, and Instrument Run Logs	Standard Logs were not submitted.

[illegible]	[illegible] - PCB [illegible] [illegible]
Client Samples	There were fifteen field sample present in this SDG. Samples RS830 and RS843 were analyzed at 10 fold dilution. All the remaining samples were analyzed as undiluted. No explanation was provided in the case narrative for using 10 fold dilution to analyze these two samples. Electronic data files were missing for samples RS830 (File ID 1028048.D), RS838 (File ID 1028059.D), and RS843 (File ID 1028047.D) Percent Solids information was not reported for nine out of fifteen samples - RS825, RS828, RS830, RF838, RS839, RS840, R843, RS844, and RS845. Quantitation Reports indicating Aroclor 1016 and 1260 as non-detects were not included in the data package for following samples - RS828, RS830, RF838, RS839, RS840, R843, RS844, and RS845. Aroclor 1260 was reported as detected above the Practical Quantitation Limit (PQL) in following samples - RS826 (280 ug/Kg), RS836 (220 ug/Kg), RS841 (150 ug/Kg), and RS842 (140 ug/Kg). All samples met surrogate percent recovery criteria.
Overview Comments	The Auditor used method 8082 (December 1996) to evaluate this package. Hard copies of Quantitation Reports indicated that DB-608 (Signal #2) was used as a primary column and DB-1701 (Signal #1) was used as a confirmation column. The hard copy of the Response Factor Report (page # 8013) indicated that Signal #2 (i.e., GC column DB-608) was from the confirmation column. All sample results were reported from Signal #1 data, which agreed with the Response Factor Report. Therefore, the Auditor used Signal #1 responses to verify reported sample results. Method 8082 clearly specifies the use of decachlorobiphenyl (DCB) as a surrogate compound when PCBs are to be determined as Aroclors. Instead of DCB, the laboratory used tetrachloro-meta-xylene (TCMX) as a surrogate compound. As per method 8082, TCMX is used when PCBs are quantitated as congeners by internal standard method. Electronic data files were not provided for five point initial calibration 1016/1260 standards. Electronic data files were also not provided for two out of seven CCVs and three out of fifteen samples.

TABLE 6: SDG RJ028 AUDIT SUMMARY – PACE

QA/QC	**SW-846 8290 – Dioxins** **Analyzed: 8/25/00 – 9/7/00** **Sample IDs: RJ028, RJ029, RJ030, RJ545,RJ546, RJ032, RJ035, RJ036, RJ553,J561, RJ563, RJ555 12 - Soil**
Mass Spectrometer Resolution	Documentation is required for a minimum of one MS tune/resolution demonstration at the beginning and end of each 12-hour analytical sequence. The Case Narrative states that "The resolution of the mass spectrometer is verified prior to each analysis to be 10,000 or greater. Hardcopies of the reference peaks are printed at the beginning and end of each analysis day." However, the auditor was unable to locate any MS tuning documentation specifying the reference masses, showing the reference peak profiles, or demonstrating the ability of either of the instruments to meet the minimum resolving power of 10,000. Section 8.2.2.3 of the method states "The result of the peak width measurement (performed at 5 % of the maximum, which corresponds to the 10% valley definition) *must appear on the hard copy* and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass)." There were no such measurements evident on the submitted data.
Column Performance Checks	Verification of acceptable GC column performance/resolution must be documented prior to each 12-hour analytical sequence. Section 8.2.1 of the method specifies that in the analysis of the performance check standard (the laboratory's column performance mix or CPM), "all peaks should be labeled and identified on the chromatograms. Furthermore, all first eluters of a homologous series should be labeled with the letter F, and all last eluters of a homologous series should be labeled with the letter L." On the CPM data submitted by the laboratory, no peaks were identified or labeled as first/last eluters. The two CPM chromatograms used for the GC Column Resolution Checks were included in the data package for the 70-SE instrument (used only for one QC sample applicable to this package). Two pages of selected ion current profiles (SICPs) for the CPM analysis on 8/25/00 were missing. For the 70-VSE instrument, the first four pages of the CPM SICPs were missing from the ICAL raw analytical data; this included the chromatogram used for the resolution check/valley measurement. Consequently, there is none of the documentation that is required to demonstrate that analyses can take place. The SICPs for all of the on-going CPM analyses (i.e., run in conjunction with the daily Calibration Verification Standards) were present and complete. The auditor was unable to verify any of the GC Column Resolution Checks (a valley of □25% between the 2,3,7,8-TCDD peak and the peaks of other unlabeled TCDD isomers) since none of the peaks were labeled. In the Instrument Run Logs valley measurements were entered for each of the CPMs analyzed; however, there were no measurements or calculations written on the chromatograms to document the resolution.
Initial Calibration Standards [ICAL, CS1-CS5]	Data for ICAL standards on each of the two instruments were submitted. With the exception of a single LCS, all samples were analyzed on instrument 70-VSE. Unless otherwise indicated, the following comments apply to ICAL analyses on both instruments. • Relative Ion Abundance ratios reported for PCDDs/PCDFs, ISTDs, and Recovery Standards were within method-specified limits. The auditor recalculated ~ 20% of the ratios, all of which were verified as correctly reported. • The %RSDs for the isomer RRFs from the 5-point ICAL met method acceptance criteria. RSDs were recalculated for 20% of the compounds, all of which were verified as correctly reported. • Approximately 5% of the RRFs for the ICAL standards (labeled and unlabeled) were recalculated by the auditor; all were verified as correctly reported.
Daily Calibration Verification Standards [CVS, CS3]	Data for CVSs on each of the two instruments were submitted. Unless otherwise indicated, the following comments apply to CVS analyses on both instruments. • Relative Ion Abundance ratios reported for PCDDs/PCDFs, ISTDs, and Recovery Standards were within method-specified limits. The auditor recalculated ~ 20% of the ratios, all of which were verified as correctly reported. • The percent difference measurements (%Ds) between the ICAL mean RRFs and the RRFs from the CVS analyses met method acceptance criteria. Percent differences were

QA/QC	**SW-846 8290 – Dioxins Analyzed: 8/25/00 – 9/7/00** **Sample IDs: RJ028, RJ029, RJ030, RJ545,RJ546, RJ032, RJ035, RJ036, RJ553,J561, RJ563, RJ555 12 - Soil**
	recalculated for 20% of the compounds, all of which were verified as correctly reported. • Approximately 5% of the RRFs for the CVSs (labeled and unlabeled) were recalculated by the auditor; all were verified as correctly reported. • Method 8290 specifies that a CVS must be run at the beginning and end of each 12-hour analytical period. In several cases on instrument 70-VSE, more than 12 hours elapsed between the CVS analyses bracketing routine or QC samples in this package: a. 8/29/00 - CVSs were run at 01:48 and 14:24 (12 hr, 36 min). b. 8/31-9/1/00 - CVSs were run at 19:40 and 10:25 (14 hr, 45 min). c. 9/6/00 - CVSs were run at 08:00 and 20:47 (12 hr, 47 min). d. 9/6-7/00 - CVSs were run at 20:47 and 08:59 (12 hr, 12 min).
Method Blanks	Method blanks were prepared/analyzed for each of the three extraction batches. • In method blanks from the 8/3-4/00 and 8/21-22/00 batches, all Relative Ion Abundance ratios reported for any identified PCDDs/PCDFs, the ISTDs, and Recovery Standards were within method-specified limits. In the method blank from the 8/7-8/00 batch, two ratios exceeded the control limits: a. 1,2,3,4,6,7,8-HpCDF ratio was 1.35; the control limits are 0.88 - 1.20. OCDF ratio was 1.11; the control limits are 0.76 - 1.02. b. In both cases, the ratios were flagged "I" indicating the presence of an interfering compound. The auditor recalculated ~ 10% of the ratios, all of which were verified as correctly reported. • All three method blanks contained target isomers. a. 8/3-4/00 blank contained OCDD at 2.0 ng/Kg (flagged "J", below calibration range). b. 8/7-8/00 blank contained 1,2,3,4,6,7,8-HpCDF, OCDF, and OCDD (the latter at ~ 36 ng/Kg; the Detected Peak List also showed "Other HpCDD" present, but no Blank Analysis Results form was submitted for this method blank, so the precise amounts of the contaminants are not readily available. c. 8/21-22/00 blank contained OCDD at 9.1ng/Kg (flagged "J"). • All ISTD and clean-up standard recoveries met the method acceptance criteria in the 8/3-4/00 and 8/21- 22/00 blanks; these data were not available for the 8/7-8/00 method blank.
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	The reported MS/MSD set was prepared using sample 200571-11, RJ563 from the 8/7-8/00 extraction batch. MS/MSD sets were prepared with the other two extraction batches; however, these soils came from other Project Numbers and the results were not included in this data package. • Relative Ion Abundance ratios reported for PCDDs/PCDFs, ISTDs, and Recovery Standards were within method-specified limits. The Auditor recalculated about 10% of the ratios, all of which were verified as correctly reported. • Recoveries for the ISTDs and clean-up standards met method criteria in both samples. • Spiked compound recoveries and relative percent differences (RPDs) met all method QC criteria.
Laboratory Control Spikes (LCS)	An LCS was prepared with each extraction batch. The LCS for the 8/7-8/00 batch was analyzed on instrument 70-SE. While the PCDD/PCDF Detected Peak Lists and SICPs were submitted for each of the three LCS analyses, the Laboratory Control Spike Results form was only included for the 8/21-22/00 LCS. • Relative Ion Abundance ratios reported for PCDDs/PCDFs, ISTDs, and Recovery Standards were within method-specified limits in all three LCSs. The auditor recalculated ~ 10% of the ratios, all of which were verified as correctly reported. • Recoveries for the ISTDs and clean-up standards met method criteria in the 8/21-22/00 LCS. recoveries were not available in the other two LCSs. • Spiked compound recoveries met laboratory QC criteria (individual spike recovery limits are not specified in Method 8290.
Client Samples	Recoveries for the ISTDs and clean-up standards met method criteria in all 12 samples. The PCDD/PCDF Detected Peak Lists for samples RJ029, RJ030, and RJ545 were not included in the data package, so the Ion Abundance Ratios for these samples could not be reviewed/confirmed by the auditor. Many of the samples had at least one Ion Abundance Ratio falling outside of the control limits. There did not seem to be any pattern, i.e., no particular isomer was more commonly out of acceptance. The most problematic sample was 200546-17, RJ036 with five of the native (unlabeled) isomers giving ratios outside of the limits; in three of the five cases, the reported results were flagged "I", indicating the presence of interferences.

QA/QC

SW-846 8290 – Dioxins **Analyzed: 8/25/00 – 9/7/00**

Sample IDs: RJ028, RJ029, RJ030, RJ545,RJ546, RJ032, RJ035, RJ036, RJ553,J561, RJ563, RJ555 12 - Soil

The Table 6A lists the polychlorinated dibenzodioxin (PCDD) and polychlorinated dibenzofuran (PCDF) isomers in the Rocketdyne site samples that showed ion abundance ratios exceeding Method 8290 control limits. The ion abundance ratios were examined as part of the audit of data from Pace Analytical Services, Inc.

SW-846 Method 8290 states in Section 7.7.1.4 that "the ratio of integrated ion current for the ions appearing in Table 8 (of the method, the homologous series quantitation ions) must be within the indicated control limits (set for each homologous series) for all unlabeled calibration standards" and "for the ions belonging to the carbon-labeled internal and recovery standards." The method also states that the ion ratios must be within the specified control limits simultaneously in one run, and that "It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits." The method does not state what type of corrective action should be taken.

For the samples listed in the **Table 6A**, analytical results should be considered "estimated" due to the reported ion abundance ratios being outside of the specified range. In two of the samples, RJ555 / 200571-03A and RJ563 / 200571-11, all ion abundance ratios met the method acceptance criteria. These two samples are not listed in the table.

General Comments

The 12 soil samples included in this audit were extracted in three batches:

Five samples were received on 7/25/00 and extracted on 8/3-4/00 (Batch 103969):

Client Sample ID	Pace Sample ID
200546-01 RJ028	2169489
200546-02 RJ029	2169497
200546-03 RJ030	2169505
200546-05 RJ545	2169513
200546-06 RJ546	2169521

Six samples were received on 8/1/00 and extracted on 8/7-8/00 (Batch 1035168):

Client Sample ID	Pace Sample ID
200546-13 RJ032	2184868
200546-16 RJ035	2184884
200546-17 RJ036	2184900
200571-01 RJ553	2184835
200571-09 RJ561	2184843
200571-11 RJ563	2184850 (MS/MSD matrix)

One sample was received on 8/7/00 and extracted on 8/21-22/00 (Batch 1035368):

Client Sample ID	Pace Sample ID
200571-03A RJ555	2196169

All 12 samples were analyzed on instrument 70-VSE, using the initial calibration (ICAL) run on 8/18/00. All but one of the associated QC samples were also analyzed with that instrument and ICAL; the laboratory spike prepared with the 8/7-8/00 extraction batch was analyzed on Instrument 70-SE using an ICAL run on 8/23/00. ICAL and daily verification standards from both instruments were included in the data package.

The ReadMe file for the electronic data states that both raw instrument data (*.DAT files) and processed data (*.APK files) from the SpectraChrom AutoPro software are included on the CD. The files for samples with Project No. 200546 (i.e. the five samples in the first batch and three of the six samples in the second batch) are on the CD, as are the applicable standards, blanks, and laboratory control spikes (laboratory terminology, LCSs).

Files for the matrix spike/matrix spike duplicate (MS/MSD) prepared using the soil sample 200571-11 (RJ563) from this set of 12 samples were not included on the CD. The remainder of the sample and associated QC files (i.e., the other three of six samples in the second batch and the single sample in the last extraction batch) were also missing from the CD provided by the laboratory.

Because Lockheed Martin does not currently have the SpectraChrom software, the auditor could not examine the raw instrument data for the areas of the individual ion masses. Reviews and recalculations of ion abundance ratios and RFs were performed using the data reported on the PCDD/PCDF Detected Peak Lists.

QA/QC	**SW-846 8290 – Dioxins** **Analyzed: 8/25/00 – 9/7/00** **Sample IDs: RJ028, RJ029, RJ030, RJ545,RJ546, RJ032, RJ035, RJ036, RJ553,J561, RJ563, RJ555 12 - Soil**
	Method 8290 states in Section 3.4 that no single column is capable of completely resolving all of the isomers, and, while the 60-m DB-5 column is capable of 2,3,7,8-TCDD isomer specificity, "in order to determine the concentration of the 2,3,7,8-TCDF (if detected on the DB-5 column) the sample extract must be reanalyzed on a column capable of 2,3,7,8-TCDF isomer specificity..." Although values (1.8 - 2.5 ng/Kg) are reported for the 2,3,7,8-TCDF isomer in five of the samples, no data from analyses of these extracts on a secondary column (e.g., DB-225, SP-2330) are included in the data package. The Case Narrative discusses the interference of polychlorinated diphenylethers (PCDEs) with PCDF isomers in several of these samples. It states that "Any responses in the PCDF ion traces with corresponding responses in the PCDE ion traces are not included in the reported PCDF concentrations. It is possible that these compounds would be resolved on a secondary column.
Documentation Review	The following form pages were missing from the data package: a. PCDD/PCDF Detected Peak List for RJ029 (all pages), RJ030 (all pages), RJ545 (pages after #1). b. Method 8290 Blank Analysis Results for BLANK-080700 (which had two PCDD isomer contaminants present) c. Method 8290 Laboratory Control Spike Results for SPIKE-080300 and SPIKE-080700 (PCDD/PCDF Detected Peak List included for all 3 LCSs) d. Instrument 70-VSE ICAL; PCDD/PCDF Detected Peak List for CS-3 (page 2). No samples to be used as MS/MSD matrices were designated on the COC from Ceimic Corporation. Also, no field blanks, trip blanks, or field sample duplicates were submitted. All applicable pages of the laboratory Extraction Logs and Instrument Run Logs were included in the data package.

Table 6A - **Isomers in Dioxin/Furan Samples with Ion Abundance Ratios __Exceeding__ Method 8290 Control Limits**

SDG / Sample Number	Isomer	Retention Time	Reported Ion Abundance Ratio	Lab Reported Interference	Method 8290 Control Limits
RJ028 / 200546-01	1,2,3,7,8-Penta-CDF	38:00	1.18	Yes	1.32 - 1.78
"	1,2,3,4,7,8-Hexa-CDF	42:38	0.97	Yes	1.05 - 1.43
"	1,2,3,7,8,9-Hexa-CDF	44:24	2.11	Yes	1.05 - 1.43
RJ029 / 200546-02	No *PCDD/PCDF Detected Peak List* was submitted so Ion Abundance Ratios were not available				
RJ030 / 200546-03	No *PCDD/PCDF Detected Peak List* was submitted so Ion Abundance Ratios were not available				
RJ545 / 200546-05	2,3,4,7,8-Penta-CDF	39:12	1.28	Yes	1.32 - 1.78
"	Pages 2 and 3 of the *PCDD/PCDF Detected Peak List* were not submitted so not all of the Ion Abundance Ratios				
RJ546 / 200546-06	2,3,7,8-Tetra-CDF	32:24	0.62	Yes	0.65 - 0.89
RJ032 / 200546-13	1,2,3,4,6,7,8-Hepta-CDD	47:02	1.50	No**	0.88 - 1.20
RJ035 / 200546-16	2,3,4,6,7,8-Hexa-CDF	43:24	1.03	No**	1.05 - 1.43
RJ036 / 200546-17	2,3,4,7,8-Penta-CDF	39:11	1.07	Yes	1.32 - 1.78
"	1,2,3,4,7,8-Hexa-CDF	42:35	0.93	Yes	1.05 - 1.43
"	2,3,4,6,7,8-Hexa-CDF	43:24	1.58	No**	1.05 - 1.43
"	1,2,3,4,7,8-Hexa-CDD	43:33	0.69	No**	1.05 -1.43
"	1,2,3,7,8,9-Hexa-CDD	43:59	1.56	Yes	1.05 - 1.43
RJ553 / 200571-01	1,2,3,4,6,7,8-Hepta-CDD	47:02	1.60	Yes	0.88 - 1.20
RJ561 / 200571-09	Octa-CDF	50:34	0.73	Yes	0.76 - 1.02

* EMPC = Estimated Maximum Possible Concentration

** An "I" (interference noted by analyst) was listed on the *PCDD/PCDF Detected Peak List* by this isomer, but was not reported on the *Method 8290 Analysis Results* form.

Table 7: Data Acceptability Summary

Analytical Method	Laboratory SDG No.	Sample ID	Sample Results	Qualification	Explanation
8270B (SVOC)	Columbia L9704259	RS001	Phenol	Estimated	CCV of 21% Difference exceeded the 20% method criteria
			Bis(2-ethylhexyl)phthalate	Acceptable	
			Diethylphthalate	Possible contamination	Sample had 460 ug/kg, PQL was 330 ug/kg, and MB had 820 ug/kg.
			Non-Detects	Acceptable	
8270B (SVOC)	Columbia L9704318	RS592	Benzo(k)fluoranthene	Estimated	ICAL of 16.59% RSD exceeded the 15% method criteria
			Carbazole	Estimated	ICAL of 21.66% RSD exceeded the 15% method criteria
			Benzo(ghi)perylene should be 5400 ug/kg.		Incorrectly reported as 4500 ug/kg
			Other Detects	Acceptable	
			Non-Detects	Acceptable	
8270SIM (PAHS)	Columbia L9703923	RF262	-------------------------------	Unable to determine due to lack of information	Quantitation report was not submitted. The auditor could not determine which calibration standards were associated with this sample.
		RF282	Non-Detects	Acceptable	
		RF286	Non-Detects	Acceptable	
		RF287	Bis(2-ethylhexyl)phthalate (320ug/kg) DF = 10	Acceptable	
			Non-Detects	Acceptable	
		RF289	Non-Detects	Acceptable	
		RF861	All non-detects should be considered non-detects at the undiluted concentration. No target compounds were detected.		Only one analysis of this sample was performed at 10-fold dilution. No other sample analyses at lower dilution/no dilution were submitted to prove that target compounds were not diluted out because of the 10-fold dilution.
		RF866	Benzo(a)anthracene (DF10)	Acceptable	
			Chrysene (DF10)	Acceptable	
			Benzo(b)fluoranthene (DF10)	Acceptable	
			Benzo(a)pyrene (DF10)	Acceptable	
			Phenanthrene (DF20)	Acceptable	
			Fluoranthene (DF20)	Acceptable	
			Pyrene (DF20)	Acceptable	
			Non-Detects	Acceptable	
		RF873	All non-detects should be considered non-detects at the undiluted concentration. No target compounds were detected.		Only one analysis of this sample was performed at 10-fold dilution. No other sample analyses at lower dilution/no dilution were submitted to prove that target compounds were not diluted out because of the 10-fold dilution.
		RF874	Non-Detects	Acceptable	
		RF877	Indeno(123-cd)pyrene (DF100) (3900 ug/kg)	Estimated	ICAL of 19.39% RSD exceeded the 15% method criteria
			Benzo(ghi)perylene (DF100) (3700 ug/kg)	Estimated	ICAL of 26.3% RSD exceeded the 15% method criteria

Analytical Method	Laboratory SDG No.	Sample ID.	Sample Results	Qualification	Explanation
			Other Detects (DF100)	Acceptable	
			Phenanthrene (DF1000)	Acceptable	
			Fluoranthene (DF1000)	Acceptable	
			Pyrene (DF1000)	Acceptable	
			Non-Detects	Acceptable	
8082 (PCBs)	Columbia L9800196	RS652	Aroclor 1254 (6000 ug/kg)	Estimated	Instrument calibration used only one peak of the arochlor standard. Quantitation was performed using only 1 peak instead of a minimum of 3 peaks as recommended by the method
8081 (PCBs)	Columbia L9803443	RS824	All results	Acceptable	
		RS825	All results	Acceptable	
		RS826	All results	Acceptable	
		RS828	All results	Acceptable	
		RS830	Non-Detects should be considered Non-Detects at the undiluted concentration. No target compounds were detected.		Only one analysis of this sample was performed at 10-fold dilution. No other sample analyses at lower dilution/no dilution were submitted to prove that target compounds were not diluted out because of the 10-fold dilution.
		RS836	All results	Acceptable	
		RS837	All results	Acceptable	
		RS838	All results	Acceptable	
		RS839	All results	Acceptable	
		RS840	All results	Acceptable	
		RS841	All results	Acceptable	
		RS842	All results	Acceptable	
		RS843	Non-detects should be considered non-detects at the undiluted concentration. No target compounds were detected.		Only one analysis of this sample was performed at 10-fold dilution. No other sample analyses at lower dilution/no dilution was submitted to prove that target compounds were not diluted out because of the 10-fold dilution.
		RS844	All results	Acceptable	
		RS845	All results	Acceptable	
8290 (Dioxins)	PACE RJ028	200546-01-RJ028	1,2,3,7,8-PECDF 1,2,3,4,7,8-HxCDF 1,2,3,7,8,9-HxCDF	Estimated (Estimated Maximum Possible concentration or EMPC) .	The Ion Abundance Ratios (IARs) did not meet method criteria due to the presence of unspecified interference; the laboratory therefore reported the EMPCs.
			1,2,3,6,7,8-HxCDF	Estimated (EMPC)	Due to interference from polychlorinated diphenyl ethers (PCDEs), the laboratory reported the EMPC.
			Other reported isomers	Acceptable	
			Total 2,3,7,8-TCDD (22 ng/kg)	Acceptable	
		200546-02-RJ029	1,2,3,6,7,8-HxCDF	Estimated (EMPC)	Due to interference from PCDEs, the laboratory reported the EMPC

Analytical Method	Laboratory SDG No.	Sample ID	Sample Results	Qualification	Explanation
			Other reported isomers	Inconclusive (Could not be verified but appear to be acceptable)	The *Detected Peak List* forms were not submitted, thus the auditor was unable to verify IARs.
			Total 2,3,7,8-TCDD (23 ng/kg)	Acceptable	
		200546-03-RJ030	1,2,3,4,6,7,8-HpCDF	Estimated (EMPC)	Due to the presence of interferences the laboratory reported the EMPC.
			Other reported isomers	Inconclusive (Could not be verified but appear to be acceptable)	The *Detected Peak List* forms were not submitted, thus the auditor was unable to verify IARs.
			Total 2,3,7,8-TCDD (.08 ng/kg)	Acceptable	
		200546-05-RJ545	2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDD 1,2,3,4,7,8,9-HpCDF	Estimated (EMPC)	The IARs did not meet method criteria due to the presence of unspecified interference; the laboratory reported the EMPCs.
			Other isomers	Inconclusive (Could not be verified but appear to be acceptable)	The *Detected Peak List* forms were not submitted, thus the auditor was unable to verify IARs, however the laboratory indicated the presence of interferences.
			1,2,3,6,7,8-HxCDF	Estimated (EMPC)	Due to interference of PCDEs, the laboratory reported EMPC.
			Total 2,3,7,8-TCDD (2.0 ng/kg)	Acceptable	
		200546-06-RJ546	2,3,7,8-TCDF	Estimated (EMPC)	The IAR did not meet method criteria due to the presence of unspecified interference, the laboratory reported the EMPC.
			1,2,3,6,7,8-HxCDF	Estimated (EMPC)	Due to interference of PCDEs, the laboratory reported EMPC
			Other isomers	Acceptable	
			Total 2,3,7,8-TCDD (18 ng/kg)	Acceptable	
		200546-13-RJ032	Total HpCDD (1.3 ng/kg) "B" OCDD (10 ng/kg) "B"	Unacceptable	MB contained 1,2,3,4,6,7,8-HpCDD, other HpCDD, OCDF and OCDD. The *Blank Analysis Results* form was not submitted, so the isomer concentrations were not available. Manual calculations from raw MB gave an OCDD level of ~36 ng/kg. According to EPA Region IV , "any compound detected in the sample and was also detected in the blank is not reported if the sample concentration is less than 5X the blank concentration" . (EPA, 1998)* The OCDD should have been reported as ND and the sample reanalyzed.
			Other isomers: Non –Detects	Acceptable	
			Total 2,3,7,8-TCDD (0.01 ng/kg)	Provisionally acceptable	Too low, the correct value is likely 0.00 ng/kg.
		200546-16-RJ035	All reported Isomers	Acceptable	

Analytical Method	Laboratory SDG No.	Sample ID	Sample Results	Qualification	Explanation
			Total 2,3,7,8-TCDD (1.8 ng/kg)	Acceptable	
		200546-17-RJ036	2,3,4,7,8-PeCDF 1,2,3,4,7,8-HxCDF 1,2,3,7,8,9-HxCDD	Estimated (EMPC)	The IARs did not meet the method criteria due to the presence of unspecified interferences; the laboratory therefore reported the EMPCs.
			Total 2,3,7,8-TCDD (3.7 ng/kg)	Acceptable	
		200571-01-RJ553	1,2,3,4,6,7,8-HpCDD	Estimated (EMPC)	The IARs did not meet the method criteria due to the presence of unspecified interferences; the laboratory therefore reported the EMPCs.
			OCDD (17 ng/kg) "B" flag	Unacceptable	The MB extracted with this sample on 8/7/00 contained several isomers including OCDD. The *Blank Analysis Results* form was not submitted for this MB, so the isomer concentrations were not available. Manual calculations gave an OCDD level of ~36 ng/kg. According to EPA Region IV, "any compound detected in the sample and was also detected in the blank is not reported if the sample concentration is less than 5X the blank concentration" . (EPA,1998) * The OCDD should have been reported as ND and the sample reanalyzed.
			Other isomers: Non-detects	Acceptable	
			Total 2,3,7,8-TCDD (0.017 ng/kg)	Provisionally acceptable	Too low, the correct value is likely 0.00 ng/kg.
		200571-09-RJ561	OCDF	Estimated (EMPC)	The IARs did not meet the method criteria due to the presence of unspecified interferences; the laboratory therefore reported the EMPCs.
			OCDD (95 ng/kg) "B" flag	Unacceptable	The MB extracted with this sample on 8/7/00 contained several isomers including OCDD. The *Blank Analysis Results* form was not submitted, so the isomer concentrations were not available. Manual calculations gave an OCDD level of ~36 ng/kg. According to EPA Region IV , "any compound detected in the sample and was also detected in the blank is not reported if the sample concentration is less than 5X the blank concentration" . The OCDD should have been reported as ND and the sample reanalyzed. (EPA, 1998)*

Analytical Method	Laboratory SDG No.	Sample ID	Sample Results	Qualification	Explanation
			Other isomers	Acceptable	
			Total 2,3,7,8-TCDD (0.53 ng/kg)	Provisionally acceptable	Due to questions about the actual quantity of OCDD present.
		200571-11-RJ563	1,2,3,4,6,7,8-HpCDD (0.79 ng/kg) "B" flag Total HpCDD (0.79 ng/kg) "B" flag OCDD (5.0 ng/kg) "B" flag	Unacceptable	The MB extracted with this sample on 8/7/00 contained 1,2,3,4,6,7,8-HpCDD, other HpCDD, OCDF and OCDD. The *Blank Analysis Results* form was not submitted, so the isomer concentrations were not available. Manual calculations from raw MB gave an OCDD level of ~36 ng/kg. According to EPA Region IV , "any compound detected in the sample and was also detected in the blank is not reported if the sample concentration is less than 5X the blank concentration" . The OCDD should have been reported as ND and the sample reanalyzed. (EPA, 1998)*
			Total 2,3,7,8-TCDD (0.13 ng/kg)	Provisionally acceptable	
		200571-03A-RJ555	Isomers: Non-Detects Total 2,3,7,8-TCDD (0 ng/kg)	Provisionally acceptable	The only analysis submitted at a 2X dilution. No analysis of the undiluted samples was provided to demonstrate that the target compounds had not been diluted out.

* EPA. 1998. *Data Validation Standard Operating Procedures for Polychlorinated Dibenzodioxin and Polychlorinated Dibenzofuran Analysis by High Resolution Gas Chromatography/ High Resolution mass Spectrometry, Revision 2.0.* United States Environmental Protection Agency Region IV, Science and Ecosystem Support Division, Office of Quality Assurance, Athens, GA 30605-2720. October 1998.

Dioxin/Furan Data Packages

NOTE: Method 8290 requires that, prior to any analyses, a gas chromatograph (GC) column performance check solution (the Pace Laboratory's Column Performance Mix or CPM) and the high-resolution calibration solution No. 3 (the Pace Laboratory's Calibration Solution 3 or CS-3) be analyzed with acceptable results for sensitivity, resolution, response factor reproducibility, and mass range calibration. In this data package, the first four pages of data for the CPM run prior to the Initial Calibration standards were missing; these included the chromatogram used for the GC resolution check. The method further states that "Deviations from criteria specified for the GC performance check or for the mass resolution check invalidate all positive sample data collected between analyses of the performance check solution...." Because a portion of the analytical data for the initial CPM is unavailable for verification, all of the data generated using the 8/18/2000 Initial Calibration standards are technically invalid. However, the auditor assumes that examination of the initial CPM data would demonstrate the method criteria to have been met.

Acronym List

CAS	Columbia Analytical Services, Inc.
COC	Chain-of-Custody
CVS	Calibration Verification Solution
%D	Percent Difference
DUP	Duplicate
EDL	Estimated Detection Limit
EPA	U.S. Environmental Protection Agency
EMPC	Estimated Maximum Possible Concentration
GW	Ground Water
HpCDD	Heptachlorodibenzo-p-dioxin
HpCDF	Heptachlorodibenzofuran
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
HxCDD	Hexachlorodibenzo-p-dioxin
HxCDF	Hexachlorodibenzofuran
ICAL	Initial Calibration (standard runs)
ISTD	Internal Standard
LOD	Limit of Detection
LRMS	Low Resolution Mass Spectrometry
LCS	Laboratory Control Spike
MB	Method Blank
MQL	Method Quantitation Limit
MS	Matrix Spike
MSD	Matrix Spike Duplicate
MW	Monitoring Well
OCDD	Octachlorodibenzo-p-dioxin
OCDF	Octachlorodibenzofuran
PCDDs	Polychlorinated dibenzo-p-dioxins
PCDFs	Polychlorinated dibenzofurans
PeCDD	Pentachlorodibenzo-p-dioxin
PeCDF	Pentachlorodibenzofuran
ppb	parts per billion
ppt	parts per trillion
pq	picograms
QA	Quality Assurance
QC	Quality Control
%R	Percent Recovery
RRF	Relative Response Factor
%RSD	Percent Relative Standard Deviation
RT	Retention Time
SIM	Selected Ion Monitoring
S/N	Signal to Noise ratio
TCDD	Tetrachlorodibenzo-p-dioxin
TCDF	Tetrachlorodibenzofuran
TEF	Toxicity Equivalence Factor
RT	Retention Time